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Supplement A to the Unregulated Contaminant Monitoring Regulation Analytical Methods and Quality Control Manual

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Foreword

This document is the first supplement to the *Unregulated Contaminant Monitoring Regulation Analytical Methods and Quality Control Manual* (EPA 815-R-00-006). These two documents, when considered jointly, provide guidance regarding sampling, analytical methods, and related quality control issues for the States, EPA offices, water systems, and analytical laboratories participating in the monitoring for List 1 contaminants as part of the Unregulated Contaminant Monitoring Regulation (UCMR) Program. These documents are written for the personnel at a water system, laboratory, or agency who will be responsible for UCMR Program planning, implementation, and oversight.

Please Note: Because of the evolving nature of the UCMR Program, supplemental rule-making efforts may add additional contaminants to be monitored and hence, the specific analytical and technical requirements of the program will need to be identified. For this reason, EPA will periodically issue supplements to the Analytical Methods and Quality Control Manual. EPA anticipates developing additional supplements when analytical methods are approved for monitoring the UCMR (1999) List 2 and 3 contaminants in subsequent rules. However, these additional supplements may also include minor modifications to the requirements for monitoring noted in the QC Manual and in this Supplement. To ensure compliance with the UCMR, you should contact the Safe Drinking Water Hotline at (800) 426-4791 to be directed to the most recent supplements to the UCMR QC Manual.

Under §1445(a)(2)(A) of the Safe Drinking Water Act (SDWA), as amended in 1996, the Environmental Protection Agency (EPA) is to promulgate regulations for a monitoring program for unregulated contaminants by August 1999. In the past, unregulated contaminant monitoring has been performed according to the program described in CFR 141.40. The 1996 SDWA Amendments direct a substantially revised UCMR Program.

The revised UCMR Program has a new list of contaminants to monitor, changes the number of public water systems (PWSs) that must conduct monitoring, and changes the frequency and schedule for monitoring (§141.40(a)). Additional regulatory actions include cancellation of unregulated contaminant monitoring for small systems serving 10,000 or fewer people under the existing unregulated contaminant monitoring program begun in 1989. The data collected in the revised UCMR will be used to support the development of the Contaminant Candidate List (CCL), to support the Administrator's determination of whether to regulate a contaminant, and to support the development of future regulations. The revised monitoring program is one of the cornerstones of the sound science approach to future drinking water regulation that is an aim of the 1996 SDWA Amendments.

Further detailed information about the revised UCMR Program can be found in the Preamble to the proposed regulation (64 FR 23398) and the final Rule (64 FR 50556), as well as other supporting documents available by contacting the EPA Water Docket at (202) 260-3027 (Docket Number W-98-02). General information can be obtained from the EPA Safe Drinking Water Hotline, (800) 426-4791, or through the EPA Office of Ground Water and Drinking Water Internet Homepage at http://www.epa.gov/ogwdw. This QC Supplement also includes a revised versions of Appendices A and B from the *UCMR Analytical Methods and Quality Control Manual.* These appendices have been revised to reflect additional acronyms and abbreviations used throughout this QC Supplement, and additional clarifications in the list of definitions.

Disclaimers

This supplemental guidance document is designed to implement national policy concerning the UCMR Program. The document does not, however, substitute for the SDWA or EPA's regulations nor is this document a regulation itself. Thus, it cannot impose legally-binding requirements on EPA, States, or the regulated community, and may not apply to a particular situation based upon the circumstances. EPA decision makers retain the discretion to adopt approaches on a case-by-case basis that differs from this guidance where appropriate. EPA may change this guidance in the future.

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Acknowledgments

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Section 1. Introduction

This supplement to the *UCMR Analytical Methods and Quality Control Manual* (EPA 815-R-00-006) provides supplemental guidance for sampling and analytical and quality control procedures for acetochlor and perchlorate, two UCMR (1999) List 1 contaminants for which methods have recently been approved (65 FR 11371). This Quality Control (QC) Supplement is not intended to be a comprehensive quality control manual for the UCMR; it is meant to be used in conjunction with the *UCMR Analytical Methods and Quality Control Manual*. For clarity, all section and subsection titles in the original UCMR QC Manual are reprinted in this QC Supplement. If no information in addition to that provided in the original UCMR QC Manual is needed for the use of EPA Method 525.2 for acetochlor or EPA Method 314.0 for perchlorate, this fact is noted. Furthermore, all additions to tables are noted in boldface and all modified or added tables are noted with the suffix "s" (e.g., Table 3.1s is the modified version of Table 3.1). If a table from the QC Manual does not require any modifications, it has not been included in this Supplement.

1.1 Background on the Unregulated Contaminant Monitoring Regulation (UCMR)

No additional information for this section of the *UCMR Analytical Methods and Quality Control Manual* is necessary for the monitoring of acetochlor and perchlorate under the UCMR. In subsequent sections, this is noted by asterisks (see Section 1.2 below).

1.2 The Unregulated Contaminant Monitoring Regulation

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1.3 Contaminants on the UCMR (1999) List

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1.3.1 UCMR (1999) List 1 Contaminants

With the publication of the Direct Final Rule (65 FR 11371), EPA has approved analytical methods for all 12 chemical contaminants on the UCMR (1999) List 1. Monitoring for these contaminants is to begin in 2001 under the Assessment Monitoring component of the UCMR Program. All UCMR (1999) List 1 contaminants and their corresponding required sampling locations, approved analytical methods, and other related analytical details are listed in Table 1.2s (§141.40(a)(5)).

Table 1.2s UCMR (1999) List 1 Contaminants

Contaminants	CAS #	Approved Analytical Methods	Minimum Reporting Level	Sampling Point
2,4-Dinitrotoluene	121-14-2	EPA 525.2	2 μg/L [a]	EPTDS [b]
2,6-Dinitrotoluene	606-20-2	EPA 525.2	2 μg/L [a]	EPTDS [b]
4,4'-DDE	72-55-9	EPA 508, EPA 508.1, EPA 525.2, D5812.96, 990.06	0.8 μg/L [a]	EPTDS [b]
Acetochlor	**34256-82-1**	**EPA 525.2**	**2 μg/L [c]**	**EPTDS [b]**
DCPA mono-acid degradate	887-54-7	EPA 515.1, EPA 515.2, D5317-93, 992.32	1 μg/L [a]	EPTDS [b]
DCPA di-acid degradate	2136-79-0	EPA 515.1, EPA 515.2, D5317-93, 992.32	1 μg/L [a]	EPTDS [b]
EPTC	759-94-4	EPA 507, EPA 525.2, D5475-93, 991.07	1 μg/L [a]	EPTDS [b]
Molinate	2212-67-1	EPA 507, EPA 525.2, D5475-93, 991.07	0.9 μg/L [a]	EPTDS [b]
MTBE	1634-04-4	EPA 524.2, D5790.95, SM6210D, SM6200B	5 μg/L [d]	EPTDS [b]
Nitrobenzene	98-95-3	EPA 524.2, D5790.95, SM6210D, SM6200B	10 μg/L [d]	EPTDS [b]
Perchlorate	**1497-73-0**	**EPA 314.0**	**4 μg/L [c]**	**EPTDS [b]**
Terbacil	5902-51-2	EPA 507, EPA 525.2, D5475-93, 991.07	2 μg/L [a]	EPTDS [b]

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

[a] Minimum Reporting Level (MRL) determined by multiplying by 10 the least sensitive method's minimum detection limit (MDL=standard deviation times the Student's T value for 99% confidence level with n-1 degrees of freedom), or when available, multiplying by 5 the least sensitive method's estimated detection limit (EDL=concentration of compound yielding approximately a five to one signal to noise ratio or the calculated MDL, whichever is greater).

[b] Entry Point to the Distribution System. This sample collection location is located at the entry point, after treatment, that represents each non-emergency water source in routine use over the 12-month period of monitoring; sampling must occur at the EPTDS, unless the State has specified other sampling points that are used for compliance monitoring under 40 CFR 141.24(f)(1), (2) and (3) (§141.40(a)(5)). If monitoring at source (raw) water sampling points indicates detection of any of the contaminants on the UCMR (1999) monitoring list, then the system must change the location of its unregulated contaminant monitoring to the EPTDS (§141.40(a)(5)).

[c] MRL was established at a concentration, which is at least one-fourth the lowest known adverse health concentration, at which acceptable precision and accuracy has been demonstrated in spiked matrix samples.

[d] MRL for VOCs determined by multiplying by 10 either the published MDL or 0.5 μg/L, whichever is greater. The MDL of 0.5 μg/L (0.0005 mg/L) was selected to conform to the VOC MDL requirements of 40 CFR 141.24(f)(17)(i)(E).

1.3.2 UCMR (1999) List 2 and List 3 Contaminants

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1.4 Analytical Methods for UCMR (1999) List 1 Contaminants

While EPA has approved the use of alternative (i.e., non-EPA) analytical methods of the voluntary consensus standard organizations (including the ASTM, AOAC, and APHA) for the laboratory analysis of some UCMR (1999) List 1 contaminants, no alternative analytical methods have been approved for use in monitoring either acetochlor or perchlorate under the UCMR.

