United States
Environmental Protection
Agency

Office of Water
4607

EPA -815-R-01-028
December 2001

UCMR (1999) List 1 and List 2 Chemical Analytical Methods and Quality Control Manual

Printed on Recycled Paper

Foreword

This document provides guidance regarding sampling, analytical methods, and related quality control issues for the States, EPA offices, water systems, and analytical laboratories participating in the Unregulated Contaminant Monitoring Rule (UCMR) Program. It replaces the previously issued UCMR Analytical Methods and Quality Control Manual (EPA 815-R-00-006, March 2000) and the Supplement A to the UCMR Analytical Methods and QC Manual (EPA 815-R-00-002, March 2000). This document is 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 additional or replacement pages to this Analytical Methods and Quality Control Manual. 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 additions to this Manual.

Under the Safe Drinking Water Act (SDWA) as amended in 1996, §1445(a)(2)(A), 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 that began 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.

The SDWA provisions and EPA regulations described in this document contain legally binding requirements. This document does not substitute for those provisions or regulations, nor is it a regulation itself. It does not impose legally-binding requirements on EPA, States, or the regulated community, and may not apply to a particular situation based upon the circumstances. EPA and State decision makers retain the discretion to adopt approaches on a case-by-case basis that differ from this guidance where appropriate. Any decisions regarding a particular facility will be made based on the applicable statutes and regulations. Therefore, interested parties are free to raise questions and objections about the appropriateness of the application of this manual to a particular situation, and EPA will consider whether or not the recommendations or interpretations in the manual are appropriate in that situation based on the law and regulations. EPA may change this manual in the future without notice or an opportunity for comment.

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

This guidance document has been prepared based on the revised UCMR regulation and the specifications found in the identified approved analytical methods. The following *Federal Register* (*FR*) publications, which will all be incorporated into 40 CFR parts 141.35 and 141.40, are frequently referenced throughout this manual.

- *Revisions to the Unregulated Contaminant Monitoring Regulation for Public Water Systems; September 17, 1999 (64 FR 50556)*
- *Unregulated Contaminant Monitoring Regulation for Public Water Systems; Analytical Methods for Perchlorate and Acetochlor; Announcement of Laboratory Approval and Performance Testing (PT) Program for the Analysis of Perchlorate; March 2, 2000 (65 FR 11372)*
- *Unregulated Contaminant Monitoring Regulation for Public Water Systems; Analytical Methods for List 2 Contaminants; Clarifications to the Unregulated Contaminant Monitoring Regulation; January 11, 2001 (66 FR 2273)*
- *Unregulated Contaminant Monitoring Regulation for Public Water Systems; Amendment to the List 2 Rule and Partial Delay of Reporting of Monitoring Results; September 4, 2001 (66 FR 46221)*

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Section 1. Introduction

1.1 Background on the Unregulated Contaminant Monitoring Rule (UCMR)

The requirement to monitor unregulated contaminants was established by the 1986 Amendments to the Safe Drinking Water Act (SDWA). Public water systems (PWSs) were required to report the monitoring results for up to 48 unregulated contaminants to the States or primacy agency under several regulations (40 CFR 141.40(e), (j), and (n)(11) and (12)). Systems with less than 150 service connections were exempt, provided those systems made their facilities available for the States to monitor. The rules required repeat monitoring every 5 years.

Under §1445(a)(2)(A) of the SDWA, as amended in 1996, the Environmental Protection Agency (EPA) was to promulgate regulations substantially revising the previous unregulated monitoring program. The revised Unregulated Contaminant Monitoring Rule (UCMR) Program has a new list of contaminants, changes the PWSs that must conduct monitoring, and changes the frequency and schedule for monitoring (§141.40(a)).

The UCMR was developed in coordination with the Contaminant Candidate List (CCL) and the National Drinking Water Contaminant Occurrence Database (NCOD). The UCMR and the CCL will operate on an evolving 5-year cycle to assess the impact of new drinking water contaminants. The data collected through the UCMR Program will be used to support the development of the subsequent CCL, to support the Administrator's determination of whether or not to regulate a contaminant, and to develop regulations. The revised monitoring program is part of a sound science approach to future drinking water regulation, which is an aim of the 1996 SDWA Amendments.

The revised UCMR includes new and emerging contaminants. The revised UCMR also requires fewer systems to conduct monitoring than was required in the previous unregulated contaminant monitoring program (§141.40(a)(1)). Therefore, the quality of data collected is a very important issue for the success of the revised program. This document provides a brief overview of the revised UCMR and outlines the required analytical methods and quality control procedures that PWSs and participating laboratories must adhere to while implementing the Assessment Monitoring and chemical Screening Survey component of the UCMR (§141.40(a)(5) and §141.40 Appendix A). This manual does not address the method or monitoring requirements for monitoring the List 2, microbiological contaminant *Aeromonas*.

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. These documents are available from the EPA Water Docket, (202) 260-3027, Docket Number W-98-02. General information can also 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 www.epa.gov/safewater/ucmr.html.

1.2 The Unregulated Contaminant Monitoring Rule

The UCMR is required by the SDWA as amended in 1996. Under the 1996 Amendments, EPA was required to promulgate a new regulation for monitoring unregulated contaminants. The regulation must include: (1) a new list of contaminants, of which not more than 30 may be required for monitoring, (2) a frequency and schedule for monitoring based on PWS size, source water type, and likelihood of finding contaminants; (3) monitoring of only a representative sample of PWSs serving 10,000 or fewer people; and, (4) requirements for placement of the monitoring data in the NCOD (in accordance with §1445 (g) of SDWA). PWSs must monitor to provide the location, concentration, and related information regarding the occurrence of these contaminants in public drinking water (§141.35(d)). EPA will analyze the monitoring data to identify which contaminants occur nationally and at concentrations that may warrant regulation. EPA will determine which contaminants pose the greatest risks to human health and, if necessary, will set priorities for regulation of the contaminants. Conversely, EPA may remove contaminants from consideration for regulation if UCMR monitoring indicates that these contaminants are not detected at significant levels in drinking water. EPA was required to develop a list of contaminants, the UCMR (1999) List, and regulations for monitoring the contaminants by August 1999. This list will be revised every 5 years.

EPA used the CCL (1998) contaminants listed as occurrence priorities as the primary basis for selecting contaminants for the UCMR (1999) List. The CCL identifies contaminants of potential concern that may occur or are likely to occur in drinking water. To establish the CCL (1998), EPA convened a Work Group to develop the CCL based on the results of previous unregulated contaminant monitoring and information from other data sources. The CCL team worked from a compendium of 10 lists containing approximately 391 chemical contaminants. The lists used in this process were: 1991 Drinking Water Priority List; Health Advisories; Integrated Risk Information System; Non-Target Analytes in Public Water Supply Samples; Comprehensive Environmental Response, Compensation, and Liability Act Priority List; stakeholder responses; Toxic Release Inventory; pesticides identified by the EPA Office of Pesticide Programs; a list of contaminants identified by the Safe Drinking Water Hotline; and a list of contaminants suspected of causing endocrine disruption.

The National Drinking Water Advisory Council's Working Group on Contaminant Occurrence and Selection, formed under the Federal Advisory Committee Act, developed the criteria for the CCL to address a contaminant's potential risk to public health and frequency of contaminant occurrence. Criteria for selecting contaminants for the CCL focused on occurrence in water at levels of health concern or indications of occurrence (production and release, coupled with contaminant properties). Health effects concentrations were used to determine the significance of occurrence. The CCL (1998) contains 50 chemical contaminants and 10 microbiological contaminants.

In establishing the CCL (1998), EPA divided the contaminants into three priority categories: those contaminants requiring additional research; those which need additional occurrence data; and those which are priorities for consideration for rule-making. EPA published a draft of the 1998 Drinking Water Contaminant Candidate List in the October 6, 1997 *Federal Register* (62 *FR* 52193). Comments submitted in response to the draft CCL were reviewed and considered in creating the final CCL, which was published in the March 2, 1998 *Federal Register* (63 *FR* 10273).

For purposes of the UCMR, EPA initially used the CCL occurrence priorities list to identify contaminants that were of national concern. The UCMR (1999) List, as initially proposed, included 32 of the 34 contaminants listed as occurrence priorities on the CCL (1998). At the time of the publication of the proposed UCMR, perchlorate and RDX were excluded from the UCMR (1999) List because it was thought that their occurrence was only a localized issue. As more data became available and after many public comments were received supporting the inclusion of these compounds, both perchlorate and RDX, as well as lead-210 and polonium-210, were added to the UCMR (1999) List.

1.3 Contaminants on the UCMR (1999) List

Although only 32 contaminants (24 chemical and 8 microbiological contaminants) listed on the CCL as occurrence priorities were initially proposed for inclusion on the UCMR (1999) List, all 34 contaminants listed as occurrence priorities on the CCL (1998) were eventually included on the final UCMR (1999) List. Two additional contaminants, lead-210 and polonium-210, were not included on the CCL (1998), but have been found in drinking water and in shallow aquifers in Florida. Because radionuclides have potential wide occurrence and consequent health risks and in response to public comments, EPA added lead-210 and polonium-210 to the UCMR (1999) List. These 36 contaminants comprise the list of UCMR (1999) contaminants (Table 1.1). For each of these contaminants, EPA evaluated the availability of analytical methods published by EPA and voluntary consensus standard organizations such as the American Society for Testing and Materials (ASTM), the Association of Official Analytical Chemists (AOAC) and the American Public Health Association (APHA). In addition, EPA prioritized analytical methods development activities for those chemical and microbiological contaminants that did not have suitable analytical methods currently available.

The revised UCMR Program consists of three distinct monitoring components based upon the availability of suitable analytical methods. The Assessment Monitoring component of the UCMR Program will monitor for UCMR (1999) List 1 contaminants; these are the UCMR contaminants for which analytical methods are currently available.

The UCMR (1999) List 2 contaminants are part of the Screening Survey which is divided into two parts: a first Screening Survey for chemical contaminants (15 chemical contaminants), and a second Screening Survey for the microbial contaminant *Aeromonas*, all of which have uncertain potential for occurrence. Only 13 of the 15 chemical contaminants on List 2 for which analytical methods have been developed are included in the chemical Screening Survey. The analytical method for *Aeromonas* was finalized in November 2001 and, pending subsequent promulgation, will be the approved method for this monitoring in 2003.

The Pre-Screen Testing component of the UCMR Program will monitor for UCMR (1999) List 3 contaminants for which analytical methods are in the early stages of development, and which may have newly emerged as concerns.

Table 1.1 UCMR (1999) Contaminants

Chemical Contaminants		
List	***Contaminant Name***	***Potential Environmental Source***
1	2,4-Dinitrotoluene	Used in the production of isocyanate and explosives
1	2,6-Dinitrotoluene	Used as mixture with 2,4-DNT (similar uses)
1	Acetochlor	Herbicide used with cabbage, citrus, coffee, and corn crops
1	DCPA di-acid degradate	Degradation product of DCPA, an herbicide used on grasses and weeds with fruit and vegetable crops
1	DCPA mono-acid degradate	Degradation product of DCPA, an herbicide used on grasses and weeds with fruit and vegetable crops
1	4,4'-DDE	Degradation product of DDT, a general insecticide
1	EPTC (s-ethyl-dipropylthio-carbamate)	Herbicide used on annual grasses, weeds, with potatoes and corn
1	Molinate	Selective herbicide used with rice, controls watergrass
1	MTBE (methyl tertiary-butyl ether)	Octane enhancer in unleaded gasoline
1, 2	Nitrobenzene	Used in the production of aniline, which is used to make dyes, herbicides, and drugs
1	Perchlorate	Oxygen additive in solid fuel propellant for rockets, missiles, and fireworks
1	Terbacil	Herbicide used with sugarcane, alfalfa, and some fruit, etc.
2	1,2-Diphenylhydrazine	Used in the production of benzidine and anti-inflammatory drugs
2	2-Methyl-phenol	Released in automobile and diesel exhaust, coal tar and petroleum refining, and wood pulping
2	2,4-Dichlorophenol	Chemical intermediate in herbicide production
2	2,4-Dinitrophenol	Released from mines, metal, and petroleum plants
2	2,4,6-Trichlorophenol	By-product of fossil fuel burning, used as bactericide and wood/glue preservative
2	Alachlor ESA and other acetanilide degradation products	Degradation product of alachlor, an herbicide used with corn, bean, peanut, and soybean crops to control grasses and weeds
2	Diazinon	Insecticide used with rice, fruit, vineyards, and corn crops
2	Disulfoton	Insecticide used with cereal, cotton, tobacco, and potato crops
2	Diuron	Herbicide used on grasses in orchards and with wheat crops