As discussed in the preamble to the Direct Final Rule approving the methods for acetochlor and perchlorate (65 FR 11371), no analytical method for the analysis of acetochlor has been published by the voluntary consensus standards bodies (ASTM, AOAC, or Standard Methods). In addition, no analytical method has been published by these organizations that is similar to EPA Method 525.2, and therefore, acetochlor cannot be added to the scope of any consensus organization's method.

The method that has been approved for use in monitoring perchlorate under the UCMR is a recently developed EPA Method. While this method is similar to methods that have been published by the California Department of Health and Dionex Corporation, neither of these methods incorporates a quality control element which assesses the impact of high concentrations of total dissolved solids (TDS), which are frequently present in water samples. The presence of high TDS in samples can result in inaccurate quantitation of perchlorate or may even mask its presence. Therefore, EPA incorporated a quality control element into EPA Method 314.0 that both identifies the presence of high concentrations of TDS and provides a mechanism to reduce their concentrations, thereby permitting accurate quantitation of perchlorate. In addition, EPA's Method 314.0 permits the use of both the California Department of Health and Dionex procedures within its scope; therefore, laboratories currently using either of these procedures can convert to EPA Method 314.0 simply by adopting the quality control procedures specified in EPA Method 314.0. None of the consensus methods organizations have published a method for the analyses of perchlorate.

EPA is specifying that only the analytical methods and procedures listed in Table 1.5s be used in obtaining data for the UCMR. Note, however, that whether EPA or one of the alternative methods are used, additional quality control measures for UCMR analyses are required (§141.40 Appendix A). The additional quality control measures are included in the revised UCMR (40 CFR 141.40) and are explained either in the UCMR QC Manual or in this Supplement. As in the QC Manual, EPA method numbers are used in the following sections of this Supplement as references for the reader. Subsequent sections of this QC Supplement provide supplemental information on methods, sampling, and quality control procedures to be used for monitoring acetochlor and perchlorate under the UCMR Assessment Monitoring program.

Table 1.5s Approved Analytical Methods for UCMR (1999) List 1 Contaminants

Chemical Contaminant	CAS #	Methodology	
		EPA Method	Equivalent Methods
Volatile Organic Compounds			
MTBE	1634-04-4	EPA 524.2 [a]	D5790-95 [b]; SM6210D [c]; SM6200B [c]
Nitrobenzene	98-95-3	EPA 524.2 [a, e]	D5790-95 [b]; SM6210D [c]; SM6200B [c]
Semivolatile Organic Compounds			
2,4-Dinitrotoluene	121-14-2	EPA 525.2 [a]	none identified
2,6-Dinitrotoluene	606-20-2	EPA 525.2 [a]	none identified
Chlorinated Hydrocarbon Pesticides			
DDE	72-55-9	EPA 525.2 [a]; EPA 508 [a]; EPA 508.1 [a]	D5812-96 [b]; 990.06 [d]
Nitrogen- and Phosphorus-Containing Pesticides			
Acetochlor	**34256-82-1**	**EPA 525.2 [a]**	**none identified**
EPTC	759-94-4	EPA 525.2 [a]; EPA 507 [a]	D5475-93 [b]; 991.07 [d]
Molinate	2212-67-1	EPA 525.2 [a]; EPA 507 [a]	D5475-93 [b]; 991.07 [d]
Terbacil	5902-51-2	EPA 525.2 [a]; EPA 507 [a]	D5475-93 [b]; 991.07 [d]
Acid Herbicides			
DCPA mono-acid degradate	887-54-7	EPA 515.1 [a, e]; EPA 515.2 [a, e]	D5317-93 [b]; 992.32 [d]
DCPA di-acid degradate	2136-79-0	EPA 515.1 [a, e]; EPA 515.2 [a, e]	D5317-93 [b]; 992.32 [d]
Inorganic Compounds			
Perchlorate	**14797-73-0**	**EPA 314.0 [f]**	**none identified**

[a] The version of the EPA methods approved for the UCMR are listed at 40 CFR 141.24 (e).

[b] *Annual Book of ASTM Standards*, 1996 and 1998, Vol. 11.02, American Society for Testing and Materials. Method D5812-96 is located in the *Annual Book of ASTM Standards*, 1998, Vol. 11.02. Methods D5790-95, D5475-93, and D5317-93 are located in the *Annual Book of ASTM Standards*, 1996 and 1998, Vol 11.02. Copies may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[c] SM 6200 B is only found in the 20th edition of *Standard Methods for the Examination of Water and Wastewater*, 1998. Sample preservation should be conducted as specified in EPA Method 524.2. SM 6210 D is only found in the 18th and 19th editions of *Standard Methods for the Examination of Water and*

Wastewater, 1992 and 1995, American Public Health Association; either edition may be used. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005.

d *Official Methods of Analysis of AOAC* (Association of Official Analytical Chemist) International, Sixteenth Edition, 4th Revision, 1998, Volume I, AOAC International, First Union National Bank Lockbox, PO Box 75198, Baltimore, MD 21275-5198. (800) 379-2622.

e EPA has included specific recommendations regarding the use of EPA Method 524.2 for measuring nitrobenzene and EPA Methods 515.1 and 515.2 for measuring the DCPA degradates in the *UCMR Analytical Methods and Quality Control Manual.*

f EPA Method 314.0 (EPA 815-B-99-003) has only recently been published. A copy of this method is available by contacting the EPA Safe Drinking Water Hotline at (800) 426-4791 or accessing the method directly at http://www.epa.gov/safewater/methods/sourcalt.html.

1.5 Laboratory Certification Requirements for UCMR (1999) List 1 Contaminants

As noted in the revised UCMR (64 FR 50556) and in the QC Manual, laboratories that provide data to the EPA in support of the UCMR must document that they are currently approved by a State and that they have State certification and/or approval to perform those analyses using UCMR-specified methods (§141.40(a)(5)(ii)(G)). Because EPA approved the use of a commonly used method for monitoring acetochlor under the UCMR, no performance testing sample analyses are required for laboratory approval for the analysis of acetochlor under the UCMR. All laboratories currently certified by their State to perform drinking water compliance monitoring using EPA Method 525.2 are automatically approved to perform acetochlor analyses under the UCMR.

However, the method that has been approved for monitoring perchlorate under the UCMR is a new method that was developed only recently by EPA. Because EPA Method 314.0 is a new method and includes matrix specific quality control criteria, laboratories must go through a separate approval process to analyze samples for perchlorate (§141.40(a)(5)(ii)(G)(ii)). The laboratory approval system is based on previous certification of the laboratory to conduct analyses in support of regulatory compliance monitoring of drinking water for any inorganic anion using an approved ion chromatographic method (as listed in §141.28, such as nitrate analysis by EPA Method 300.0). In addition, a laboratory must successfully complete the perchlorate Performance Testing (PT) Program to be approved for UCMR perchlorate analyses (§141.40(a)(5)(ii)(G)(ii)). This PT Program involves a blind control study, using a test sample with an unknown value.

Any laboratory wishing to participate in the perchlorate PT Program and obtain approval must submit a letter of request, to be received by EPA no later than March 31, 2000 (§141.40(a)(5)(ii)(G)(ii)). Any interested laboratory which does not meet this deadline or fails to successfully pass this initial PT study and still wishes to support this monitoring, will need to submit a request letter by October 6, 2000 in order to be eligible for a second PT study. EPA will not be able to consider any laboratory request letters received after October 6, 2000. Any laboratory gaining approval in the first PT study will not be required to participate in the second PT study. These will be the only two PT studies offered, through December 31, 2003, for laboratories wishing to gain approval to conduct perchlorate analysis in support of UCMR Assessment Monitoring. Any laboratory which does not request participation by October 6, 2000 and fails to pass either of these two PT studies will not be approved to support this perchlorate monitoring. The submitted request letter must be signed by the laboratory manager with a statement that the laboratory is currently certified, by the primacy agency in which the laboratory is located, to perform drinking water compliance monitoring using an approved ion chromatographic method (§141.40(a)(5)(ii)(G)(ii)). A copy of the letter or certificate issued by the State or primacy agency detailing this certification must also be submitted, along with a request letter containing the information listed below

(§141.40(a)(5)(ii)(G)(ii)). Details pertaining to laboratory certification can be found on-line at http://www.epa.gov/OGWDW/labcint.html.

The laboratory's request letter should include the following information:

1) Laboratory name

2) Complete laboratory mailing address

3) Ion chromatography analytical method the laboratory is certified to perform

4) A copy of the letter or certificate issued by the State or primacy agency which issued the certification to the laboratory

5) Contact person

6) Contact phone, facsimile, and e-mail (if available)

The letter should be mailed to:

Perchlorate PT Program Coordinator
U.S. EPA, MLK 140
26 W. Martin Luther King Dr.
Cincinnati, Ohio 45268

To participate successfully in this program the laboratory will also need to become proficient in the application of U.S. EPA Method 314.0, *Determination of Perchlorate in Drinking Water Using Ion Chromatography* (EPA 815-B-99-003). Laboratories must follow the procedure as well as all the quality control procedures prescribed in the method and outlined in this Supplement (§141.40(a)(5) and §141.40 Appendix A). To obtain a copy of EPA Method 314.0, contact the Safe Drinking Water Hotline at (800) 426-4791 or access an electronic copy of the method directly on-line at http://www.epa.gov/safewater/methods/sourcalt.html.