List	*Contaminant Name*	*Potential Environmental Source*
2	Fonofos	Soil insecticide used on worms and centipedes
2	Linuron	Herbicide used with corn, soybean, cotton, and wheat crops
2	Prometon	Herbicide used on annual and perennial weeds and grasses
2	RDX	Used in explosives; ammunition plants
2	Terbufos	Insecticide used with corn, sugar beet, and grain sorghum crops
3	Lead-210	Part of the uranium decay series; naturally occurring
3	Polonium-210	Part of the uranium decay series; naturally occurring
Microbiological Contaminants		
2	*Aeromonas*	Present in all freshwater and brackish water
3	Adenoviruses	Fecal or hand to mouth transmission
3	Cyanobacteria (blue-green algae, other freshwater algae, and their toxins)	Bloom in surface water bodies; produce toxins
3	Caliciviruses	Contaminated food and water; raw shellfish
3	Coxsackieviruses	Fecal or hand to mouth transmission
3	Echoviruses	Fecal or hand to mouth transmission
3	*Helicobacter pylori*	Fecal or hand to mouth transmission
3	Microsporidia	Occur in rivers, ponds, lakes, and unfiltered water

Note: UCMR (1999) List 1 and List 2 chemical contaminants require monitoring under the Assessment Monitoring and Chemical Screening Survey component, respectively, of the revised UCMR (§141.40(a)(3)). The method for *Aeromonas* is currently being finalized. Following method finalization and promulgation, quality control and methodology will be clarified in a microbial methods manual. EPA is conducting analytical methods development for UCMR (1999) List 3 contaminants. When methods for these contaminants are ready for use, EPA will issue supplements to this Analytical Methods and Quality Control Manual. For more information on the Assessment Monitoring, Screening Surveys, and Pre-Screen Testing components of the UCMR, the reader may refer to the proposed UCMR Preamble and Rule (64 *FR* 23398), the proposed UCMR List 2 Preamble and Rule (65 *FR* 55362) or the final UCMR Rule (64 *FR* 50556) and final UCMR List 2 Rule (66 *FR* 2273).

Because of the evolving nature of the UCMR Program, the specific analytical and technical requirements for monitoring contaminants may change with supplemental rule-making. When analytical methods and quality control details for the UCMR (1999) List 2 Screening Survey for *Aeromonas* and List 3 contaminant monitoring are developed and approved, additional or replacement pages for this manual will be issued. 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 additions to this Manual.

A more complete review of methods availability is summarized in the proposed UCMR Preamble and Rule (64 *FR* 23398), the proposed UCMR List 2 Preamble and Rule (65 *FR* 55362), the final UCMR Rule (64 *FR* 50556) and the final UCMR List 2 Rule (66 *FR* 2273), as well as the *Contaminant Selection, Methods, and Sampling: Technical Background Information for the UCMR* (EPA 815-R-99-007). (This background document and other UCMR supporting documents are available from the EPA Water Docket, (202) 260-3027, Docket Number W-98-02. General information can also 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 www.epa.gov/safewater/ucmr.html.) For identification of terms used throughout this Manual, see Appendix A (for a list of abbreviations and acronyms) and Appendix B (for a list of definitions).

1.3.1 UCMR (1999) List 1 Contaminants

With the publication of the Direct Final Rule (65 *FR* 11371), EPA approved analytical methods for acetochlor and perchlorate, thus completing methods approval 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 1.2 (§141.40(a)(5)).

Table 1.2 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, D 5812-96, 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, EPA 515.3, EPA 515.4, D 5317-93, 992.32	1 µg/L [a]	EPTDS [b]
DCPA di-acid degradate	2136-79-0	EPA 515.1, EPA 515.2, EPA 515.3, EPA 515.4, D 5317-93, 992.32	1 µg/L [a]	EPTDS [b]
EPTC	759-94-4	EPA 507, EPA 525.2, D 5475-93, 991.07	1 µg/L [a]	EPTDS [b]
Molinate	2212-67-1	EPA 507, EPA 525.2, D 5475-93, 991.07	0.9 µg/L [a]	EPTDS [b]

Contaminants	CAS #	Approved Analytical Methods	Minimum Reporting Level	Sampling Point
MTBE	1634-04-4	EPA 502.2, EPA 524.2, D 5790-95, SM 6210D, SM 6200B, SM 6200C	5 µg/L [d]	EPTDS [b]
Nitrobenzene	98-95-3	EPA 524.2, D 5790-95, SM 6210D, SM 6200B	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, D 5475-93, 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 1.5 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

The UCMR (1999) List 2 contaminants and their corresponding required sampling locations, suitable EPA analytical methods, and other related analytical details are listed in Table 1.3 (§141.40 (a)(3)). There are a total of 16 contaminants; 15 organic chemicals and 1 microorganism (*Aeromonas*). Nitrobenzene has been added to List 2 from the original UCMR (1999) List to track its occurrence at a concentration lower than the List 1 nitrobenzene minimum reporting level (MRL). Polonium-210 has been moved from List 2 to List 3 because further analytical method research and development is needed. Methods for the analysis of RDX and Alachlor ESA and other acetanilide degradation products need further refinement before approval. When methods for these contaminants are approved and ready for use, EPA will issue an additional supplement. Monitoring for the 13 organic chemicals with approved analytical methods is scheduled to begin in 2001 for the representative sample of small systems (systems serving 10,000 persons or less) and 2002 for large systems (serving greater than 10,000 persons).

Table 1.3 UCMR (1999) List 2 Contaminants

Contaminant	CAS #	Analytical Methods	Minimum Reporting Level	Sampling Point
1,2-Diphenylhydrazine	122-66-7	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
2-Methyl-phenol	95-48-7	EPA Method 528	1.0 µg/L [a]	EPTDS [b]
2,4-Dichlorophenol	120-83-2	EPA Method 528	1.0 µg/L [a]	EPTDS [b]
2,4-Dinitrophenol	51-28-5	EPA Method 528	5.0 µg/L [a]	EPTDS [b]
2,4,6-Trichlorophenol	88-06-2	EPA Method 528	1.0 µg/L [a]	EPTDS [b]
Alachlor ESA and other acetanilide degradation products	NA [d]	Reserved [c]	Reserved [c]	Reserved [c]
Diazinon	333-41-5	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
Disulfoton	298-04-4	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
Diuron	330-54-1	EPA Method 532	1.0 µg/L [a]	EPTDS [b]
Fonofos	944-22-9	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
Linuron	330-55-2	EPA Method 532	1.0 µg/L [a]	EPTDS [b]
Nitrobenzene (low level)	98-95-3	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
Prometon	1610-18-0	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
RDX	121-82-4	Reserved [c]	Reserved [c]	Reserved [c]
Terbufos	13071-79-9	EPA Method 526	0.5 µg/L [a]	EPTDS [b]
Aeromonas	N/A [d]	Reserved [c]	Reserved [c]	Distribution System

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method.

[a] Minimum Reporting Level represents the value of the lowest concentration precision and accuracy determination made during methods development and documented in the method. If method options are permitted, the concentration used was for the least sensitive option.

[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, source water sampling points are not permitted for List 2 contaminant monitoring (§141.40(a)(5)).

[c] To be determined. Neither approved method, MRL, nor monitoring period (except for *Aeromonas*) has been defined for these contaminants.

[d] CAS number is Not Applicable.

1.3.3 UCMR (1999) List 3 Contaminants

Currently, there are no suitable analytical methods for the UCMR (1999) List 3 contaminants. Therefore, monitoring is not currently required for these contaminants, but will be required in the future as analytical methods are developed and finalized. Table 1.4 contains the current UCMR (1999) List 3 contaminants, including polonium-210. These contaminants are scheduled to be monitored during the Pre-Screen Testing component of the UCMR Program, which may be conducted in 2004, exclusively at selected List 3 systems if methods are available.

Table 1.4 UCMR (1999) List 3 Contaminants

Contaminant	CAS #	Anticipated Analytical Method	Minimum Reporting Level	Anticipated Sampling Point
Lead-210	14255-04-0	Reserved [a]	Reserved [a]	Reserved [a]
Polonium-210	13981-52-7	Reserved [a]	Reserved [a]	Reserved [a]
Adenoviruses	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Caliciviruses	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Coxsackieviruses	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Cyanobacteria (blue green algae, other freshwater algae, and their toxins)	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Echoviruses	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Helicobacter pylori	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]
Microsporidia	NA [b]	Reserved [a]	Reserved [a]	Reserved [a]

[a] To be determined. Neither approved method, MRL, nor monitoring period has been defined for these contaminants.

[b] CAS number is Not Applicable.

1.4 Analytical Methods for UCMR (1999) List 1 Contaminants

Table 1.5 includes the UCMR (1999) List 1 contaminants and related analytical methods that are required for monitoring under the Assessment Monitoring component of the revised UCMR (§141.40(a)(3)). The contaminants listed in this table are grouped according to compound characteristics. The purpose of the revised UCMR is to obtain contaminant occurrence data in support of future regulatory decisions. The data required for regulatory decision-making must be of high quality. Most analytical methods are subject to some degree of false-negative test results (not detecting a contaminant when it is present), false-positive test results (either incorrectly

identifying or detecting a contaminant, or introducing a contaminant into a sample when it is not present), and errors in the accuracy and precision of quantitative results.

The data quality needs of drinking water compliance monitoring differ from the evaluation and use of occurrence data. The purpose of compliance monitoring is to determine whether or not a contaminant is present in the drinking water above the established maximum contaminant level (MCL). Unless the concentration of the contaminant closely approaches the MCL, even imprecise data can be used to assure the data user that the contaminant is not present at a concentration above the MCL. In contrast, the usefulness of occurrence data is much more dependent on the precision of the measurement. The ability to perform valid and meaningful statistical analyses is directly dependent on the precision of the data when determining, for example, the percentage of U.S. waters which have contaminant X above the minimum reporting level (MRL) or if contaminant X occurs more frequently or at higher concentrations in one type of water or geographical region than in another.

The ability to correctly identify a chemical contaminant is directly related to the type of chemical, the analytical method used, and the capabilities of the laboratory. For example, contaminants such as the disinfection by-products are far less likely to be misidentified than pesticides or herbicides, because disinfection by-products are typically present at relatively high concentrations in disinfected waters, while pesticides and herbicides are much less likely to be present, or are present at much lower concentrations. The analytical method selected will also determine the accuracy of the qualitative identification. In general, the most reliable qualitative identifications come from methods which use mass spectral data for contaminant identification. However, these methods are typically less sensitive than methods that rely on less selective detectors.

For the UCMR (1999) List 1 contaminants laboratory analyses, 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). These methods are identified and listed with the equivalent EPA method in Table 1.5.

The method that has been approved for use in monitoring perchlorate under the UCMR is EPA Method 314.0. 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 very high TDS (representing matrix conductance exceeding 3000 uS/cm) 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.

To ensure that the data collected under this regulation are of sufficient quality to meet the requirements of these regulatory decisions, EPA is specifying that only the analytical methods and procedures listed in Table 1.5 be used in obtaining these data. Note, however, that whether an EPA method or alternative method is used, specific quality control measures for UCMR

analyses are required (§141.40 Appendix A). This Manual explains these quality control requirements and contaminant confirmation procedures that are specified in the revised UCMR (40 CFR 141.40). The subsequent sections of this Manual provide an overview of methods, sampling, and quality control procedures to be used throughout the UCMR. EPA method numbers are used in this Manual as references for the reader. EPA will issue additional supplements to this Manual as new methods become available for the additional contaminants listed in Tables 1.3 and 1.4.

Table 1.5 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 502.2 [a, g]; EPA 524.2 [a]	D 5790-95 [b]; SM 6210D [c]; SM 6200B [c]; SM 6200C [c, g]
Nitrobenzene	98-95-3	EPA 524.2 [a, e]	D 5790-95 [b]; SM 6210D [c]; SM 6200B [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			
4,4'-DDE	72-55-9	EPA 525.2 [a]; EPA 508 [a]; EPA 508.1 [a]	D 5812-96 [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]	D 5475-93 [b]; 991.07 [d]
Molinate	2212-67-1	EPA 525.2 [a]; EPA 507 [a]	D 5475-93 [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]; EPA 515.3 [h, i]; EPA 515.4 [j]	D 5317-93 [b]; 992.32 [d]
DCPA di-acid degradate	2136-79-0	EPA 515.1 [a, e]; EPA 515.2 [a, e]; EPA 515.3 [h, i]; EPA 515.4 [j]	D 5317-93 [b]; 992.32 [d]

Chemical Contaminant	CAS #	Methodology	
		EPA Method	Equivalent Methods
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 and SM 6200 C are found only 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 found only 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 this document.

[f] EPA Method 314.0, "Determination of Perchlorate in Drinking Water by Ion Chromatography," EPA 815-B-99-003, November 1999. Also published in "Methods for the Determination of Organic and Inorganic Compounds in Drinking Water," EPA 815-R-00-014, August 2000. A copy of EPA Method 314.0 is also 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.

[g] Sample preservation and holding times should be conducted as specified in EPA method 524.2.

[h] Since Method 515.3 does not include a solvent wash step following hydrolysis, the parent DCPA is not removed prior to analysis, therefore, only non-detect data may be reported using Method 515.3. All samples with results above the MRL must be analyzed by one of the other approved methods (§141.40 (a)(3)).

[i] EPA Method 515.3, "Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with Electron Capture Detection," EPA 815-R-00-014, "Methods for the Determination of Organic and Inorganic compounds in Drinking Water, Volume 1," August 2000. Available from the National Technical Information Service, NTIS PB2000-106981, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll free number is (800)-553-6847. Alternatively, the method can be assessed and downloaded directly on-line at www.epa.gov/safewater/methods/sourcalt.html.

[j] EPA Method 515.4, "Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Fast Gas Chromatography with Electron Capture Detection," April 2000, EPA 815/B-00/001. Available by requesting a copy from the EPA Safe Drinking Water Hotline within the United States at (800)-426-4791 (Hours are Monday through Friday, excluding federal holidays, from 9:00 a.m. to 5:30 p.m. Eastern Time). Alternatively, the method can be assessed and downloaded directly on-line at www.epa.gov/safewater/methods/sourcalt.html.

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

Laboratories are automatically approved for the analysis of UCMR contaminants in Table 1.5 if they are already certified by a State or primacy agency to conduct compliance monitoring for a contaminant included in the same method being approved for UCMR analysis.

As noted in the revised UCMR (64 *FR* 50556), laboratories that provide data to 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)).

1.5.1 Laboratory Approval for Monitoring Acetochlor

EPA approved the use of EPA Method 525.2 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 (§141.40(a)(5)(ii)(G)(1)).

1.5.2 Laboratory Approval for Monitoring Perchlorate

The method that has been approved for monitoring perchlorate under the UCMR is not currently used nationally for compliance monitoring. Consequently, State certification programs do not include certification for this method. Because EPA Method 314.0 includes matrix specific quality control criteria, laboratories must have gone through a separate approval process to analyze samples for perchlorate (§141.40(a)(5)(ii)(G)(2)). 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 have successfully completed the perchlorate Performance Testing (PT) Program to be approved for UCMR perchlorate analyses (§141.40(a)(5)(ii)(G)(2)).

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.

Any laboratory who wished to participate in the perchlorate PT Program and obtain approval should have submitted a letter of request, received by EPA no later than March 31, 2000 (§141.40(a)(5)(ii)(G)(2)). Any interested laboratory that did not meet this deadline or failed to successfully pass this initial PT study and still wished to support this monitoring, should have submitted a request letter by October 6, 2000 in order to have been eligible for a second PT study. EPA did not consider any laboratory request letters received after October 6, 2000. Any laboratory that gained approval in the first PT study was not required to participate in the second PT study. These were the only two PT studies offered, through December 31, 2003, for laboratories who wished to gain approval to conduct perchlorate analysis in support of UCMR Assessment Monitoring. Any laboratory which did not request participation by October 6, 2000 and failed to pass either one of these two PT studies was not approved to support this perchlorate monitoring.

EPA provided each laboratory, upon successful completion of the perchlorate PT Program, with an approval letter identifying the laboratory by name and the approval date. This letter may be presented to any PWS as evidence of laboratory approval for perchlorate analysis supporting the UCMR. Laboratory approval is retained as long as the laboratory maintains certification by a

State or primacy agency 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. A list of perchlorate approved laboratories can be found on-line at: www.epa.gov/safewater/standard/ucmr/aprvlabs.html.

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

1.5.3 Laboratory Approval for Monitoring DCPA, mono- and di-acid degradates

As noted in the revised UCMR (66 *FR* 46221), laboratories certified under §141.28 for compliance analysis using EPA Method 515.3 are automatically approved to conduct UCMR analysis for the DCPA mono- and di-acid degradates using EPA Method 515.4 (§141.40(a)(5)(ii)(G)(1)).

1.6 Analytical Methods for UCMR (1999) List 2 Contaminants

Table 1.6 includes the UCMR (1999) List 2 chemical contaminants that are required for monitoring under the Screening Survey for chemical contaminants grouped by chemical class (66 *FR* 2273). EPA has not approved the use of any alternative analytical methods; therefore, only the EPA approved methods listed in Table 1.6 may be used to obtain data for the UCMR. The required quality control measures included in the final UCMR (64 *FR* 50556) and final UCMR List 2 Rule (66 *FR* 2273) are explained in this Manual.

Table 1.6 Approved Analytical Methods for UCMR (1999) List 2 Contaminants

Chemical Contaminant	CAS #	Methodology	
		EPA Method	Equivalent Methods
Semivolatile Organic Compounds			
1,2-Diphenylhydrazine	122-66-7	EPA 526 [a]	none identified
Diazinon	61790-53-2	EPA 526 [a]	none identified
Disulfoton	298-04-4	EPA 526 [a]	none identified
Fonofos	944-22-9	EPA 526 [a]	none identified
Nitrobenzene (low-level)	98-95-3	EPA 526 [a]	none identified
Prometon	1610-18-0	EPA 526 [a]	none identified

Chemical Contaminant	CAS #	Methodology	
		EPA Method	Equivalent Methods
Terbufos	13071-79-9	EPA 526 [a]	none identified
Phenols			
2-Methyl-phenol	95-48-7	EPA 528 [a]	none identified
2,4-Dichlorophenol	120-83-2	EPA 528 [a]	none identified
2,4-Dinitrophenol	51-28-5	EPA 528 [a]	none identified
2,4,6-Trichlorophenol	88-06-2	EPA 528 [a]	none identified
Phenlyureas			
Diuron	330-54-1	EPA 532 [a]	none identified
Linuron	330-55-2	EPA 532 [a]	none identified

[a] The version of the EPA methods which must be followed are listed in §141.40(a)(3).

1.7 Laboratory Certification Requirements for UCMR (1999) List 2 Contaminants

All laboratories currently certified by the appropriate primacy agency to conduct drinking water compliance monitoring using EPA Method 525.2 will automatically be approved to conduct UCMR analysis using EPA Method 526 and/or EPA Method 528. Those laboratories currently certified by the appropriate primacy agency to conduct drinking water analysis using EPA Methods 549.1 or 549.2 will automatically be approved to conduct UCMR analysis using Method 532. EPA will select up to eight contract laboratories nationally to analyze UCMR (1999) List 2 contaminants for small systems. Those laboratories must demonstrate that they meet certification requirements specified in §141.40 (a)(5)(ii)(G)(3).

Section 2. Sampling Plan

2.1 Monitoring by Public Water Systems

The focus of the monitoring in the revised UCMR is on occurrence or likely occurrence of contaminants in the drinking water of community and non-transient, non-community water systems. For regulatory purposes, PWSs are categorized as "community water systems," or "non-community water systems." Community water systems (CWSs) are specifically defined as "public water systems which serve at least 15 service connections used by year-round residents or regularly serve at least 25 year-round residents," while a non-transient non-community water system (NTNCWS) means "a public water system that is not a community water system" (40 CFR 141.2). These non-community systems are available to serve the public, but do not do so on a year-round basis in most cases, or do so but are used by people on a temporary basis (e.g., used by people traveling).

PWSs will monitor at the sampling sites and at the sampling frequencies specified in the revised UCMR (40 CFR 141.40). EPA or the State may provide further specifications and guidance on the monitoring schedule and other requirements to the PWSs. The subsequent general discussion of sampling is for informational purposes only and does not alter the requirements specified in the regulation or in directions from the State or EPA to PWSs.

2.1.1 Systems Required To Monitor

Under the Assessment Monitoring portion of this program, all CWSs and NTNCWSs serving more than 10,000 people (large systems) are required to monitor for unregulated contaminants (§141.40(a)(1)(ii)). However, PWSs that purchase 100% of their water must only monitor for UCMR contaminants that must be sampled for in the distribution system (i.e., the sampling point is listed as "distribution system") (§141.40(a)(1)(iii) and §141.40(a)(1)(v)). For systems serving 10,000 or fewer people (small systems), only a randomly selected, nationally representative sample of 800 CWSs and NTNCWSs must monitor (§141.40(a)(1)(iv)). EPA will pay for the reasonable costs of monitoring for this representative sample of small systems. The State or EPA will notify those systems selected for inclusion in the national representative sample. Transient non-community water systems will not be included in this monitoring (§141.40(a)(1)(i)).

The Screening Survey portion of the UCMR requires a smaller, randomly selected sample of 300 systems which represent large and small community and non-transient non-community water systems. EPA has selected 300 systems (for both the chemical and the microbial screening surveys), of which 180 are small systems and 120 are large systems, from those required to conduct Assessment Monitoring. EPA will pay for the reasonable costs of monitoring for the 180 small systems.

2.2 Sampling Frequency

PWSs will conduct their Assessment Monitoring during a 12 consecutive month period of the Assessment Monitoring period from 2001 to 2003. The year of monitoring and the time of sample collection may coincide with other scheduled compliance monitoring. For example, a

low-vulnerability system that may only monitor for compliance purposes during 1 year in a 3-year period could collect its required UCMR samples during that same year. Further, to the extent practical, analyses for the UCMR can be concurrently coordinated with analyses for other required State primacy compliance monitoring using the same methods approved for compliance monitoring to help reduce costs.

PWSs using surface water sources, or ground water under the direct influence of surface water, must sample four times per year for a 12 consecutive month period during the Assessment Monitoring period (§141.40(a)(5)). One of the sampling times must occur between May 1 and July 31, or another period of greatest vulnerability specified by the State or EPA (§141.40(a)(5)). Large PWSs using surface water or ground water under the direct influence of surface water must select either the first, second, or third month of a quarter and sample in that same month of each of four consecutive quarters (§141.40(a)(5)). In other words, systems must monitor under one of the following quarterly sampling schedules: January, April, July, October; or February, May, August, November; or March, June, September, December. PWSs using ground water sources will sample two times per year for a 12 consecutive month period during the Assessment Monitoring period, with one of these sampling times occurring between May 1 and July 31, or another period of greatest vulnerability as specified by the State or EPA. The second set of samples for ground water systems must be collected 5 to 7 months before or after the vulnerable period sampling event (§141.40(a)(5)). For all small PWSs participating in the national representative sample of small systems, the State or EPA will specify the month in which samples must be collected (§141.40(a)(4)(iii)(B)).

Monitoring for the Screening Survey for Chemical Contaminants will be conducted for 1 year, starting in January 2001 for small systems and January 2002 for large systems. EPA is limiting the time frame for conducting this monitoring (conducted over a one year period not 12 months over the 3-year period as with List 1 Assessment Monitoring). This proposed timing will allow monitoring of UCMR (1999) List 2 and List 1 contaminants concurrently at small systems. Small systems will monitor first because EPA is paying for the small system monitoring and desires to evaluate methods performance so that any necessary adjustments can be made prior to large system monitoring, which large systems must pay for themselves. The frequency of sampling for chemical contaminants on the UCMR (1999) List 2 is the same as for List 1 Assessment Monitoring.

2.2.1 Sampling Frequency Deviations

EPA recognizes that on occasion, circumstances beyond a PWS's or laboratory's control can invalidate data. Numerous QC failures are possible in the "real" world, from receiving samples broken or not within temperature parameters, to specific QC problems during sample analysis. If for any reason the sample data becomes invalid, samples should be recollected as soon as possible. QC failures that invalidate data could be immediately apparent and related to sample receipt temperature or storage, or may result from failures within the analysis batch or within a specific individual sample analysis. These QC problems could be interpreted as "sampling deviations" as identified in §141.40(a)(5)(ii)(F) of the September 17, 1999 UCMR. Any QC problems that may occur at the lab will need to be relayed back to the PWS through, "...notification from the laboratory that you must resample." Resampling should occur ".....within 14 days of observing the occurrence of the error" (§141.40(a)(5)(ii)(F)). Specific QC

criteria can be found in Appendix A to §141.40, in the analytical methods, as well as summarized in this Manual.

The laboratory should make every effort to inform the utility as soon as they learn of a QC problem which has invalidated UCMR sample data for a specific method. When a surface water (SW) system collects sample late in the quarter, and a QC failure is found in the first month of the next quarter that invalidates the data, the system should immediately recollect the sample, document the QC problem from the original sampling (verifying the intent to comply with the regulation), and get valid data from a subsequent sampling. The subsequent sampling event will be out of the official sampling month, and technically out of the quarter in which the majority of monitoring has been done, but data slightly out of phase is better than no data at all.