Upon successful completion of the perchlorate PT Program, EPA will provide each successful laboratory with an approval letter identifying the laboratory by name and the approval date. This letter may then be presented to any Public Water System (PWS) as evidence of laboratory approval for perchlorate analysis supporting the UCMR. Laboratory approval is retained as long as the laboratory maintains certification by the State or primacy agency in which the laboratory is located to perform drinking water compliance monitoring using an approved ion chromatographic method. If a laboratory maintains this certification, the laboratory approval for perchlorate analysis supporting the UCMR will be limited to the time period beginning on the date specified in the EPA issued approval letter and extending through January 28, 2004.

To allow data on perchlorate occurrence in public water systems obtained prior to January 2001 to be grandparented, the data must meet the reporting requirements of the UCMR which include the successful completion of the perchlorate PT Program by a laboratory approved to perform the original analyses (§141.35(g)).

Section 2. Sampling Plan

2.1 Monitoring By Public Water Systems

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2.1.1 Systems Required To Monitor

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2.2 Sampling Frequency

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2.3 Sampling Points

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Section 3. Sample Collection and Preservation

3.1 Chemical Contaminants

The sample collection, preservation, and holding time details for perchlorate are described in Section 3.1.6. Sampling procedures for acetochlor are identical to those listed in the QC Manual for EPA Method 525.2. For all UCMR (1999) List 1 contaminants, sample collection, preservation, and holding time details are summarized in Table 3.1s. The sample collection and preservation procedures as summarized below and in the QC Manual must be followed for all samples collected for the UCMR (§141.40 Appendix A). If these procedures are not followed, the Rule specifies that resampling is required within 14 days of the observance of the error (§141.40(a)(5)).

3.1.1 Nitrogen- and Phosphorus-Containing Pesticides

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3.1.2 Chlorinated Hydrocarbon Pesticides

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3.1.3 Acid Herbicides

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3.1.4 Volatile Organic Compounds

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3.1.5 Semi-volatile Organic Compounds

With the addition of acetochlor, there are seven UCMR (1999) List 1 semi-volatile organic compounds that may be analyzed with EPA Method 525.2. These compounds are: 2,4-dinitrotoluene, 2,6-dinitrotoluene, 4,4'-DDE[1], acetochlor, EPTC[2], molinate,[2] and terbacil.[2] Note that three of these semi-volatile organic compounds (2,4-dinitrotoluene, 2,6-dinitrotoluene, and acetochlor) may only be analyzed with the approved EPA analytical method; there are no approved

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1 This semi-volatile organic compound is a pesticide and is specifically identified and listed as a chlorinated pesticide in Table 1.5s.

2 These three semi-volatile organic compounds are pesticides and are specifically identified and listed as nitrogen/phosphorus pesticides in Table 1.5s.

equivalent methods for these three contaminants. See Table 1.5s for a full listing of the approved analytical methods for each contaminant.

The sampling procedures for contaminants to be monitored with EPA Method 525.2 remain unchanged from those listed in the QC Manual. For the general sampling procedures for this method, please refer to Section 3.1.5 of the QC Manual.

3.1.6 Inorganic Compounds

Perchlorate is the only inorganic chemical contaminant on List 1 of the UCMR (1999) List. Under the UCMR, perchlorate must be analyzed with EPA Method 314.0 (see Table 1.5s; §141.40(a)(5)). Sampling procedures based on EPA Method 314.0, including sample containers, chlorine testing, and sample collection, storage, and holding times are described below.

Sample containers - Use 30 milliliter, 125 milliliter, or 250 milliliter high density polyethylene (HDPE) or glass (amber or clear) bottles. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis and laboratory fortified matrix analysis (if required), and minimize waste disposal.

Residual chlorine determination - If water to be sampled is known or suspected to contain residual chlorine, determination of the level of residual chlorine is necessary. Diethyl-p-phenylenediamine (DPD) test-kits are commercially available and can be used to determine the level of residual chlorine in the field. Determine and record the level of residual chlorine in the water to be sampled. However, unlike all other samples collected for UCMR (1999) List 1 contaminants, samples collected for EPA Method 314.0 should *not* be dechlorinated, as chemicals typically used for dechlorination will likely interfere with perchlorate analyses.

Sample collection - When sampling from a water tap, remove any aeration equipment, open the tap and allow the system to flush until the water temperature has stabilized, usually about two minutes. Collect the sample directly from a tap and not through any plastic or rubber hoses or tubing. Adjust the flow from the tap to a slow but steady stream (about the diameter of a pencil) and collect the sample from the flowing stream. Fill sample to almost overflowing. After the sample bottle has been filled, close the sample bottle.

Sample storage - As described in EPA Method 314.0, samples do not need to be shipped iced or stored cold in a refrigerator, but every effort should be taken to protect the samples from temperature extremes. As all other UCMR (1999) List 1 samples must be refrigerated (§141.40(a)(5)), samples for EPA Method 314.0 may be placed with other UCMR samples on ice or with frozen cold packs in a cooler, or placed in a refrigerator that can maintain the samples at 4°C (± 2°). Samples should not be allowed to freeze, and should be protected from extreme temperatures from the time of collection until analysis.

Sample holding time - Analyze all samples within 28 days of collection (see Table 3.1s for a summary of holding times). If samples are not analyzed within this period, discard and replace the samples.

Table 3.1s Preservation and Holding Times for Approved Analytical Methods

Method(s)	Preservation	Sample Holding Time	Extract Holding Time	Sample Size	Container
EPA 314.0	**None**	**28 days**	**Not Applicable**	**30 mL, 125 mL, or 250 mL**	**HDPE or Glass (Amber or Clear)**
EPA 507 D5475-93 991.07	Sodium thiosulfate; Cool 4°C; Dark	14 days	14 days (4°C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 508 D5812-96 990.06	Sodium thiosulfate; Cool 4°C; Dark	7 days	14 days (4°C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 508.1 D5812-96 990.06	Sodium sulfite; 6 N HCl - pH < 2; Cool 4°C	14 days	30 days	1 L	Amber Glass with PTFE-lined Cap
EPA 515.1 D5317-93 992.32	Sodium thiosulfate; Cool 4°C; Dark	14 days	28 days (4°C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 515.2 D5317-93 992.32	Sodium thiosulfate; 6 N HCl - pH < 2; Cool 4°C; Dark	14 days	14 days (4°C, Dark)	250 mL	Amber Glass with PTFE-lined Cap
EPA 524.2 D5790-95 SM6210D SM6200B	Ascorbic acid or Sodium thiosulfate; 1:1 HCl - pH < 2; Cool 4°C	14 days	Not Applicable	40 - 120 mL	Glass with PTFE-lined Septum
EPA 525.2	Sodium sulfite; 6 N HCl - pH < 2; Cool 4°C; Dark	14 days	30 days from extraction	1 L	Amber Glass with PTFE-lined Cap

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

3.2 Monitoring of Routine Water Quality Parameters

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Section 4. Sample Transport

Sample transport requirements are generally the same as those described in the QC Manual. While EPA Method 314.0 states that samples collected for this method do not need to be shipped iced or stored cold in a refrigerator, to ensure adequate protection for other UCMR samples, all UCMR samples should be placed on ice or with frozen cold packs in an insulated container, cooler, or place in a refrigerator to cool the samples to 4°C (±2°). Keep the samples at 4°C (±2°) from the time of collection until analysis, including during any necessary transport. It is very important that no UCMR samples are allowed to freeze. In particular, EPA Method 314.0 indicates that samples should be protected from temperature extremes.

As stated in the QC Manual, all UCMR samples should be appropriately cooled and transported to the analytical laboratory as soon as possible after sample collection. Note that samples must be packed with sufficient ice or frozen cold packs to ensure that samples are maintained at 4°C (± 2°) during the entire transport period, but do not let any samples freeze (§141.40 Appendix A). All samples must be processed by the laboratory within 7 to 14 days of sample collection; therefore, provide adequate time for sample pick-up, transport, delivery, extraction, and analysis (§141.40 Appendix A). Immediate (sample collection day) transport of samples to the laboratory will ensure adequate sample analysis time and will greatly reduce the chance that systems will need to re-collect samples due to late samples exceeding holding times. Transporting the samples within 2 days of sample collection is strongly recommended; transporting the samples immediately—the same day of sample collection—is strongly advised.

Section 5. UCMR Quality Control Requirements

The specific quality control (QC) requirements for the use of EPA Method 525.2 for acetochlor and EPA Method 314.0 for perchlorate are described in Section 5 of this Supplement. With the exception of recommending specific ion peaks for the confirmation of acetochlor using EPA Method 525.2, the QC requirements for measuring acetochlor with this method are identical to those described previously in the *UCMR Analytical Methods and Quality Control Manual.*

While many of the QC requirements listed in EPA Method 314.0 are the same as those required for the UCMR, there are a few minor modifications that have been noted in this Supplement. In addition, QC requirements unique to EPA Method 314.0 are discussed in Section 5.10 of this Supplement.