Systems should strive to collect samples within the regulatory defined sampling periods. When circumstances such as sample QC failures which invalidate data and, in turn, affect the monitoring frequency, the system should still immediately recollect the sample, document the QC problem from the original sampling (verifying the intent to comply with the regulation), and get valid data from a subsequent re-sampling event. The extent of the QC problem and how it will affect the entire data set should be evaluated. If the entire data set for all UCMR samples collected are invalid for a system, a determination should be made as to why such universal QC problems exist in the lab across all methods before proceeding with other analyses. After a determination has been made, all invalidated samples should be recollected as a second set of samples. If the impact is on an individual method's data, the system can maintain their original sampling schedule and just recollect for that method.

2.3 Sampling Points

Sampling must be performed at the locations specified in the UCMR Program (§141.40(a)(5)). These UCMR sampling locations, referred to as sampling points, are contaminant specific and are summarized in Tables 1.2 and 1.3 for the UCMR (1999) List 1 and List 2 contaminants, respectively. Sampling points for the UCMR (1999) List 3 contaminants listed in Table 1.4 are currently reserved.

For contaminants on the UCMR (1999) List 1, samples must be collected at entry points to the distribution system (EPTDSs) representing each non-emergency water source in routine use over the 12-month period of monitoring, 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)). Some States mandate source water (prior to treatment) sampling for regulated contaminant compliance monitoring. In these States, source water monitoring of UCMR contaminants allows systems to concurrently conduct UCMR monitoring with their regulated contaminant compliance monitoring. Thus UCMR samples may be collected from either the EPTDS or from the source (raw) water if the State has specified that source water sampling points are to be used for regulated contaminant compliance monitoring. However, if monitoring at source water sampling points indicates detection of any of the contaminants on the UCMR monitoring list, the system must then shift its unregulated contaminant monitoring (for the detected contaminant(s)) to the EPTDS (§141.40(a)(5)). This EPTDS monitoring, for a detected contaminant, must then be conducted for a new complete 12-month cycle at the required frequency respective of surface water or ground water (§141.40(a)()(ii)(B)). In this case, additional sampling for non-detected UCMR contaminants at the EPTDS would not be required. An exception to the EPTDS

resampling requirement applies if the State or EPA determines that no treatment or processing was in place between the source and EPTDS that would affect the measurement of the contaminants (§141.40(a)(5)(ii)(C)). The requirement for UCMR samples to be collected at the EPTDS follows the existing regulatory approach and provides data for exposure assessment.

EPA is seeking a representative result from the 300 systems that are required to monitor for UCMR (1999) List 2 chemical contaminants. Therefore, EPA requires that all Screening Survey samples for chemical contaminants be collected from entry points to the distribution system (EPTDS) to obtain nationally consistent results (§141.40(a)(5)). This condition of specifying exclusive EPTDS monitoring for List 2 chemicals is based on the fact that the approved methods for these contaminants are not currently used for any State compliance parameter monitoring and therefore concurrent compliance monitoring should not be an issue.

Section 3. Sample Collection and Preservation

3.1 UCMR (1999) List 1 Assessment Monitoring Chemical Contaminants

Sample preservation and holding times for the contaminant-specific analytical determinative methods specified in the UCMR Program are summarized in Table 3.1. The sample collection and preservation procedures as summarized below 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)(ii)(F)).

3.1.1 Nitrogen- and Phosphorus-Containing Pesticides

The three UCMR (1999) List 1 nitrogen- and phosphorus-containing pesticides, EPTC[1], molinate[1], and terbacil[1], may be analyzed with EPA Method 525.2, EPA Method 507 or the approved equivalent methods including ASTM Method D 5475-93 or AOAC Method 991.07 (see Table 1.5). For reference, see EPA Method 507 - *Determination of Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector* (Table 1.5). Sampling procedures based on EPA Method 525.2 are described below in Section 3.1.5. Sample procedures based on EPA Method 507, including sample containers, dechlorination, and sample collection, preservation, storage, and holding times are described below. The sampling and preservation requirements specified for ASTM Method D 5475-93 and AOAC Method 991.07 closely parallel those identified in EPA Method 507. Consequently, the following specifications also apply when laboratories choose to utilize those approved equivalent methods.

EPA Method 507 - *Determination of Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector*
(Also applicable to ASTM Method D 5475-93 and AOAC Method 991.07 for use in the UCMR)

Sample container - Use one-liter or one-quart amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used to protect samples from light. The bottle should be washed and dried as described in Section 4.1.1 of the EPA Method before use to minimize contamination. PTFE-faced cap liners should be extracted with methanol overnight prior to use to remove any potential contamination.

Sample dechlorination - To dechlorinate the sample, add approximately 80 milligrams of sodium thiosulfate per liter of sample to the sample containers prior to filling. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

[1] These pesticides are also semi-volatile organic compounds and therefore are also discussed in Section 3.1.5.

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 to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After the sample bottle has been filled, close the bottle, invert three or four times, and keep the sample bottle sealed until analysis.

The method specifies the addition of the biocide mercuric chloride to the sample to retard microbiological degradation. Mercuric chloride, however, is being withdrawn because it is highly toxic and poses handling and disposal problems. Mercuric chloride should not, therefore, be used to preserve samples for the UCMR Program.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory.

Sample holding time - Extract samples within 14 days. Preservation study results indicated that most method contaminants present in samples were stable for 14 days when stored under these conditions. The contaminants EPTC and terbacil exhibited recoveries of less than 60% after 14 days; consequently, the maximum sample holding time for these contaminants is 14 days. If samples are not extracted within this period, discard and replace the samples.

Sample extract storage and holding time - Analyze extracts within 14 days. Store extracts at 4˚C (± 2˚) away from light. Preservation study results indicate that most contaminants are stable for 28 days; however, a 14-day maximum extract storage time is recommended. (See Table 3.1 for a summary of holding times.) If sample extracts are not analyzed within the appropriate period, discard and replace the samples.

3.1.2 Chlorinated Hydrocarbon Pesticides

The UCMR (1999) List 1 chlorinated hydrocarbon pesticide, 4,4'-DDE[2], may be analyzed with EPA Methods 508, 508.1, 525.2 or the approved equivalent methods, including ASTM Method D 5812-96 and AOAC Method 990.06 (see Table 1.5). For reference, see EPA Method 508 - *Determination of Chlorinated Pesticides in Water by GC with an Electron Capture Detector* or EPA Method 508.1 - *Determination of Chlorinated Pesticides, Herbicides, and Organohalides by Liquid-Solid Extraction and Electron Capture Gas Chromatography.* Sampling procedures based on EPA Method 525.2 are described below in Section 3.1.5. Sampling procedures based on EPA Methods 508 and 508.1, including sample containers, dechlorination, and sample collection, preservation, storage, and holding times are described below. The sampling and

[2] The pesticide 4,4'-DDE is a semi-volatile organic compound and is therefore also discussed in Section 3.1.5.

preservation requirements specified for ASTM Method D 5812-96 and AOAC Method 990.06 closely parallel those identified in EPA Method 508. Consequently, the specifications below respective of Method 508 apply when laboratories choose to utilize those approved equivalent methods.

EPA Method 508 - *Determination of Chlorinated Pesticides in Water by GC with an Electron Capture Detector* (see Table 1.5).
(Also applicable to ASTM Method D 5812-96 and AOAC Method 990.06 for use in the UCMR)

Sample container - Use one-liter amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used to protect samples from light. The container should be washed and dried before use as described in Section 4.1.1 of the EPA Method to minimize contamination. PTFE-faced cap liners should be extracted with methanol overnight prior to use to remove any potential contamination.

Sample dechlorination - To dechlorinate the sample, add approximately 80 milligrams of sodium thiosulfate per liter of sample to the sample containers prior to filling. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other similar parts or materials that may leach chemicals into the sample (§141.40 Appendix A).

Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After the sample has been collected, close the bottle, invert three or four times, and keep the sample sealed from collection time until analysis.

The method specifies the addition of the biocide mercuric chloride to the sample to retard microbiological degradation. Mercuric chloride, however, is being withdrawn because it is highly toxic and poses handling and disposal problems. Mercuric chloride should not, therefore, be used to preserve samples for the UCMR Program.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory.

Sample holding time - Extract samples within 7 days. Preservation study results indicate that most of the target contaminants present in the samples are stable for 7 days when stored under

these conditions. Contaminant stability may be affected by the matrix. If samples are not extracted within the appropriate period, discard and replace the samples.

Sample extract storage and holding time - Store sample extracts at 4˚C (± 2˚), away from light. A 14-day maximum extract storage time is recommended. However, contaminant stability may be affected by the matrix; therefore, the analyst should verify appropriate extract holding times applicable to the samples under study. If sample extracts are not analyzed within the appropriate period, discard and replace the samples. (See Table 3.1 for a summary of holding times.)

EPA Method 508.1 - *Determination of Chlorinated Pesticides, Herbicides, and Organohalides by Liquid-Solid Extraction and Electron Capture Gas Chromatography* (see Table 1.5).

Sample container - Use one-liter or one-quart amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used because some of the method contaminants are sensitive to light and are oxidized or decomposed upon exposure to light.

Sample dechlorination - To dechlorinate the sample, add approximately 80 milligrams of sodium sulfite per liter of sample to the sample containers prior to filling. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After collecting the sample, close the bottle (PTFE face down), invert three or four times, and wait one minute until preserving the sample with acid.

Sample preservation - The one-minute waiting period after sample collection is crucial; it is important to reduce the level of residual chlorine before preserving the sample with acid. If the acid is added immediately following collection, the dechlorination reaction may be incomplete. Also, do *not* directly mix hydrochloric acid and sodium sulfite prior to sampling.

After waiting one minute, adjust the pH to less than 2 by carefully adding 6 N hydrochloric acid (this may require as much as 4 milliliters of acid). This should retard the microbiological degradation of the contaminants in water. Also, this is the same pH used in the extraction, and is necessary to support the recovery of acidic compounds. Close the sample bottle, PTFE face down, invert three or four times, and keep the sample sealed until analysis. After samples have been received, laboratories should use a pH meter or pH test strip to confirm the sample was properly acidified to a pH of 2 or less.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory.

Sample holding time - Extract samples within 14 days. Preservation study results show that the UCMR contaminants are stable for 14 days in samples that are preserved as described in Sections 8.2 and 8.3 of the EPA Method. If samples are not extracted within this period, discard and replace the samples.

Sample extract holding time - Analyze extracts within 30 days. Extracts can be held for 30 days when stored at 4˚C (± 2˚). If sample extracts are not analyzed within this period, discard and replace the samples. (See Table 3.1 for a summary of holding times.)

3.1.3 Acid Herbicides

The two UCMR (1999) List 1 acid herbicide-based contaminants, the mono- and di-acid degradates of dimethyl tetrachloroterephthalate (DCPA), may be analyzed with EPA Method 515.1, EPA Method 515.2, EPA Method 515.3, EPA Method 515.4, or the approved equivalent methods including ASTM Method D 5319-93 and AOAC Method 992.32 (see Table 1.5). For reference, see EPA Method 515.1 - *Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector*, EPA Method 515.2 - *Determination of Chlorinated Acids in Water Using Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector,* EPA Method 515.3 - *Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with Electron Capture Detection*, or EPA Method 515.4 - *Determination of Chlorinated Acids in Drinking Water By Liquid-Liquid Microextraction, Derivatization, and Fast Gas Chromatography with Electron Capture Detection* (Table 1.5). It is important to note that because the approved methods do not allow for the identification and quantification of the individual acids, the single analytical result obtained should be reported as total DCPA mono- and di-acid degradates. If the analytical results using EPA Method 515.3 is equal to or greater than the MRL, a duplicate sample must be analyzed within the method-specified holding time, or a replacement sample must be collected and analyzed within the same month as the original sample using one of the other approved methods, since EPA Method 515.3 does not differentiate between the DCPA parent compound and the degradates. For specific clarifications concerning the use of EPA Methods 515.1, 515.2, 515.3, 515.4, or their approved equivalent methods, please see Section 6.1 of this Manual. Sampling procedures based on EPA Methods 515.1, 515.2, 515.3, and 515.4, including sample containers, and dechlorination, and sample collection, preservation, storage and holding times are described below. The sampling and preservation requirements specified for ASTM Method D 5317-93 and AOAC Method 992.32 closely parallel those identified in EPA Method 515.1. Consequently, the following specifications, respective of Method 515.1, also apply when laboratories choose to utilize those approved equivalent methods.

EPA Method 515.1 - *Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector*
(Also applicable to ASTM Method D 5317-93 and AOAC Method 992.32 for use in the UCMR)

Sample container - Use one-liter or one-quart amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used to protect samples from light. The container should be washed and dried as described in Section 4.1.1 of the EPA Methods before use to minimize contamination. PTFE-faced cap liners should be extracted with methanol overnight prior to use to remove any potential contamination.

Sample dechlorination - To dechlorinate the sample, add approximately 80 milligrams of sodium thiosulfate per liter of sample to the sample container prior to filling. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After collecting the sample, close the sample bottle, invert three or four times, and keep the sample bottle sealed until analysis.

Sample preservation - The method specifies the addition of the biocide mercuric chloride to the sample to retard microbiological degradation. Mercuric chloride, however, is being withdrawn because it is highly toxic and poses handling and disposal problems. Mercuric chloride should not, therefore, be used to preserve samples for the UCMR Program.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory.

Sample holding time - Extract samples within 14 days. Preservation study results indicate that the contaminants (measured as total acid) present in samples are stable for 14 days when stored under these conditions. If samples are not extracted within the appropriate period, discard and replace the samples.

Sample extract storage and holding time - Store extracts at 4˚C (± 2˚) away from light. Analyze extracts within 28 days. Preservation study results indicate that analytes are stable for 28 days. (See Table 3.1 for a summary of holding times.) If sample extracts are not analyzed within the appropriate period, discard and replace the samples.

EPA Method 515.2 - *Determination of Chlorinated Acids in Water Using Liquid-Solid Extraction and Gas Chromatography with an Electron Capture Detector*

Sample container - Use 250 milliliter amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used to protect samples from light. The container should be washed and dried as described in Section 4.1.1 of the EPA Methods before use to minimize contamination. PTFE-faced cap liners should be extracted with methanol overnight prior to use to remove any potential contamination.

Sample dechlorination - To dechlorinate the sample, add approximately 20 milligrams of sodium thiosulfate per 250 mL of sample to the sample container prior to filling. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After the sample bottle has been filled, close the bottle (PTFE face down), invert three or four times, and then wait one minute before preserving the sample with acid.

Sample preservation - The one-minute waiting period after sample collection is crucial to reduce the level of residual chlorine before preserving the sample with acid. If the acid is added immediately following collection, the dechlorination reaction may be incomplete. After waiting one minute, adjust the pH to less than 2 by carefully adding 6 N hydrochloric acid (this may require as much as 1 milliliter of acid). This should retard microbiological degradation of the contaminants in the water. After samples have been received, laboratories should use a pH meter or pH test strip to confirm the sample was properly acidified to a pH of 2 or less.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory.

Sample holding time - Extract samples within 14 days. Preservation study results for EPA Method 515.2 indicate that the sample contaminants (measured as total acid) are stable in water for 14 days when stored under these conditions. If samples are not extracted within the appropriate period, discard and replace the samples.

Sample extract storage and holding time - Store extracts at 4˚C (± 2˚) away from light. Analyze extracts within 14 days according to EPA Method 515.2. Preservation study results

indicate that most contaminants are stable for 14 days according to EPA Method 515.2. (See Table 3.1 for a summary of holding times.) If sample extracts are not analyzed within the appropriate period, discard and replace the samples.

EPA Method 515.3 - *Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with Electron Capture Detection*.
NOTE: If the analytical results using EPA Method 515.3 is equal to or greater than the MRL, a duplicate sample must be analyzed within the method-specified holding time, or a replacement sample must be collected and analyzed within the same month as the original sample using one of the other approved methods, since EPA Method 515.3 does not differentiate between the DCPA parent compound and the degradates.

Sample container - Use an amber glass container of at least a 50 milliliter capacity with a PTFE-lined screw-cap. Meticulously wash and dry container as directed in section 4.1.1 of the EPA Method to minimize contamination. Amber bottles should be used to protect samples from light.

Sample dechlorination - To dechlorinate the sample, add 4 milligrams of sodium thiosulfate per 50 milliliters of sample to the sample bottle prior to collection. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 the tap and not through any plastic or rubber hoses or tubing. Adjust the flow to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A). Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After collecting the sample, cap the bottle, agitate by hand for 15 seconds, and keep the sample bottle sealed until analysis.

Sample preservation - Because of the several pH adjustments made to the samples in the course of this method, the addition of hydrochloric acid to the samples to retard biological activity has been omitted. However, the analyst should be aware of the potential for the biological degradation of the analytes.

Sample storage - Immediately store the samples at 4˚C (± 2˚) in a refrigerator or packed in ice. Samples must be protected from light until extraction.

Sample holding time - Extract samples within 14 days. Holding studies performed to date show that samples, when stored at 4˚C (± 2˚) or less, protected from light in glass vials with PTFE-lined caps and preserved with sodium thiosulfate, will remain stable for up to 14 days. If samples are not extracted within 14 days, discard and replace the samples.

Sample extract holding time - Analyze extracts within 14 days. Holding studies performed to date show that extracts, when stored at 4˚C (± 2˚) or less, protected from light in glass vials with PTFE-lined caps, will remain stable for up to 14 days. If extracts are not analyzed within 14 days, discard and replace the samples. (See Table 3.1 for a summary of holding times.)

EPA Method 515.4 - *Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Microextraction, Derivatization, and Fast Gas Chromatography with Electron Capture Detection*

Sample Container - Use an amber glass container with a PTFE-lined screw cap of at least 125 milliliter capacity. Amber bottles should be used to protect samples from light. Sample bottles must be washed and dried according to section 4.1 of the Method.

Sample dechlorination - To dechlorinate the sample, add 2 milligrams of sodium sulfite per 40 milliliters of sample volume to the sample bottle prior to collecting the sample. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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 the tap and not through any plastic or rubber hoses or tubing. Adjust the flow to a slow but steady stream (about the diameter of a pencil) and collect samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A). Fill the sample bottles until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After collecting the sample, cap the bottle, agitate by hand for 15 seconds, and keep the sample bottle sealed until analysis.

Sample preservation - Because of the several pH adjustments made to the samples in the course of this method, the addition of hydrochloric acid to the samples to retard biological activity has been omitted. However, the analyst should be aware of the potential for the biological degradation of the analytes.

Sample storage - All samples should be iced during shipment and must not exceed 10˚ C during the first 48 hours. Samples that are stored in the laboratory must be at or below 6˚ C and protected from light until extraction, but should not be frozen.

Sample holding time - Extract samples within 14 days. Holding studies performed to date show that samples, when stored at 6˚ C or less, protected from light in glass vials with PTFE-lined caps and preserved with sodium thiosulfate, will remain stable for up to 14 days. If samples are not analyzed within 14 days, discard and replace the samples.

Sample extract holding time - Analyze extracts within 21 days. Holding studies performed to date show that extracts, when stored at 6˚ C or less, protected from light in glass vials with

PTFE-lined caps, will remain stable for up to 21 days. If extracts are not analyzed within 21 days, discard and replace the samples. (See Table 3.1 for a summary of holding times.)

3.1.4 Volatile Organic Compounds

The two UCMR (1999) List 1 volatile organic compounds (VOCs) monitored under the revised UCMR Program, methyl tertiary-butyl ether (MTBE) and nitrobenzene, may be analyzed with EPA Method 524.2, or an approved equivalent method, such as ASTM Method D 5790-95 or APHA (Standard Methods) SM 6210D or SM 6200B (see Table 1.5). In addition, MTBE (but not nitrobenzene) may also be analyzed with EPA Method 502.2, or the approved equivalent method, APHA (Standard Methods) SM 6200C. For reference, see EPA Method 524.2 - *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*, or EPA Method 502.2 - *Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series*. It is important to keep the sample bottles in an area known to be free of VOCs prior to sample collection. General sampling procedures based on EPA Method 524.2 and 502.2, including sample containers, dechlorination, sample collection, sample preservation, and storage and holding times, are described below. For specific analytical method recommendations pertaining to the use of EPA Method 524.2 for measuring nitrobenzene, please see Section 6.2 of this Manual. **The sampling requirements specific to EPA Method 524.2 must also be followed when using any of the approved equivalent methods (§141.40(a)(4)(i)(A)) or EPA Method 502.2 (see Table 1, footnote n, §141.40(a)(3)).**

EPA Method 524.2 - *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*
(Also applicable to ASTM Method D 5790-95, APHA (Standard Methods) SM 6210D and SM 6200B for use in the UCMR)

EPA Method 502.2 - *Volatile Organic Compound in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series*
(Also applicable to APHA (Standard Methods) SM 6200C for use in the UCMR)

Sample containers - Use 40-milliliter to 120-milliliter screw cap glass vials, each equipped with a PTFE-faced silicon septum. To prepare sample bottles: wash vials and septa with detergent and rinse with distilled water; air dry the vials and septa at room temperature; place in a 105˚C oven for one hour; then remove and allow to cool in an area known to be free of organics.

Sample dechlorination - To dechlorinate the sample, add approximately 25 milligrams ascorbic acid (or 3 milligrams sodium thiosulfate[3]) per 40 milliliters of sample volume to sample container prior to collecting the sample. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories

[3] Because neither MTBE nor nitrobenzene boil below 25˚C, sodium thiosulfate may be used to reduce residual chlorine.

should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident. For these methods, this will likely need to be performed qualitatively on the remaining volume of sample left in the vial after the autosampler has processed the sample vial. Do not open the VOC vial prior to processing since the target analytes are volatile and sample integrity will be compromised.

Sample collection - Collect all samples in duplicate or triplicate. 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. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill sample to almost overflowing, but take care not to flush out any dechlorination chemicals that are in the sample bottle. Do not let air bubbles pass through the sample as the sample bottle is filled, and, when sealed, the sample bottle should not contain air bubbles. After the sample bottle has been filled, close the bottle (PTFE face down), invert three or four times, and then wait one minute before preserving the sample with acid.

Sample preservation - The one-minute waiting period after sample collection is crucial; it is important to reduce the residual chlorine before preserving the sample with acid. If the acid is added immediately following sample collection, the dechlorination reaction may be incomplete.

After one minute, adjust the pH to less than 2 by carefully adding four drops of 1:1 hydrochloric acid for every 40 milliliters of sample. (The hydrochloric acid preservation reduces sample pH in order to retard microbiological degradation of the contaminants being analyzed.) Ensure that no air bubbles are trapped in the completely full sample bottle. Close the sample bottle, PTFE face down, and invert three or four times. Keep the sample bottle sealed from collection time until analysis. After samples have been received, laboratories should use a pH meter or pH test strip to confirm the sample was properly acidified to pH of 2 or less. For these methods, this will likely need to be performed on the remaining volume of sample left in the vial after the autosampler has processed the sample vial. Do not open the VOC vial prior to processing since the target analytes are volatile and sample integrity will be compromised.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory. The sample storage area must be free of organic solvent vapors, excess heat and direct light (§141.40 Appendix A).

Sample holding time - Analyze all samples within 14 days of collection (see Table 3.1 for a summary of holding times). If samples are not analyzed within this period, discard and replace the samples.

3.1.5 Semi-Volatile Organic Compounds

The seven UCMR (1999) List 1 semi-volatile organic compounds, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 4,4'- DDE[4], acetochlor[5], s-ethyl-dipropylthiocarbamate (EPTC)[5], molinate[5], and terbacil[5], may be analyzed with EPA Method 525.2. In addition, aforementioned equivalent methods ASTM Method D 5812-96 or AOAC Method 990.06 can be used for 4,4'-DDE, and ASTM Method D 5475-93 or AOAC Method 991.07 can be used for EPTC, molinate and terbacil. Note that three of these semi-volatile organic compounds (2,4-dinitrotoluene, 2,6-dinitrotoluene, and acetochlor) may only be analyzed with EPA Method 525.2; there are no approved equivalent methods for these three contaminants. See Table 1.5 for a full listing of the approved analytical methods for each contaminant. For reference, see EPA Method 525.2 - *Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry*. General sampling procedures based on EPA Method 525.2, including sample containers, dechlorination, sample collection, preservation, storage, and holding times, are described below.

EPA Method 525.2 - *Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry*

Sample containers - Use one-liter or one-quart amber glass bottles fitted with PTFE-lined screw caps. Amber bottles should be used for the UCMR because some of the method contaminants are very sensitive to light and are oxidized or decomposed upon exposure to light. It is important to keep the sample bottles in an area known to be free of volatile and semi-volatile organic compounds prior to sample collection.

Sample dechlorination - To dechlorinate the sample, add approximately 40-50 milligrams of sodium sulfite to sample container prior to collecting the sample. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

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[4] This semi-volatile organic compound is a pesticide and is specifically identified and listed as a chlorinated pesticide in Table 1.5.

[5] These four semi-volatile organic compounds are pesticides and are specifically identified and listed as nitrogen/phosphorus pesticides in Table 1.5.