As noted in the QC Manual, UCMR Assessment Monitoring must be conducted only using the analytical methods specified in the UCMR (see Table 1.5s; §141.40(a)(5)). The QC procedures specified in these approved analytical methods and in Appendix A of the Rule are further described in the QC Manual and this Supplement and must be followed to ensure accurate and precise data (§141.40 Appendix A).

Because EPA cannot accept monitoring data if the applicable QC requirements are not met, laboratories must strictly adhere to the QC described in this section (§141.40 Appendix A). The following failures (in addition to the those listed in the QC Manual) will cause monitoring data to be excluded from the database:

- when applicable, failure to meet the acceptance criteria for the instrument performance check
- when applicable, failure to meet the acceptance criteria for the laboratory fortified blank

5.1 Minimum Reporting Level

Minimum reporting level (MRL) concentrations are shown in Table 5.1s. The MRL concentrations for perchlorate and acetochlor were established at a concentration at which acceptable precision and accuracy has been demonstrated in spiked matrix samples and which is at least 1/4th the lowest known adverse health concentration. Please consult Section 5.1 of the QC Manual for the MRL determination process for all other contaminants listed in Table 5.1s.

Laboratories must demonstrate that they can achieve reliable data at the MRL for each contaminant. Therefore, the calibration curve must encompass the MRL concentration (§141.40 Appendix A). The laboratory must verify the accuracy of the curve at the MRL by analyzing a calibration check standard at the MRL concentration (see Section 5.2 of the QC Manual and Section 5.2 of this Supplement; §141.40 Appendix A).

Table 5.1s UCMR Methods and Minimum Reporting Levels

Contaminant	Approved UCMR Analytical Methods	Minimum Reporting Level
2,4-Dinitrotoluene	EPA 525.2	2 μg/L [a]
2,6-Dinitrotoluene	EPA 525.2	2 μg/L [a]
4,4'-DDE	EPA 508; EPA 508.1; EPA 525.2; D5812.96; 990.06	0.8 μg/L [a]
Acetochlor	**EPA 525.2**	**2 μg/L [b]**
DCPA mono- and di-acid degradates	EPA 515.1; EPA 515.2; D5317-93; 992.32	1 μg/L [a]
EPTC	EPA 507; EPA 525.2; D5475-93; 991.07	1 μg/L [a]
Molinate	EPA 507; EPA 525.2; D5475-93; 991.07	0.9 μg/L [a]
MTBE	EPA 524.2; D5790.95; SM6210D; SM6200B	5 μg/L [c]
Nitrobenzene	EPA 524.2; D5790.95; SM6210D; SM6200B	10 μg/L [c]
Perchlorate	**EPA 314.0**	**4 μg/L [b]**
Terbacil	EPA 507; EPA 525.2; D5475-93; 991.07	2 μg/L [a]

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

[a] Minimum reporting level (MRL) determined by multiplying by 10 the least sensitive method's minimum detection limit (MDL = standard deviation times the Student's t value for 99% confidence level with n-1 degrees of freedom), or when available, multiplying by 5 the least sensitive method's estimated detection limit (EDL=concentration of compound yielding approximately a five to one signal to noise ratio or the calculated MDL, whichever is greater).

[b] MRL was established at a concentration, which is at least 1/4th the lowest known adverse health concentration, at which acceptable precision and accuracy has been demonstrated in spiked matrix samples.

[c] MRL for VOCs determined by multiplying by 10 either the published MDL or 0.5 μg/L, whichever is greater. The MDL of 0.5 μg/L (0.0005 mg/L) was selected to conform to the VOC MDL requirements of 40 CFR 141.24(f)(17)(i)(E).

EPA recognizes that some laboratories are able to provide reliable data at concentrations lower than those shown in Table 5.1s. To achieve consistency in the National Drinking Water Contaminant Occurrence Database (NCOD), laboratories are only required to report quantitative results for concentrations equal to or greater than the MRLs (§141.35(d)). However, EPA also recognizes the usefulness of receiving more complete results to allow evaluation of the analytical method implementation. Systems and laboratory that are able to reliably report results below the MRL are

encouraged to do so. To report reliable results obtained below the MRL, laboratories and/or water systems may consult the instructions provided in Section 5.1 of the QC Manual.

5.2 Calibration

All methods specified in the UCMR, including EPA Method 314.0 and EPA Method 525.2, require that calibration span the expected concentration range of the samples being analyzed. The number of calibration standards necessary to meet this requirement varies from three to six, depending on the method. The UCMR does not require laboratories to change method calibration procedures with the exception that the low level standard must be at or below the MRL specified for each contaminant, and the mid-level standard must simply be near the midpoint of the calibration range (§141.40 Appendix A).

5.2.1 Calibration Verification

In general, the calibration verification specifications for the analysis of acetochlor with EPA Method 525.2 remain unchanged from those listed in the method and in the QC Manual. Tables 5.2s, 5.3s, and 5.4s provide a brief summary of these specifications.

As with all other methods approved for UCMR monitoring, complete calibration curves for EPA Method 314.0 are not required on a daily basis. However, to ensure accuracy of the analytical results, the calibration curve must be verified for each analysis batch (as described below; §141.40 Appendix A). The frequency requirements for verifying calibration are presented in Table 5.2s.

It is important to note that an analysis batch for EPA Method 314.0 is different than an analysis batch as defined for all other UCMR analyses in the QC Manual. For EPA Method 314.0, an analysis batch is defined as a sequence of samples which are analyzed within a 30-hour (rather than 24-hour) period and include no more than 20 (rather than 30) field samples.

The 30-hour period begins with the analysis of the instrument performance check (IPC) standard (see Section 5.10.2.2 of this Supplement), and ends with the analysis of the end calibration check standard. The 30-hour period does not necessarily include the analysis time used to generate the calibration curve. However, if a new curve is prepared each time samples are analyzed, the 30-hour period still begins with the analysis of the IPC standard.

EPA Method 314.0 specifies that the lowest level standard used in preparing the calibration curve must be used for the initial calibration check (§141.40 Appendix A). This initial calibration check standard (ICCS) must be verified prior to the analysis of any UCMR samples (§141.40 Appendix A). Acceptance criteria for the Method 314.0 ICCS and the low-level calibration check standards for all other UCMR methods are listed in Table 5.3s. If the criteria cannot be met, identify and eliminate the source of the problem, then perform a new instrument calibration according to the method calibration procedures.

For EPA Method 314.0, unlike all other methods currently approved for UCMR monitoring, EPA is requiring that the analyst alternate between mid- and high-level calibration check standards (rather than low- and mid- level standards) every 10 field samples (§141.40(a)(5)). Acceptance criteria for the mid-level calibration check standards are summarized in Table 5.4s. The high-level calibration

check standard specifications for EPA Method 314.0 are listed in Table 5.4.1s. If the acceptance criteria for any of these standards are not met, reanalyze all samples that were analyzed between this standard and the last standard meeting acceptance criteria after the calibration problem is resolved. As with all other methods approved for the UCMR, if the samples cannot be re-analyzed, then the data are considered invalid for those samples and should not be reported to EPA.

It is important to note that an acceptable end calibration check standard should be analyzed before analyses of other samples (i.e., non-UCMR samples) may begin (§141.40 Appendix A). Thus, if the last five samples analyzed were part of the UCMR, the analyst should perform an acceptable end calibration check standard before starting non-UCMR analyses.

Table 5.2s Frequency Requirements for Verifying Calibration

Methods	Method Specifications	UCMR Specifications	
		Initial	Continuing
EPA 314.0	**each analysis batch**	**each batch (not to exceed 20 samples) or daily (whichever is more frequent)**	**alternate between mid- and high-level standard after every 10 samples**
EPA 507 D5475-93 991.07	each working day	each batch (not to exceed 30 samples) or daily (whichever is more frequent)	alternate between low- and mid-level standard after every 10 samples
EPA 508 D5812-96 990.06	each working day		
EPA 508.1 D5812-96 992.32	each 12 hour shift		
EPA 515.1 D5317-93 992.32	each working day		
EPA 515.2 D5317-93 992.32	each working shift		
EPA 524.2 D5790-95 SM6210D SM6200B	every 12 hours		
EPA 525.2	every 12 hours		

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

Table 5.3s UCMR Low-Level Calibration Check Standard Concentrations and Acceptance Criteria

Contaminant	Minimum Reporting Level (MRL)	Concentration of Low-Level Standard	Acceptance Criteria
2,4-dinitrotoluene	2 µg/L	≤ MRL	± 40 %
2,6-dinitrotoluene	2 µg/L	≤ MRL	± 40 %
4,4'-DDE	0.8 µg/L	≤ MRL	± 40 %
Acetochlor	**2 µg/L**	**≤ MRL**	**± 40 %**
DCPA mono- and di-acid degradates	1 µg/L	≤ MRL	± 40 %
EPTC	1 µg/L	≤ MRL	± 40 %
Molinate	0.9 µg/L	≤ MRL	± 40 %
MTBE	5 µg/L	≤ MRL	± 40 %
Nitrobenzene	10 µg/L	≤ MRL	± 40 %
Perchlorate [a]	**4 µg/L**	**≤ MRL**	**± 25 %**
Terbacil	2 µg/L	≤ MRL	± 40 %

[a] For EPA Method 314.0, these specifications are only for the initial calibration check standard.