Fill sample to almost overflowing, but take care not to flush out any dechlorination chemicals that are in the sample bottle. After the sample bottle has been filled, close the bottle (PTFE face down), invert three or four times, and then wait one minute before preserving the sample with acid.

Sample preservation - The one-minute waiting period after sample collection is crucial to reduce the level of residual chlorine before preserving the sample with acid. If the acid is added immediately following collection, the dechlorination reaction may be incomplete. Also, do *not* directly mix hydrochloric acid and sodium sulfite prior to sampling.

After waiting one minute, adjust the pH to less than 2 by carefully adding 6 N hydrochloric acid (this may require as much as 4 milliliters of acid). This should retard the microbiological degradation of the contaminants in water. Also, this is the same pH used in the extraction, and is necessary to support the recovery of acidic compounds. Close the sample bottle, PTFE face down, invert three or four times, and keep the sample sealed until analysis. After samples have been received, laboratories should use a pH meter or pH test strip to confirm the sample was properly acidified to pH of 2 or less.

Sample storage - Immediately store the samples at 4˚C (± 2˚). To do so, place the samples on ice or with frozen cold packs in a cooler, or place in a refrigerator that can maintain the samples at 4˚C (± 2˚). Keep the samples stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory. Sample storage area must be free of organic contaminants, excess heat, and direct light (§141.40 Appendix A).

Sample holding time - Extract the samples within 14 days of sample collection. Results of the holding time and storage study of all method contaminants showed that most are stable for 14 days in water samples when the samples are dechlorinated, preserved, and stored as described above.

Sample extract holding time - Analyze the extracts within 30 days of extraction. (See Table 3.1 for a summary of holding times.) If samples are not analyzed within this period, discard and replace the samples.

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 1.5; §141.40(a)(5)). A copy of Method 314.0 can be obtained at: http://www.epa.gov/safewater/methods/met314.pdf or in, "*Methods for the Determination of Organic and Inorganic Compounds in Drinking Water*," EPA 815-R-00-014, August 2000. Sampling procedures based on EPA Method 314.0, including sample containers, and sample collection, storage, and holding times are described below.

EPA Method 314.0 - *Determination of Perchlorate in Drinking Water by Ion Chromatography*

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 (LFM) analysis (also referred to as the MS/MSD pair), and minimize waste disposal.

Sample dechlorination - This method does not include any dechlorination requirements.

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 preservation - This method does not include any special preservation requirements.

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 3.1 for a summary of holding times). If samples are not analyzed within this period, discard and replace the samples.

Table 3.1 Preservation and Holding Times for Approved UCMR (1999) List 1 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 502.2 SM 6200C	Sodium thiosulfate; 1:1 HCl - pH < 2; Cool 4 ˚ C (± 2˚); Dark	14 days	Not Applicable	40 - 120 mL	Screw Cap Vial with PTFE-lined Septum
EPA 507 D 5475-93 991.07	Sodium thiosulfate; Cool 4˚C (± 2˚); Dark	14 days	14 days (≤6˚C, Dark)	1 L	Amber Glass with PTFE-lined Cap

Method(s)	Preservation	Sample Holding Time	Extract Holding Time	Sample Size	Container
EPA 508 D 5812-96 990.06	Sodium thiosulfate; Cool 4˚C (± 2˚); Dark	7 days	14 days (≤6˚C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 508.1	Sodium sulfite; 6 N HCl - pH < 2; Cool 4˚C (± 2˚)	14 days	30 days (≤6˚C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 515.1 D 5317-93 992.32	Sodium thiosulfate; Cool 4˚C (± 2˚); Dark	14 days	28 days (≤6˚C, Dark)	1 L	Amber Glass with PTFE-lined Cap
EPA 515.2	Sodium thiosulfate; 6 N HCl - pH < 2; Cool 4˚C (± 2˚); Dark	14 days	14 days (≤6˚C, Dark)	250 mL	Amber Glass with PTFE-lined Cap
EPA 515.3	Sodium thiosulfate; Cool 4˚ C (± 2˚); Dark	14 days	14 days (≤6˚C, Dark)	50 mL	Amber Glass with PTFE-lined Cap
EPA 515.4	Sodium sulfite; Maintain ≤10˚ C for no longer than first 48 hrs (during shipment), then ≤6˚ C; Dark	14 days	21 days (≤6˚C, Dark)	125 mL	Amber Glass with PTFE-lined Cap
EPA 524.2 D 5790-95 SM 6210D SM 6200B	Ascorbic acid or Sodium thiosulfate; 1:1 HCl - pH < 2; Cool 4˚ C (± 2˚)	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 (± 2˚); Dark	14 days	30 days from extraction (≤6˚C, Dark)	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 1.5 for the full reference for each analytical method.

3.2 UCMR (1999) List 2 Screening Survey for Chemical Contaminants

Table 3.2 provides a summary of the preservation and holding times for approved analytical methods for UCMR (1999) List 2 chemical contaminants. The sample collection and preservation procedures as summarized below 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)(ii)(F)).

3.2.1 Semi-Volatile Organic Compounds

There are seven UCMR (1999) List 2 semi-volatile contaminants, diazinon, 2,4 dichlorophenol, 1,2 diphenolhydrazine, disulfoton, fonofos, prometon, and terbufos, that may be analyzed using EPA Method 526. For reference, see EPA Method 526 - *Determination of Selected Semi-volatile Organic Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)*. A copy of the method can be obtained at: http://www.epa.gov/safewater/methods/526.pdf or in, "*Methods for the Determination of Organic and Inorganic Compounds in Drinking Water*," EPA 815-R-00-014, August 2000. General sampling procedures, based on EPA Method 526, including sample containers, and dechlorination, and sample collection, preservation, storage, and holding times, are described below.

EPA Method 526 - *Determination of Selected Semi-volatile Organic Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)*

Sample containers - Use one-liter or one-quart amber or clear glass bottles fitted with PTFE-lined screw caps. The bottle should be washed and dried as described in Section 4.1 of the EPA Method to minimize contamination. It is important to keep the sample bottles in an area known to be free of volatile and semi-volatile organic compounds prior to sample collection.

Sample dechlorination - It is important that sodium thiosulfate and sodium sulfite not be used as they have been found to degrade target analytes. To dechlorinate a sample, add 100 milligrams of ascorbic acid to sample container prior to collection. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

Sample preservation - Along with ascorbic acid for dechlorination, three other preservation reagents must be added to each sample bottle prior to shipment to the field. 350 milligrams per liter of ethylenediaminetetraacetic acid (EDTA) trisodium salt to inhibit metal-catalyzed hydrolysis of target analytes, 1000 milligrams per liter of diazolidinyl urea must be added to inhibit biodegradation of the analytes, and each sample collected must be buffered to a pH of 7 using a mixture of 470 milligrams/L of tris(hydroxymethyl)aminomethane and 7.28 g/L of tris(hydroxymethyl)aminomethane hydrochloride. Alternately, 7.75 g/L of a commercial buffer crystal mixture of these constituents blended in the aforementioned proportions can be substituted.

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. Adjust the flow from the tap to a slow but steady stream (about the diameter of a pencil). Collect the sample from the flowing stream taking care not to flush out sample preservation agents. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

After the sample bottle has been filled to almost overflowing, cap the bottle (PTFE face down), and invert three or four times until preservatives are dissolved.

Sample storage - All samples should be iced during shipment, or shipped with frozen cold packs. Laboratories should confirm that samples arrive within 48 hours of collection and are ≤10˚ C upon receipt. Samples stored in the laboratory must be held at or below 6˚ C until extraction but should not be frozen.

Sample holding time - Extract samples within 14 days of sample collection. Results of the holding time and storage study of all method contaminants show that most are stable for 14 days in water samples when the samples are dechlorinated, preserved, and stored as described above. If samples are not extracted within this holding period, discard and replace the samples.

Sample extract holding time - Analyze extracts within 28 days. Sample extracts must be stored at 0˚ C or less and analyzed within 28 days after extraction. If samples are not extracted within this period, discard and replace the samples. (See Table 3.2 for a summary of holding times for List 2 analytical methods.)

3.2.2 Phenols

There are four UCMR (1999) List 2 phenolic contaminants, 2-methyl-phenol, 2,4 dichlorophenol, 2,4 dinitrophenol, and 2,4,6 trichlorophenol, that may be analyzed using EPA Method 528. For reference, see EPA Method 528 - *Determination of Phenols in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)*. A copy of the Method can be obtained at: http://www.epa.gov/nerlcwww/m_528.pdf or in, "*Methods for the Determination of Organic and Inorganic Compounds in Drinking Water*," EPA 815-R-00-014, August 2000. It is important that samples be stored or extracted in areas free of airborne phenols. General sampling procedures, based on this EPA Method, including sample containers, and dechlorination, and sample collection, preservation, storage, and holding times are described below.

EPA Method 528 - *Determination of Phenols in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)*

Sample container - Use one-liter amber glass bottles with PTFE-lined screw caps. Amber bottles should be used because method analytes are sensitive to light and may degrade upon exposure. All glassware must be cleaned and dried according to Section 6.1 of the EPA Method. Phenolic resin bottle caps should be avoided due to possible contamination.

Sample dechlorination - To dechlorinate the sample, add 40-50 milligrams of sodium sulfite to the sample bottle prior to transportation to the field or at time of collection. This is typically best done by adding the preservative salt to the sample container as part of the sampling kit preparation process at the laboratory prior to delivery of the kit to the field sampling site. After samples have been received, laboratories should employ a N,N-diethyl-*p*-phenylenediamine (DPD) test kit to confirm proper sample dechlorination. When effectively dechlorinated, no immediate red color change should be evident.

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. 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. Take care not to allow any plastic tubings, gaskets, and other parts to leach interfering analytes into the sample. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

The sample should fill the one-liter bottle to almost overflowing, but take care not to flush out any dechlorination chemicals that are in the sample bottle. After the sample bottle has been filled, close the bottle (PTFE face down), invert three or four times, and then wait one minute before preserving the sample with acid.

Sample preservation - The one-minute waiting period after sample collection is crucial; it is important to reduce the level of residual chlorine before preserving the sample with acid. If the acid is added immediately following sample collection, the dechlorination reaction may be incomplete. After one minute, the sample is adjusted to less than pH 2 by using 4 mL of 6 N hydrochloric acid. This serves to retard microbiological degradation of the contaminants in the water. Keep sample bottles sealed from collection time until analysis. After sample have been received, laboratories should use a pH meter or pH test strip to confirm the sample was properly acidified to pH of 2 or less.

Sample storage - All samples should be iced during shipment, or shipped with frozen cold packs. Laboratories should confirm that samples arrive within 48 hours of collection and are $\leq 10^{\circ}$ C upon receipt. Samples stored in the laboratory must be held at or below 6° C until extraction but should not be frozen.

Sample holding time - Extract samples within 14 days of collection. According to results of holding time studies of all method analytes, samples that are dechlorinated, preserved, and stored as described above, will remain stable for 14 days. Samples must be extracted within 14 days of collection. If samples are not analyzed within this period, discard and replace the samples.

Sample extract holding time - Analyze sample extracts within 30 days. Extracted samples must be analyzed within 30 days after extraction when stored at 0° C or less. If sample extracts are not analyzed within this period, discard and replace the samples. (See Table 3.2 for a summary of holding times for List 2 analytical methods.)

3.2.3 Phenylureas

The two UCMR (1999) List 2 phenylurea compounds, diuron and linuron, may be analyzed using EPA Method 532. For reference, see EPA Method 532 - *Determination of Phenylurea Compounds in Drinking Water by Solid Phase Extraction and High Performance Liquid Chromatography with UV Detection.* A copy of the Method can be obtained at: http://www.epa.gov/safewater/methods/532.pdf or in, "*Methods for the Determination of Organic and Inorganic Compounds in Drinking Water*," EPA 815-R-00-014, August 2000. General sampling procedures based on this method, including sample containers, dechlorination, and sample collection, preservation, storage, and holding times, are described below.

EPA Method 532 - *Determination of Phenylurea Compounds in Drinking Water by Solid Phase Extraction and High Performance Liquid Chromatography with UV Detection*

Sample containers - Use a 500 milliliter clear or amber glass container fitted with a PTFE-lined screw cap. Sample containers should be cleaned according to the procedures outlined in Section 4.1 of Method 532.

Sample dechlorination - To dechlorinate the sample, add 2.5 grams of Trizma crystals (a premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl) aminomethane hydrochloride] prior to shipment to the field. The Trizma crystals should be added to the sample bottles in solid form due to the uncertainty of its stability in concentrated aqueous solution.

Sample preservation - Each 500 milliliter sample bottle must contain 250 milligrams of cupric sulfate as a means of inhibiting microbiological decay of method analytes. Cupric sulfate must be added prior to shipment to the field along with the Trizma crystals used for dechlorination. Additional reagents to change the sample pH are unnecessary due to the buffering capabilities of the Trizma crystals.

Sample collection - When sampling from a water tap, remove any aeration equipment, open the tap and allow the system to flush until the temperature has stabilized, usually about two minutes. Collect the sample directly from the tap and not through any plastic or rubber hoses or tubing. Adjust the flow to a slow but steady stream (about the diameter of a pencil) and collect the samples from the flowing stream. Sampling equipment must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample (§141.40 Appendix A).

Fill the sample bottle until almost full, but take care not to flush out any dechlorination chemicals from the sample bottle. After the sample bottle has been filled, close the bottle, invert three or four times, and keep the sample bottle sealed until analysis.

Sample storage - All samples should be iced during shipment, or shipped with frozen cold packs. Laboratories should confirm that samples arrive within 48 hours of collection and are $\leq 10^{\circ}$ C upon receipt. Samples stored in the laboratory must be held at or below 6° C until extraction but should not be frozen.

Sample holding time - Extract samples within 14 days. Preservation study results indicated that most method analytes were stable for 14 days when stored under these conditions. Samples that are not extracted within this period must be discarded and replaced with new samples.

Sample extract holding time - Analyze sample extracts within 21 days of extraction. Sample extracts may be stored in methanol at 0° C or less for up to 21 days after extraction. Samples that are exchanged into reagent water/acetonitrile (60/40) for confirmational analysis may be stored for up to 7 days at 0° C. The combined extract holding time may not exceed 21 days. If sample extracts are not analyzed within this period, discard and replace the samples. (See Table 3.2 for a summary of holding times for List 2 analytical methods.)

Table 3.2 Preservation and Holding Times for Approved UCMR (1999) List 2 Analytical Methods

Method(s)	Preservation	Sample Holding Time	Extract Holding Time	Sample Size	Container
EPA 526	Ascorbic acid; EDTA trisodium salt; Diazolindinyl urea; crystal mixture (tris(hydroxymethyl) aminomethane and tris(hydroxymethyl) aminomethane hydrochloride)- pH = 7; Maintain ≤10˚ C for no longer than first 48 hrs (during shipment), then ≤6˚ C	14 Days	28 days at 0˚ C	1 L or 1qt	Amber or Clear Glass with PTFE-lined Cap
EPA 528	Sodium Sulfite; 6 N HCL - pH < 2; Maintain ≤10˚ C for no longer than first 48 hrs (during shipment), then ≤6˚ C	14 Days	30 days at 0˚ C	1 L	Amber Glass with PTFE-lined Cap
EPA 532	Trizma crystals; Cupric Sulfate Maintain ≤10˚ C for no longer than first 48 hrs (during shipment), then ≤6˚ C	14 Days	21 days at 0˚ C	500 mL	Amber or Clear Glass with PTFE-lined Cap

Note: EPA = EPA Methods
See Table 1.6 for the full reference for each analytical method.

3.3 Monitoring of Routine Water Quality Parameters

In the final UCMR Preamble and Rule (64 *FR* 50556), EPA required the monitoring of water quality parameters (pH) when collecting samples for unregulated chemical contaminants. After further evaluation of the UCMR Program, EPA believes that analyzing the pH of finished drinking water will not provide relevant data on chemical contaminant occurrence. While pH is important when monitoring raw water, finished water goes through many treatments that can alter pH. For many systems, even if pH were a significant factor in determining the fate of a particular contaminant reaching the drinking-water supply, such correlations are lost in the finished water by the purposeful adjustment of the pH. Having pH as a water quality parameter for chemical contaminants would be of limited use. Therefore, EPA has eliminated the monitoring of pH for chemical contaminants (§141.40(a)(4)(i)(B)) and has only retained the requirement for water quality parameters for UCMR microbial contaminant monitoring.

Section 4. Sample Transport

4.1 UCMR (1999) List 1 contaminants

Immediately after sample collection, place all UCMR (1999) List 1 samples (see below for perchlorate sample exception), 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 stored at 4˚C (± 2˚) during shipment and upon receipt at the laboratory. Do not let any samples freeze (§141.40 Appendix A). If transporting samples to an off-site laboratory, pack samples in insulated containers or coolers carefully to protect against sample bottle breakage during transport.

Perchlorate samples using EPA Method 314.0 do not need to be shipped in ice 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.

Transport the appropriately cooled [i.e., 4˚C (± 2˚)] and packed samples 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. All samples, except perchlorate, must be processed (meaning either extracted or analyzed when exclusively applied to methods where no extraction is performed) by the laboratory within 7 to 14 days of sample collection depending on which respective method is being used. Therefore, provide adequate time for sample pick-up, transport, delivery, extraction, and analysis (§141.40 Appendix A). Immediate (i.e., 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 ideal. Overnight delivery to the laboratory is strongly recommended in order to maintain proper temperature conditions.

4.2 UCMR (1999) List 2 Chemical Contaminants

All UCMR (1999) List 2 samples collected for chemical contaminants must follow the same procedures as those previously mentioned for the UCMR (1999) List 1 contaminants (§141.40 Appendix A). However, samples collected for all UCMR (1999) List 2 chemical contaminants must be received at the lab at 10˚C or below for sample transport. In the laboratory, all UCMR (1999) List 2 samples must be stored at 4˚C (± 2˚) and must be extracted within 14 days of sample collection (§141.40 Appendix A).

Section 5. UCMR Quality Control Requirements

Several methods approved by EPA for UCMR monitoring are currently used for compliance monitoring. As a result, mechanisms for reviewing laboratory qualifications and establishing certification for use of these methods are already in place in all states which have primacy over compliance monitoring to meet National Primary Drinking Water Standards (NPDWSs). Laboratories are approved to provide data to EPA in support of the UCMR using these methods if they are currently certified by a State or primacy agency to perform these analytical methods for NPDWS compliance (§141.40(a)(5)(ii)(G)).

Monitoring for perchlorate, by EPA Method 314.0, has special laboratory approval requirements (§141.40(a)(5)(ii)(G)). 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 manual. In addition, QC requirements unique to EPA Method 314.0 are discussed in Section 5.10.

UCMR approval for the List 2 chemical screening methods is also built on existing state or primacy certification. Approval to use EPA Methods 526 and 528 is contingent upon certification in EPA Method 525.2. UCMR approval to use EPA Method 532, is contingent upon certification in EPA Method 549.1 or 549.2.(§141.40(a)(5)(ii)(G)).

Approval for EPA Method 515.4 for Assessment Monitoring of the List 1 contaminants DCPA mono-acid and di-acid DCPA degradates is contingent upon certification in EPA Method 515.3.

UCMR Assessment Monitoring and the Screening Survey for chemical contaminants must be conducted using only the analytical methods specified in the UCMR (see Tables 1.5 and 1.6; §141.40(a)(5)). The QC procedures specified in Appendix A of the Rule as well as those additionally listed in the approved analytical methods are further described in this Manual and must be followed to ensure accurate and precise data (§141.40 Appendix A). In cases where a conflict exists between the specifications listed in Appendix A of the Rule and the criteria identified in the approved analytical method, the Rule defined criteria takes precedent as the defined QC requirement.

QC procedures and the frequency of QC testing vary among the methods. Many of the methods specified in the UCMR provide criteria to be used in evaluating and accepting laboratory performance based on related QC data. This section describes the various QC procedures EPA requires as part of the UCMR and the rationale for acceptance criteria. 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 are justifiable circumstances for excluding monitoring data from the database:

- failure to use the correct calibration check standard concentration
- failure to verify the calibration curve at the specified frequency
- failure to meet the acceptance criteria for verifying calibration

- contaminants detected in the laboratory reagent (method) blank at concentrations equal to or greater than one-half the minimum reporting level (MRL)
- when applicable, failure to meet the acceptance criteria for the internal standard
- when applicable, failure to meet the acceptance criteria for the surrogate standard
- failure to analyze samples and/or extracts within the specified holding times

Certain QC data must also be reported as part of the analytical services performed for the UCMR (§141.35(d)). These data are being collected to evaluate the quality of the monitoring data. These reporting requirements are noted in the reporting elements in Section 7 of this Manual.

5.1 Minimum Reporting Level

The minimum reporting level (MRL) concentrations for the UCMR (1999) List 1 Assessment Monitoring are listed in Table 5.1. They were determined by multiplying by 10 the least sensitive method's minimum detection limit (MDL),[6] or, when available, multiplying by 5 the least sensitive method's estimated detection limit (EDL)[7]. The MRL for VOCs was determined by multiplying by 10 either the published MDL or 0.5 micrograms per liter, whichever is greater. The MDL of 0.5 micrograms per liter (0.0005 milligrams per liter) was selected to conform to the VOC MDL requirements of 40 CFR 141.24(f)(17)(i)(E). The MRLs 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.

The MRL for the UCMR (1999) List 2 Screening Survey for chemical contaminants are listed in Table 5.1.2. They represent the value of the lowest concentration at which precision and accuracy determination were made during methods development and which are documented in the method. If method options are permitted, the concentration used was for the least sensitive option.

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 this Manual; §141.40 Appendix A).

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[6] The MDL equals the standard deviation times the Student's t value for 99% confidence level with n-1 degrees of freedom, where n is the number of replicates samples.

[7] The EDL equals the concentration of compound yielding approximately a five to one signal to noise ratio or the calculated MDL, whichever is greater.

Table 5.1 UCMR (1999) List 1 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; D 5812-96; 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; EPA 515.3; EPA 515.4; D 5317-93; 992.32	1 µg/L [a]
EPTC	EPA 507; EPA 525.2; D 5475-93; 991.07	1 µg/L [a]
Molinate	EPA 507; EPA 525.2; D 5475-93; 991.07	0.9 µg/L [a]
MTBE	EPA 502.2; EPA 524.2; D 5790-95; SM 6210D; SM 6200B; SM 6200C	5 µg/L [c]
Nitrobenzene	EPA 524.2; D 5790-95; SM 6210D; SM 6200B	10 µg/L [c]
Perchlorate	EPA 314.0	4 µg/L [b]
Terbacil	EPA 507; EPA 525.2; D 5475-93; 991.07	2 µg/L [a]

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5 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).

Table 5.1.1 UCMR (1999) List 2 Methods and Minimum Reporting Levels

Contaminant	Approved UCMR Analytical Methods	Minimum Reporting Level
1,2-Diphenylhydrazine	EPA 526	0.5 µg/L [a]
2-Methyl-phenol	EPA 528	1 µg/L [a]

Contaminant	Approved UCMR Analytical Methods	Minimum Reporting Level
2,4-Dichlorophenol	EPA 528	1 µg/L [a]
2,4-Dinitrophenol	EPA 528	5 µg/L [a]
2,4,6-Trichlorophenol	EPA 528	1 µg/L [a]
Diazinon	EPA 526	0.5 µg/L [a]
Disulfoton	EPA 526	0.5 µg/L [a]
Diuron	EPA 532	1 µg/L [a]
Fonofos	EPA 526	0.5 µg/L [a]
Linuron	EPA 532	1 µg/L [a]
Nitrobenzene	EPA 526	0.5 µg/L [a]
Prometon	EPA 526	0.5 µg/L [a]
Terbufos	EPA 526	0.5 µg/L [a]

Note: EPA = EPA Methods

[a] Minimum Reporting Level represents the value of the lowest concentration precision and accuracy determination made during methods development and documented in the method. If method options are permitted, the concentration used was for the least sensitive option.

5.2 Calibration

Each method describes calibration procedures that are used to determine the concentrations of the method contaminants. Some methods allow several options:

- a calibration curve based on either external standards or detector responses to the contaminant relative to an internal standard
- an average response factor for each contaminant

The laboratory must select and follow one of the calibration procedures outlined in the approved method to meet the requirements of the UCMR (§141.40 Appendix A). In addition, the gas chromatographic/mass spectrometry (GC/MS) methods, which include EPA Methods 524.2, 525.2, 526, and 528 have specific tuning criteria that must be met prior to performing the calibration procedure (§141.40 Appendix A).

All methods specified in the UCMR 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

Laboratories are not required to establish, on a daily basis, completely new calibration curves. However, the analyst must periodically verify calibration during sample analysis to ensure accuracy of the analytical results (§141.40 Appendix A). The frequency for verifying calibration varies according to the analytical method used. Frequency requirements for verifying calibration have been established by EPA to meet the accuracy requirements for the UCMR (1999) List 1 and List 2 methods and are presented in Table 5.2 and 5.5, respectively (§141.40 Appendix A).