Table 5.4s Mid-Level Calibration Check Standard Concentrations and Acceptance Criteria

Contaminant	Mid-Level Standard	Acceptance Criteria
2,4-dinitrotoluene	middle of calibration range	± 20 %
2,6-dinitrotoluene	middle of calibration range	± 20 %
4,4'-DDE	middle of calibration range	± 20 %
Acetochlor	**middle of calibration range**	**± 20 %**
DCPA mono- and di-acid degradates	middle of calibration range	± 20 %
EPTC	middle of calibration range	± 20 %
Molinate	middle of calibration range	± 20 %

Contaminant	Mid-Level Standard	Acceptance Criteria
MTBE	middle of calibration range	± 20 %
Nitrobenzene	middle of calibration range	± 20 %
Perchlorate	**middle of calibration range**	**± 15 %**
Terbacil	middle of calibration range	± 20 %

Table 5.4.1s High-Level Calibration Check Standard Concentration and Acceptance Criteria for EPA Method 314.0

Contaminant	High-Level Standard	Acceptance Criteria
Perchlorate	**Highest-level calibration standard**	**± 15 %**

5.3 Method Detection Limit

Laboratories must calculate an MDL for acetochlor according to the procedure in CFR §136 Appendix B (included in the QC Manual as Appendix C; §141.40 Appendix A). Laboratories must calculate an MDL for perchlorate according to the procedure in CFR §136 Appendix B, but should extend this MDL determination beyond seven replicate analyses on a single day and instead conduct these seven replicate analyses over a period of three days, as specified in Method 314.0, Section 9.2.6 (§141.40(a)(5)).

5.4 Laboratory Reagent (Method) Blank

To determine if method contaminants or other interferences are present in the laboratory environment, the reagents, or the apparatus, all of the methods approved for the UCMR require periodic analysis of a laboratory reagent (method) blank (LRB).

The required frequencies for analyzing method blanks for the UCMR are listed in Table 5.5s (§141.40 Appendix A). For EPA Method 525.2, these specifications are the same as those described in the QC Manual. The specifications listed for EPA Method 314.0 are explained below.

For EPA Method 314.0, the method blank must be analyzed as the first sample on the instrument (immediately following the IPC standard; §141.40(a)(5)). This differs slightly from all other methods approved for use under the UCMR, as the method blank for EPA Method 314.0 is analyzed immediately before, rather than immediately after, the ICCS.

Consistent with the method blank specifications for all other UCMR-approved methods, if perchlorate is detected with EPA Method 314.0 at a concentration equal to or greater than one-half the MRL (see Table 5.6s), no further analyses should be performed until the source of the problem

is identified and eliminated. If the source is traced to any material that was used in the preparation of the set of samples to be analyzed, then discard all these prepared samples and repeat the preparation procedure using another aliquot of each sample. If another aliquot is not available, then consider all data invalid for all samples in the analysis batch and flag the monitoring data as not meeting QC criteria. Data not meeting UCMR QC criteria should not be reported to EPA.

For EPA Method 314.0, a second method blank may need to be prepared and analyzed if sample matrices have been pretreated to reduce the risk of high common anion interference. QC procedures related to the pretreatment of samples for EPA Method 314.0 are explained further in Section 5.10.2.3 of this Supplement. Analysis of a pretreated method blank is necessary to confirm that no background effects from the pretreatment process are present. If an analysis batch contains only pretreated samples, then only a pretreated method blank is required (§141.40(a)(5)).

Table 5.5s Frequency Requirements for Analyzing Laboratory Reagent (Method) Blanks

Method	Method Specifications	UCMR Specifications
EPA 314.0	**1 per analysis batch (≤20 samples) [a]**	**1 per analysis batch (≤20 samples) [a]**
EPA 507 D5475-93 991.07	1 per sample set or if reagents changed	1 per sample batch (≤20 samples)
EPA 508 D5812-96 990.06	1 per sample set or if reagents changed	1 per sample batch (≤20 samples)
EPA 508.1 D5812-96 992.32	1 per sample set per 12 hour shift	1 per sample batch (≤20 samples)
EPA 515.1 D5317-93 992.32	1 per sample set	1 per sample batch (≤20 samples)
EPA 515.2 D5317-93 992.32	1 per sample set	1 per sample batch (≤20 samples)
EPA 524.2 D5790-95 SM6210D SM6200B	each batch or 1 per 20 samples	1 per sample batch (≤20 samples)
EPA 525.2	every 12 hour extraction batch	1 per sample batch (≤20 samples)

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

[a] As required in EPA Method 314.0 and for the UCMR, a pretreated method blank may need to be prepared and analyzed if a sample matrix has been pretreated to reduce the risk of high common anion interference (§141.40(a)(5)).

Table 5.6s UCMR Acceptance Criteria for Laboratory Reagent (Method) Blanks

Contaminant	Minimum Reporting Level	Maximum Allowable Background Concentration (≤ ½ MRL)
2,4-Dinitrotoluene	2 μg/L	≤ 1 μg/L
2,6-Dinitrotoluene	2 μg/L	≤ 1 μg/L
4,4'-DDE	0.8 μg/L	≤ 0.4 μg/L
Acetochlor	**2 μg/L**	**≤ 1 μg/L**
DCPA mono- and di-acid degradates	1 μg/L	≤ 0.5 μg/L
EPTC	1 μg/L	≤ 0.5 μg/L
Molinate	0.9 μg/L	≤0.45 μg/L
MTBE	5 μg/L	≤ 2.5 μg/L
Nitrobenzene	10 μg/L	≤ 6 μg/L
Perchlorate	**4 μg/L**	**≤ 2 μg/L**
Terbacil	2 μg/L	≤ 1 μg/L

5.4.1 Field Reagent Blank (Shipping or Travel Blank)

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5.5 Quality Control Sample

For EPA Method 525.2, the specifications for the analysis of a quality control (QC) sample obtained from a source external to the laboratory are identical to those listed in the method and in the QC Manual.

For EPA Method 314.0, a QC sample must be analyzed after initially establishing or re-establishing a calibration curve or at least quarterly (§141.40 Appendix A). If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial demonstration of capability or continuing with on-going analyses (§141.40(a)(5)).

5.6 Sample Matrix Spike and Matrix Spike Duplicate

To provide data on the analytical precision and analytical accuracy of laboratories performing analyses for the UCMR, matrix spike (MS) and matrix spike duplicate (MSD) samples must be prepared and analyzed for all methods approved for the UCMR (§141.40 Appendix A). For information on calculating analytical precision and analytical accuracy (reported as data elements 14 and 15, respectively), as well as more general information on UCMR specifications for MS/MSD analyses, please refer to the QC Manual.

The frequencies required under the UCMR for the preparation and analysis of MS/MSD samples are listed in Table 5.7s (§141.40 Appendix A). For EPA Methods 314.0 and 525.2, laboratories are required to perform MS/MSD sample analyses on a minimum of 5% of the UCMR samples that are processed in each individual analysis batch (Method 314.0) or extraction batch (Method 525.2) (§141.40 Appendix A). This would represent 1 sample out of 20 as a minimum or at least one with each analysis batch. For methods that involve extractions, divide one sample from each extraction batch into two aliquots (one MS, one MSD), and spike each with a known concentration of the contaminants prior to extraction. Carry the entire set of unspiked sample and MS/MSD samples through the entire extraction and analysis process. For methods that do not involve extractions and for analysis batches of 20 or less, spike and analyze two aliquots of one of the UCMR samples in the batch. If the analysis batch contains more than 20 UCMR samples,[3] then divide, spike, and analyze two samples.

Laboratories must also choose a spiking concentration from one of the two concentrations listed in Table 5.8s (§141.40 Appendix A). As described in the QC Manual, the spiking concentration should be within ± 20% of one of the levels provided in the table. To determine precision data from laboratory MS/MSD samples at the MRL level and at a higher concentration, spike the samples at the concentrations listed in approximately a 50% ratio. For example, if a set of 40 samples are received, spike two aliquots of a sample from the first 20 samples with the low-level (± 20 % MRL) spike, and spike the MS/MSD for the second set of 20 samples with the mid-level spike.

As noted in Section 5.10.2.3 of this QC Supplement, if the conductivity of the sample matrix exceeds the matrix conductivity threshold (MCT) and pretreatment is required, a pretreated MS/MSD set must also be prepared and analyzed (§141.40(a)(5)). This pretreated MS/MSD set should be subjected to the same pretreatment techniques employed for the pretreated field samples.

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3 Note: A maximum of 20 samples can be included in an analysis batch for EPA Method 314.0.

Table 5.7s Frequency Requirements for Performing Spiked Sample Analyses

Method	Method Specifications	UCMR Specifications
EPA 314.0	**1 per 20 samples or analysis batch, whichever is more frequent**	**MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level**
EPA 507 D5475-93 991.07	1 per 20 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 508 D5812-96 990.06	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 508.1 D5812-96 992.32	1 per sample matrix	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 515.1 D5317-93 992.32	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 515.2 D5317-93 992.32	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 524.2 D5790-95 SM6210D SM6200B	Not required unless matrix effects suspected	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 525.2	1 per extraction batch, 1 per 20 samples	MS/MSD per 20 samples or per batch, whichever is more frequent[a]; alternate low-, mid-level

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

[a] For example, if a batch contains 20 or fewer samples, then 1 MS/MSD set must be analyzed for that batch. However, if a batch contains more than 20 samples, then at least two MS/MSD sets must be analyzed (§141.40 Appendix A). For EPA Method 314.0, a batch may not contain more than 20 samples.

Table 5.8s Concentrations for Spiking MS/MSD Samples

Contaminant	Low-Level Spike Concentration	Mid-Level Spike Concentration
2,4-Dinitrotoluene	2 μg/L ± 20 %	± 20% of mid-level standard
2,6-Dinitrotoluene	2 μg/L ± 20 %	± 20% of mid-level standard
4,4'-DDE	0.8 μg/L ± 20 %	± 20% of mid-level standard

Contaminant	Low-Level Spike Concentration	Mid-Level Spike Concentration
Acetochlor	**2 μg/L ± 20 %**	**± 20% of mid-level standard**
DCPA mono- and di-acid degradates	1 μg/L ± 20 %	± 20% of mid-level standard
EPTC	1 μg/L ± 20 %	± 20% of mid-level standard
Molinate	0.9 μg/L ± 20 %	± 20% of mid-level standard
MTBE	5 μg/L ± 20 %	± 20% of mid-level standard
Nitrobenzene	10 μg/L ± 20 %	± 20% of mid-level standard
Perchlorate	**4 μg/L ± 20 %**	**± 20% of mid-level standard**
Terbacil	2 μg/L ± 20 %	± 20% of mid-level standard

5.7 Internal Standard

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5.8 Surrogate Standard

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5.9 Confirmation

5.9.1 Gas Chromatographic Methods

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5.9.2 Gas Chromatography/Mass Spectrometry Confirmation

As described in the QC Manual, the UCMR requires confirmation of any organic contaminant detected above the MRL by Gas Chromatography/Mass Spectrometry (GC/MS) methods (§141.40 Appendix A). Laboratories retain the option of confirming the presence of an organic analyte using a second chromatography column prior to submitting the sample for GC/MS analyses, or going directly from the primary column analyses to GC/MS confirmation. If the organic contaminant detection is confirmed by the secondary column, then reconfirm by GC/MS using three specified ion peaks for contaminant identification. Recommended ion peaks for identification purposes are listed in Table 5.11s. Please refer to the QC Manual for additional specifications for GC/MS confirmation.

Table 5.11s Recommended Confirmation Ions

Contaminant	Recommended Confirmation Ions
2,4-dinitrotoluene	63, 89, 165
2,6-dinitrotoluene	63, 89, 165
4,4'-DDE	246, 316, 318
Acetochlor	**59, 132, 146**
DCPA dimethyl ester	299, 300, 302
EPTC	86, 128, 189
Molinate	83, 126, 187
MTBE	41, 57, 73
Nitrobenzene	51, 77, 123
Perchlorate	**Not Applicable**
Terbacil	116, 160, 161

Note: These ions are recommended for use in confirming all positive results. However, since mass spectrometers using different mass selection techniques may display spectra with different mass intensities, the analyst may choose alternate ions that better characterize the spectra displayed by their mass spectrometer.

5.9.3 Mass Spectrometry Methods

Identification and confirmation of acetochlor using EPA Method 525.2 must be performed as described in the method and the QC Manual (§141.40(a)(5)).

5.9.4 Ion Chromatography Identification

EPA Method 314.0 is the only ion chromatography method approved for the UCMR. To confirm perchlorate detection using this method, compare the retention time of a suspected perchlorate peak within the retention time window in the sample chromatogram to the actual retention time of a known analyte peak in a calibration standard. If the retention time of a suspect peak corresponds, within the acceptable limits, to the retention time in the daily calibration check standards, and the retention time is reproducible during the analysis batch, then identification is presumed positive.

The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards measured over several days. Three times the standard deviation of retention time may be used as a suggested window size but the retention time window should not extend beyond ± 5% of the retention time for perchlorate. Table 5.11.1s lists the estimated retention time for perchlorate that has been achieved by this method. However, the experience of the analyst should weigh heavily in the interpretation of these chromatograms.

Table 5.11.1s Estimated Retention Time for Perchlorate Using EPA Method 314.0	
Analyte	**Retention Time (minutes)**
Perchlorate	**10.1 ± 0.2**

If a low concentration of perchlorate is suspected in an unknown sample, but the retention time has drifted to the edge of the retention time window, a low-level perchlorate matrix spike should be prepared and analyzed to confirm the matrix-induced retention time shift.

Dilution of a sample may also be performed if more complete resolution is needed between a suspected perchlorate peak and a coeluting (i.e., shoulder) peak. If dilution is performed, the analyst should realize that the dilution will alter the MRL for that diluted sample analysis by a proportion equivalent to the dilution.

Dilution may also be necessary if a perchlorate response exceeds the highest calibration standard concentration. After conducting the analysis of the diluted sample, back calculate the actual field sample concentration by applying the correct dilution factor. Report only those values that fall between the MRL and the highest calibration standards. For information on reporting results below the UCMR perchlorate MRL of 4 μg/L, please refer to Section 5.1 of the QC Manual.

5.10 Additional Quality Controls

The requirements for rejecting samples for improper sample collection or shipment are unchanged from those described in the QC Manual. In addition, the laboratory must ensure each sample is analyzed within the required holding time listed in Table 5.12s (§141.40 Appendix A). When appropriate, EPA standardized the holding times across analytical methods for the same contaminant group.

Table 5.12s Maximum Holding Times for Samples and Extracts		
Approved Analytical Methods	**Sample**	**Extract**
EPA 314.0	**28 days**	**Not Applicable**
EPA 507 D5475-93 991.07	14 days	14 days
EPA 508 D5812-96 990.06	7 days	14 days
EPA 508.1 D5812-96 992.32	14 days	30 days

Approved Analytical Methods	Sample	Extract
EPA 515.1 D5317-93 992.32	14 days	28 days
EPA 515.2 D5317-93 992.32	14 days	14 days
EPA 524.2 D5790-95 SM6210D SM6200B	14 days	Not Applicable
EPA 525.2	14 days	30 days from extraction

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5s for the full reference for each analytical method.

5.10.1 Laboratory Fortified Blank

To ensure the quality of the solution used to prepare the MS/MSD samples, EPA Method 314.0 also requires the preparation and analysis of a laboratory fortified blank (LFB) (§141.40(a)(5)). This sample is prepared from the same secondary dilution stock used to fortify the MS/MSD samples. This secondary dilution stock should be prepared from the same stock used to prepare the calibration standards, and must not be prepared from an external source stock such as that used to prepare the QC sample (§141.40(a)(5)).

Laboratories are required to analyze a LFB (filtered, for particulate removal, as if it were a field sample) with each analysis batch immediately following the ICCS (§141.40(a)(5)). The LFB must be prepared with the same solution used to prepare the matrix spike and should be prepared at concentrations no greater than ten times the highest concentration observed in any field sample and should be varied to reflect the range of concentrations observed in field samples (§141.40(a)(5)).

If any deviations in the perchlorate secondary dilution stock concentration are present, it will be reflected in the LFB and not exclusively attributed to a matrix upon analysis of the matrix spike. Calculate accuracy as percent recovery (see Section 9.4.1.3 of EPA Method 314.0). The recovery for perchlorate must fall in the range of 85 - 115% prior to analyzing samples (§141.40(a)(5)). If the LFB recovery for an analysis batch does not meet these recovery criteria, the data are considered invalid, and the source of the problem should be identified and resolved before continuing analyses.

5.10.2 Matrix Conductivity Threshold Quality Control Requirements

One of the initial problems in the development of an analytical method for monitoring low-levels of perchlorate in drinking water was the interference caused by high background concentrations of total dissolved solids (TDS). To address this problem, EPA Method 314.0 incorporates several steps designed to keep this interference to a minimum. These steps are centered around a requirement of each laboratory to determine the matrix conductivity threshold (MCT) of their system during their

initial demonstration of capability (see Section 9.2.8 of EPA Method 314.0; §141.40(a)(5)). If a sample's measured conductivity exceeds this threshold, certain steps (either dilution or pretreatment of the sample) must be taken to ensure data quality (§141.40(a)(5)). Once a laboratory determines their MCT, they must also confirm that perchlorate can be recovered at the MRL (± 30%) from samples that have been prepared with a conductivity at the MCT (± 10% ; see Section 9.2.8.11 of EPA Method 314.0; §141.40(a)(5)).

5.10.2.1 Conductivity Meter Calibration Verification and Conductance Determination

Prior to analyzing a sample for perchlorate, the conductance of that sample matrix must be measured (§141.40(a)(5)). To ensure the accuracy of these measurements, the conductivity meter calibration must first be verified or established (as described in Section 10.4 of EPA Method 314.0) for each analysis batch (§141.40(a)(5)).

5.10.2.2 Instrument Performance Check

To verify the MCT as part of each an analysis batch, EPA Method 314.0 requires that an instrument performance check (IPC) standard be prepared and analyzed prior to the analysis of any other samples (including the method blank and the ICCS). The IPC should be prepared with a mixed common anion solution at the MCT (±10%) and spiked with a perchlorate (at a suggested level of 25 μg/L). As specified in the method, before any further analyses are conducted, the analyst must verify that:

1) the IPC solution conductance, measured with the calibrated conductivity meter, is within ±10% of original measured value (when the MCT was initially determined and the solution was originally prepared);

2) the area to height ratio percent difference ($PD_{A/H}$; see Section 9.2.8.8 of the method) for the observed perchlorate response in the IPC is less than 25%;

3) the level of perchlorate measured in the IPC is between 80 -120% of the spiked level; and

4) the shift in perchlorate retention time is less than 5%.

If the IPC fails to meet any of the above criteria, the source of the problem must be identified before sample analyses may begin (§141.40(a)(5)). As discussed in the method, if a laboratory frequently fails to meet these IPC criteria, it may be necessary for that laboratory to revise their MCT to a more appropriate lower level.

5.10.2.3 Additional Quality Control Procedures if Dilution or Pretreatment is Required

As mentioned in Section 5.10.2.1 above, before any field sample is analyzed, the conductance of the sample matrix must be measured (§141.40(a)(5)). If the conductivity of the sample matrix exceeds the MCT, then dilution or pretreatment of the sample is necessary.

If dilution is necessary, the sample should be diluted with reagent water by a factor large enough to ensure that the diluted sample conductivity is below the MCT. Dilution will also raise the required MRL for that sample by the same factor. If perchlorate is measured in the diluted sample above the elevated MRL, back calculate the actual field sample concentration and report the results.

If dilution does not provide the required results, pretreatment of the sample should be performed. Pretreatment is described in Section 11.1.4 of the method, and usually requires the addition of three pretreatment cartridges. If pretreatment is performed, EPA Method 314.0 requires that the following additional quality control samples must also be prepared:

1. a pretreated laboratory reagent (method) blank;
2. a pretreated laboratory fortified blank (LFB); and
3. a pretreated MS/MSD set.

All of these QC samples should be subject to the same pretreatment techniques employed for the pretreated field samples. It is important to note that the pretreated method blank and laboratory fortified blank (LFB) must be prepared and analyzed *before* any field samples are pretreated (§141.40(a)(5)). This is required to ensure that no background interference or bias is contributed by the pretreatment process. If background interference or bias is observed, the appropriate steps must be taken before field samples are pretreated (§141.40(a)(5)). These steps may include increasing the volume of rinse for the pretreatment columns. Once the interference has been eliminated and the pretreated method blank and LFB have met the appropriate acceptance criteria, the analyst may begin pretreating field samples as appropriate.

Section 6. Additional Analytical Method Specifications

6.1 Clarifications Concerning EPA Methods 515.1 and 515.2 and the Approved Equivalent Methods

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6.2 Recommendations for EPA Method 524.2 and the Approved Equivalent Methods

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Section 7. Reporting

7.1 Public Water Systems Reporting to EPA

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7.1.1 Reporting of Results Obtained for the DCPA Mono- and Di-Acid Degradates

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7.1.2 Reporting of Data Below the Specified Minimum Reporting Level

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7.1.3 Reporting of Water Quality Parameter Data

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7.1.4 Reporting of Matrix Conductance and Pretreated QC Data for Perchlorate

Each laboratory performing perchlorate analyses using EPA Method 314.0 will need to monitor and record the conductance of every matrix analyzed for perchlorate (§141.40(a)(5)(ii)(G). Furthermore, EPA Method 314.0 specifies that when field samples exceed the matrix conductivity threshold (MCT), and pretreatment is employed to reduce the high ionic character of the matrix, additional quality control samples must be analyzed (§141.40(a)(5)). These additional QC requirements are discussed in Section 5 of this Supplement and include a pretreated method blank, pretreated laboratory fortified blank (LFB), and a pretreated MS/MSD set for each pretreated matrix.

It is the responsibility of the laboratory to provide the matrix conductance data as well as these additional pretreated QC data to their client PWS and to retain these data in the laboratory's official analytical data archive. It should be clarified, however, that these results do not need to be reported to EPA since they were not promulgated as specific data reporting elements. The only case in which the reporting of these data would be required is the rare situation in which all the field samples in an analysis batch require pretreatment. In this case, the only LRB and LFB analyzed with that analysis batch might be the pretreated LRB and LFB, as specified in Sections 9.3.1.1 and 9.3.3.1 of EPA Method 314.0. When this occurs, those pretreated QC samples should be reported as the official LRB and LFB samples, and the first pretreated MS/MSD should be designated as the official MS/MSD for the analysis batch.

7.2 Public Notification of Results (Report of PWS to Consumers)

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Appendix A (revised)

Abbreviations and Acronyms

2,4-DNT	- 2,4-dinitrotoluene
2,6-DNT	- 2,6-dinitrotoluene
4,4'-DDE	- 4,4'-dichloro dichlorophenyl ethylene, a degradation product of DDT
Alachlor ESA	- alachlor ethanesulfonic acid, a degradation product of alachlor
AOAC	- Association of Official Analytical Chemists
APHA	- American Public Health Association
ASDWA	- Association of State Drinking Water Administrators
ASTM	- American Society for Testing and Materials
BGM	- Buffalo Green Monkey cells, a specific cell line used to grow viruses
CAS	- Chemical Abstract Service
CASRN	- Chemical Abstract Service Registry Number
CCL	- Contaminant Candidate List
CCR	- Consumer Confidence Reports
CERCLA	- Comprehensive Environmental Response, Compensation & Liability Act
CFR	- Code of Federal Regulations
CFU	- colony forming unit
CFU/mL	- colony forming units per milliliter
CWS	- community water system
DCPA	- dimethyl tetrachloroterephthalate, chemical name of the herbicide dacthal
DCPA mono- and di-acid degradates	- degradation products of DCPA
DDE	- dichloro dichlorophenyl ethylene, a degradation product of DDT
DDT	- dichloro diphenyl trichloroethane, a general insecticide
DNA	- deoxyribonucleic acid
EDL	- estimated detection limit
EPA	- Environmental Protection Agency
EPTC	- s-ethyl-dipropylthiocarbamate, an herbicide
EPTDS	- Entry Point to the Distribution System
ESA	- ethanesulfonic acid, a degradation product of alachlor
FACA	- Federal Advisory Committee Act
FTE	- full-time equivalent
GC	- gas chromatography, a laboratory method
GLI method	- Great Lakes Instruments method
GW	- ground water
GUDI	- ground water under the direct influence (of surface water)

HPLC	- high performance liquid chromatography, a laboratory method
ICCS	- initial calibration check standard
ICR	- Information Collection Request / Rule
IMS	- immunomagnetic separation
IPC	- instrument performance check
IRFA	- initial regulatory flexibility analysis
IRIS	- Integrated Risk Information System
IS	- internal standard
LFB	- laboratory fortified blank
LFM	- laboratory fortified matrix (matrix spike)
LLE	- liquid/liquid extraction, a laboratory method
MAC	- *Mycobacterium avium* complex
MOA	- Memorandum of Agreement
MCL	- maximum contaminant level
MCT	- matrix conductivity threshold
MDL	- method detection limit
MRL	- minimum reporting level
MS	- mass spectrometry, a laboratory method
MS	- sample matrix spike
MSD	- sample matrix spike duplicate
MTBE	- methyl tertiary-butyl ether, a gasoline additive
NAWQA	- National Water Quality Assessment Program
NCOD	- National Drinking Water Contaminant Occurrence Database
NDWAC	- National Drinking Water Advisory Council
NERL	- National Environmental Research Laboratory
NPS	- National Pesticide Survey
NTIS	- National Technical Information Service
NTNCWS	- non-transient non-community water system
NTTAA	- National Technology Transfer and Advancement Act
OGWDW	- Office of Ground Water and Drinking Water
OMB	- Office of Management and Budget
PAH	- Polycyclic aromatic hydrocarbon
PB	- particle beam
PBMS	- Performance-Based Measurement System
^{210}Pb	- Lead-210 (also Pb-210), a lead isotope and radionuclide; part of the uranium decay series
pCi/L	- picocuries per liter
PCR	- polymerase chain reaction
^{210}Po	- Polonium-210 (also Po-210), a polonium isotope and radionuclide; part of the uranium decay series
PT	- performance testing
PWS	- Public Water System
PWSF	- Public Water System Facility

QA	- quality assurance
QC	- quality control
RDX	- royal demolition explosive, hexahydro-1,3,5-trinitro-1,3,5-triazine
RFA	- Regulatory Flexibility Act
RPD	- relative percent difference
RSD	- relative standard deviation
SBREFA	- Small Business Regulatory Enforcement Fairness Act
SD	- standard deviation
SDWA	- Safe Drinking Water Act
SDWIS	- Safe Drinking Water Information System
SDWIS FED	- the Federal Safe Drinking Water Information System
SM	- Standard Methods
SMF	- Standard Compliance Monitoring Framework
SOC	- synthetic organic compound
SPE	- solid phase extraction, a laboratory method
SRF	- State Revolving Fund
STORET	- Storage and Retrieval System
SW	- surface water
TBD	- to be determined
TDS	- total dissolved solids
TNCWS	- transient non-community water system
UCM	- Unregulated Contaminant Monitoring
UCMR	- Unregulated Contaminant Monitoring Regulation/Rule
UMRA	- Unfunded Mandates Reform Act of 1995
USEPA	- United States Environmental Protection Agency
UV	- ultraviolet
VOC	- volatile organic compound
μg/L	- micrograms per liter

Appendix B (revised)

Definitions

Assessment Monitoring means sampling, testing, and reporting of listed contaminants that have available analytical methods and for which preliminary data indicate their possible occurrence in drinking water. Assessment Monitoring will be conducted for the UCMR (1999) List 1 contaminants.

Index Systems means a limited number of small CWSs and NTNCWSs, selected from the Assessment Monitoring systems in State Plans, that will be required to provide more detailed and frequent monitoring for the UCMR (1999) List 1 contaminants (§141.40(a)(6)). The Index Systems will be selected to geographically coincide with watersheds and areas studied under the United States Geological Survey's National Water Quality Assessment program. In addition to the reporting information required for Assessment Monitoring, the Index Systems must also report information on system operating conditions (such as water source, pumping rates, and environmental setting) (§141.40(a)(6)). These systems must monitor each year of the 5-year UCMR cycle, with EPA paying for all reasonable monitoring costs (§141.40(a)(4)(i)(A)). This more detailed and frequent monitoring will provide important information with which EPA can more fully evaluate the conditions under which small systems operate.

Listed contaminant means a contaminant identified as an analyte in Table 1, 141.40(a)(3) of the Unregulated Contaminant Monitoring Regulation (UCMR). To distinguish the current 1999 UCMR listed contaminants from potential future UCMR listed contaminants, all references to UCMR contaminant lists will identify the appropriate year in parenthesis immediately following the acronym UCMR and before the referenced list. For example, the contaminants included in the UCMR (1999) List include the component lists identified as UCMR (1999) List 1, UCMR (1999) List 2 and UCMR (1999) List 3 contaminants.

Listing cycle means the 5-year period for which each revised UCMR list is effective and during which no more than 30 unregulated contaminants from the list may be required to be monitored. EPA is mandated to develop and promulgate a new UCMR List every 5 years.

Monitored systems means all community water systems serving more than 10,000 people, and the national representative sample of community and non-transient non-community water systems serving 10,000 or fewer people that are selected to be part of a State Plan for the UCMR. (Note that for this round of Assessment Monitoring, systems that purchase their primary source of water are not included in the monitoring.)

Monitoring (as distinct from Assessment Monitoring) means all aspects of determining the quality of drinking water relative to the listed contaminants. These aspects include drinking water sampling and testing, and the reviewing, reporting, and submission to EPA of analytical results.

*Most vulnerable systems (*or *Systems most vulnerable)* means a subset of 5 to not more than 25 systems of all monitored systems in a State that are determined by that State in consultation with the EPA Regional Office to be most likely to have the listed contaminants occur in their drinking waters, considering the characteristics of the listed contaminants, precipitation, system operation, and environmental conditions (soils, geology and land use).

Pre-Screen Testing means sampling, testing, and reporting of the listed contaminants that may have newly emerged as drinking water concerns and, in most cases, for which methods are in an early stage of development. Pre-Screen Testing will be conducted by a limited number of systems (up to 200). States will nominate up to 25 of the most vulnerable systems per State for Pre-Screen Testing. The actual Pre-Screen Testing systems will be selected from the list of nominated systems through the use of a random number generator. Pre-Screen Testing will be performed to determine whether a listed contaminant occurs in sufficient frequency in the most vulnerable systems or sampling locations to warrant its being included in future Assessment Monitoring or Screening Surveys. Pre-Screen Testing will be conducted for the UCMR (1999) List 3 contaminants.

Random Sampling is a statistical sampling method by which each member of the population has an equal probability (an equal random chance) of being selected as part of a sample (the sample being a small subset of the population which represents the population as a whole).

Representative Sample (or *National Representative Sample*) means a small subset of all community and non-transient non-community water systems serving 10,000 or fewer people which EPA selects using a random number generator. The systems in the representative sample are selected using a stratified random sampling process that ensures that this small subset of systems will proportionally reflect (is "representative" of) the actual number of size- and water type-categories of all small systems nationally. In finalizing State Plans, a State may substitute a system from the replacement list for a system selected as part of the original representative sample, if a system on the representative sample list in the State Plan is closed, merged or purchases water from another system.

Sampling means the act of collecting water from the appropriate location in a public water system (from the applicable point from an intake or well to the end of a distribution line, or in some limited cases, a residential tap) following proper methods for the particular contaminant or group of contaminants.

Sampling Point means a unique location where samples are to be collected.

Screening Survey means sampling, testing, and reporting of the listed contaminants for which analytical methods are recently developed and have uncertain potential for occurrence in drinking water by a subset of approximately 300 systems from all monitored systems selected through use of a random number generator for public water system identification numbers. These systems must conduct the Screening Survey for the contaminants on UCMR (1999) List 2 as will be further described in the List 2 Rule (§141.40(a)(7)). Two Screening Surveys may be conducted for the UCMR (1999) List 2 contaminants.

State means, for the purposes of this section, each of the fifty States, the District of Columbia, U.S. Territories, and Tribal lands. For the national representative sample, Guam, the Commonwealth of Puerto Rico, the Northern Mariana Islands, the Virgin Islands, American Samoa, and the Trust Territories of the Pacific Islands are each treated as an individual State. All Tribal water systems in the U.S. which have status as a State under Section 1451 of the Safe Drinking Water Act for this program will be considered collectively as one State for the purposes of selecting a representative sample of small systems.

*State Monitoring Plan (*or *State Plan)* means a State's portion of the national representative sample of CWSs and NTNCWSs serving 10,000 or fewer people which must monitor for unregulated

contaminants (Assessment Monitoring, Screening Survey(s) and Index Systems) and all large systems (systems serving greater than 10,000 people) which are required to monitor for Screening Survey contaminants. A State Plan may be developed by a State's acceptance of EPA's representative sample for that State, or by a State's selection of systems from a replacement list for systems specified in the first list that are closed, are merged, or purchase water from another system. A State Plan also includes the process by which the State will inform each public water system of its selection for the plan and of its responsibilities to monitor. A State Plan will also include the systems required to conduct Pre-Screen Testing, selected from the State's designation of vulnerable systems. The State Plan may be part of the Partnership Agreement (PA) between the State and EPA.

Stratified Random Sampling is a procedure to draw a random sample from a population that has been divided into subpopulations or strata, with each stratum comprised of a population subset sharing common characteristics. Random samples are selected from each stratum proportional to that stratum's proportion of the entire population. The aggregate random sample (compiled from all the strata samples) provides a random sample of the entire population that reflects the proportional distribution of characteristics of the population. In the context of the UCMR, the population served by public water systems was stratified by size (with size categories of 500 or fewer people served, 501 to 3,300 people served, and 3,301 to 10,000 people served) and by water source type supplying the water system (ground water or surface water). This stratification was done to ensure that systems randomly selected as nationally representative sample systems would proportionally reflect the actual number of size and water type categories nationally.

Testing means, for the purposes of the UCMR and distinct from *Pre-Screen Testing*, the submission and/or shipment of samples following appropriate preservation practices to protect the integrity of the sample; the chemical, radiological, physical and/or microbiological analysis of samples; and the reporting of the sample's analytical results for evaluation. Testing is a subset of activities defined as *monitoring*.

Unregulated contaminants means chemical, microbiological, radiological and other substances that occur in drinking water or sources of drinking water that are not currently regulated under the federal drinking water program. EPA has not issued standards for these substances in drinking water (i.e., maximum contaminant levels or treatment technology requirements). EPA is required by Congress to establish a program to monitor for selected unregulated contaminants in public water systems to determine whether they should be considered for future regulation to protect public health. The selected contaminants are listed in 141.40(a)(3), Table 1, the UCMR List.

Vulnerable time (or *vulnerable period*) means the time (or, in some cases, the 3-month quarter) of the year determined as the most likely to have the listed group of contaminants present at their highest concentrations or densities in drinking water. The vulnerable determination, in the case of the UCMR, is made by the EPA or by the State (under arrangement with the EPA) for a system, subset of systems, or all systems in a State. The vulnerable determination is based on characteristics of the contaminants, precipitation, system operations, and environmental conditions such as soil types, geology, and land use. This determination does not indicate or imply that the listed contaminants will be identified in the drinking water with certainty, but only that sampling conducted during the vulnerable period presumably has the highest likelihood of identifying those contaminants in higher concentrations relative to other sampling times of the year, if and when the contaminants occur.