Most of the methods recommend checking the instrument calibration using a mid-level calibration check standard. The method acceptance criteria for verifying calibration are based on this standard. However, to meet the objectives of the UCMR, calibration must be verified across the range of contaminant concentrations that are being measured (§141.40 Appendix A). Based on the recommendations from technical experts experienced with these methods, EPA is specifying calibration verification at low- (MRL level) and mid- levels for each method.

Analysis of the low-level calibration check standard (at or below the UCMR analyte MRL) must be completed prior to analysis of any samples; each contaminant must meet the acceptance criteria provided in Table 5.3 for UCMR (1999) List 1 contaminants and in Table 5.6 for the UCMR (1999) List 2 contaminants (§141.40 Appendix A). 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 all methods, after analyses of no more than 10 UCMR samples (not including method blanks, shipping blanks, matrix spikes (MSs), matrix spike duplicates (MSDs), and any independent QC samples that are analyzed with the UCMR samples), the calibration curve must be verified using either a low- or mid-level continuing calibration check standard; each contaminant must meet the respective acceptance criteria listed in Tables 5.3, 5.4, 5.6, or 5.7 (§141.40 Appendix A). If the criteria are not met, reanalyze all samples or extracts that were analyzed between this standard and the last standard meeting acceptance criteria for the problem contaminant(s) after the calibration problem is resolved. If the samples or extracts cannot be re-analyzed, then the data for the problem contaminant(s) are considered invalid for those samples and should not be reported to EPA.

It is important to note that an analysis batch for EPA Method 314.0 is defined as a sequence of samples which are analyzed within a 30-hour period and include no more than 20 field samples. The 30-hour period begins with the analysis of the instrument performance check (IPC) standard (see EPA Method 314.0 and Section 5.10.2.2), 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.

For EPA Method 314.0, unlike all other methods currently approved for UCMR monitoring, the method specifically requires the analyst to alternate between mid- and high-level calibration check standards (rather than low- and mid- level standards) every 10 field samples

(§141.40(a)(5)). This is the specification listed in the method and contradicts the regulatory prescribed low- and mid-levels. In addition, the acceptance criteria identified in method 314.0 are tighter than that which is listed in Appendix A of the Rule. To be consistent with the specifications of the regulation, Appendix A, EPA will accept the more liberal regulatory defined criteria for acceptance limits on calibration check standards and expects laboratories to alternate calibration check standards between the low- and mid- levels (data collected with the method prescribed initial low- followed by alternating mid- and high- calibration standards will not be rejected).

It is important to note that an acceptable end calibration check standard is highly recommended at the conclusion of the analysis batch after the analysis of the last field sample. This end calibration check can be at the low-level or the mid-level and should meet the respective acceptance criteria listed in Tables 5.3, 5.4, 5.6, or 5.7. This end calibration check standard might be analyzed soon after the previous calibration check standard as a result of a limited number (less than ten) of remaining field samples, but it will serve to validate the calibration at the very end of the analysis batch.

5.3 Detection Limit

The detection limit is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the contaminant concentration is greater than zero. Usually, measurements at the detection limit concentration are considered qualitative, because they are not precise enough to meet the needs of the data user. The initial calibration check standard in a laboratory analysis batch must be conducted at or below this regulatory defined MRL level and this demonstrates the laboratory's capability to accurately quantify at the MRL level. In order to ensure that the UCMR data is the highest in quality, do not report data to EPA below the statutory MRLs (Tables 5.1 and 5.1.1).

Laboratories should refer to the detection limit calculations in each method rather than in CFR 136 Appendix B (§141.40 Appendix A).

Table 5.2 UCMR (1999) List 1 Frequency Requirements for Verifying Calibration		
Methods	**Method Specifications**	**UCMR Specifications**
EPA 314.0	Analyze low-level standard at beginning; alternate between mid and high level standards after each 10 samples; analyze standard after last sample. Analysis batch should not exceed 20 samples or 30 hours	Analyze low-level standard at beginning; alternate between mid- and low-level standards after each 10 samples Recommend analyzing a standard after last sample, if not specifically required by method
EPA 502.2	Minimum of 1 CCC in a 12 hour shift; recommend analyzing CCC at beginning and end of shift and periodically in between	
SM 6200 C	Analyze CCC every 10 samples or every 12 hours - whichever is more frequent (6200 A.5.b.2)	
EPA 507	Beginning and end of each workday; recommend periodically during the day	
D 5475-93	Beginning of each workday; recommend after every 10 samples or at the end of workday	
991.07	1 or 2 CCC daily	
EPA 508 D 5812-96	Beginning and end of each workday; recommend periodically during the day	
990.06	1 or 2 CCC daily	
EPA 508.1	Minimum of 1 CCC in a 12 hour shift; recommend periodically analyzing during and at end of shift	
EPA 515.1 D 5317-93 992.32	Beginning and end of each workday; recommend periodically during the day	
EPA 515.2	Minimum of 1 CCC in a 12 hour shift; recommend beginning and end of workday	
EPA 515.3	Beginning each batch, after every 10 samples, and after last sample in batch	
EPA 515.4	Within a 24 hour period: At the beginning, after every 10 samples, and after last sample	
EPA 524.2	Minimum of 1 CCC at the beginning of each 12 hour shift; recommend periodically analyzing during the shift and at the end	
D 5790-95	Minimum of 1 CCC at the beginning of each 12 hour shift	
SM 6200 B	Analyze CCC every 20 samples or every 12 hours - whichever is more frequent (6200 A.5.b.2)	
SM 6210 D	Calibrate system daily (6210 C.5.c)	
EPA 525.2	Minimum of 1 CCC at the beginning of each 12 hour shift	

Note 1: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5 for the full reference for each analytical method.

Table 5.3 UCMR (1999) List 1 Low-Level Calibration Check Standard Concentrations and Acceptance Criteria

Contaminant	MRL, µg/L	Concentration of Low-Level Standard	Acceptance Criteria
2,4-Dinitrotoluene	2	≤ MRL	± 40 %
2,6-Dinitrotoluene	2	≤ MRL	± 40 %
4,4'-DDE	0.8	≤ MRL	± 40 %
Acetochlor	2	≤ MRL	± 40 %
DCPA mono- and di-acid degradates	1	≤ MRL	± 40 %
EPTC	1	≤ MRL	± 40 %
Molinate	0.9	≤ MRL	± 40 %
MTBE	5	≤ MRL	± 40 %
Nitrobenzene	10	≤ MRL	± 40 %
Perchlorate	4	≤ MRL	± 40 %
Terbacil	2	≤ MRL	± 40 %

Table 5.4 UCMR (1999) List 1 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	± 20 %
Terbacil	middle of calibration range	± 20 %

Table 5.5 UCMR (1999) List 2 Frequency Requirements for Verifying Calibration

Methods	Method Specifications	UCMR Specifications
EPA 526	Within a 24 hour period: At the beginning, after every 10 samples, and after last sample	Analyze low-level standard at beginning; alternate between mid- and low-level standards after each 10 samples As specified in these methods- analyze a standard after last sample
EPA 528	Within a 24 hour period: At the beginning, after every 10 samples, and after last sample	
EPA 532	Within a 24 hour period: At the beginning, after every 10 samples, and after last sample	

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method

Table 5.6 UCMR (1999) List 2 Low-Level Calibration Check Standard Concentrations and Acceptance Criteria

Contaminant	MRL, µg/L	Concentration of Low-Level Standard	Acceptance Criteria
1,2-Diphenylhydrazine	0.5	≤ MRL	± 40 %
2-Methyl-phenol	1	≤ MRL	± 40 %
2,4-Dichlorophenol	1	≤ MRL	± 40 %
2,4-Dinitrophenol	5	≤ MRL	± 40 %
2,4,6-Trichlorophenol	1	≤ MRL	± 40 %
Diazinon	0.5	≤ MRL	± 40 %

Contaminant	MRL, µg/L	Concentration of Low-Level Standard	Acceptance Criteria
Disulfoton	0.5	≤ MRL	± 40 %
Diuron	1	≤ MRL	± 40 %
Fonofos	0.5	≤ MRL	± 40 %
Linuron	1	≤ MRL	± 40 %
Nitrobenzene	0.5	≤ MRL	± 40 %
Prometon	0.5	≤ MRL	± 40 %
Terbufos	0.5	≤ MRL	± 40 %

Table 5.7 UCMR (1999) List 2 Mid-Level Calibration Check Standard Concentrations and Acceptance Criteria

Contaminant	Mid-Level Standard	Acceptance Criteria
1,2-Diphenylhydrazine	middle of calibration range	± 20 %
2-Methyl-phenol	middle of calibration range	± 20 %
2,4-Dichlorophenol	middle of calibration range	± 20 %
2,4-Dinitrophenol	middle of calibration range	± 20 %
2,4,6-Trichlorophenol	middle of calibration range	± 20 %
Diazinon	middle of calibration range	± 20 %
Disulfoton	middle of calibration range	± 20 %
Diuron	middle of calibration range	± 20 %
Fonofos	middle of calibration range	± 20 %
Linuron	middle of calibration range	± 20 %
Nitrobenzene	middle of calibration range	± 20 %
Prometon	middle of calibration range	± 20 %
Terbufos	middle of calibration range	± 20 %

5.4 Laboratory Reagent (Method) Blank

All of the methods approved for the UCMR require periodic analysis of a laboratory reagent (method) blank. For all methods, a method blank is defined as an aliquot of reagent water that is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. This blank is used to determine if method contaminants or other interferences are present in the laboratory environment, the reagents, or the apparatus.

The frequency of the method blank analysis depends on the type of sample manipulation required prior to the instrumental analysis. Methods that involve extraction of the sample usually stipulate analysis of a method blank with each set of samples that are extracted together. When the samples are analyzed directly, a blank is analyzed on a daily basis.

The required frequencies for analyzing method blanks for UCMR Assessment Monitoring and the Screening Survey for chemical contaminants are listed in Tables 5.8 and 5.10 (§141.40 Appendix A). To meet the objectives of the UCMR, analyze the method blank as the first sample on the instrument (immediately following the initial calibration check standard). For methods that involve extractions, carry the method blank through the extraction process. **Each extraction batch of samples must include a method blank** (§141.40 Appendix A).

An extraction batch is defined as all samples prepared/extracted together by the same person(s) during a work day (normally an 8-10 hour period for routine working schedules). Use the same lot of extracting solvent, internal standard spiking solution, and surrogate standard spiking solution for all samples included in a batch. When applicable, derivatize all samples in an extraction batch with the same batch of derivatizing agent. It is strongly recommended, when not specifically a method requirement, that extraction batches contain a maximum of 20 UCMR samples, not including method blanks, shipping blanks, any independent QC samples, matrix spike samples and matrix spike duplicate samples.

While some methods state that background interferences should be below the MDL, the general goal for all methods approved for the UCMR is to ensure that the background levels are low enough to not interfere with an accurate measurement. If any of the method analytes are detected at a concentration equal to or greater than one-half the MRLs, then perform no further analyses 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 (or extracts) and repeat the preparation procedure using another aliquot of each sample. If the samples cannot be re-extracted, then consider all data for the problem contaminant(s) invalid for all samples in the extraction or analysis batch, as appropriate, and flag the monitoring data as not meeting QC criteria. Data not meeting UCMR QC criteria should not be reported to EPA. When samples are invalided, the lab should notify the client PWS regarding the QC failure and attempt to collect a replacement sample for the analysis within 14 days after the QC error was found (§141.40(a)(5)(ii)(F)).

Contamination problems in the extraction process cannot be detected until the analysis step. If a problem is discovered, then the data for the contaminants affected by the contamination problem in all the samples in the extraction batch are lost unless the laboratory has a back-up aliquot of each sample which can be extracted. It is due to this concern that EPA strongly recommends

limiting the extraction batch to 20 UCMR samples in order to minimize the number of samples that could be potentially lost as a consequence of a blank contamination problem.

Laboratories should be aware of the potential for carry-over between samples when highly contaminated samples are analyzed. To avoid this, laboratories may find that additional blanks are needed to "rinse" the system after high concentration samples are analyzed.

For EPA Method 314.0, the method blank must be analyzed as the first sample on the instrument (immediately following the IPC standard.) 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.

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 Method 314.0 and Section 5.10.2.3 of this Manual. 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 5.8 UCMR (1999) List 1 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 502.2 SM 6200C	1 per analysis batch	1 per sample analysis batch
EPA 507 D 5475-93 991.07	1 per extraction batch or if reagents changed	1 per sample extraction batch
EPA 508 D 5812-96	1 per extraction batch or if reagents changed	1 per sample extraction batch
990.06	Frequency not specified	
EPA 508.1	1 per extraction batch processed in a 12 hour shift or with new batch of disks, cartridges, or new supply of reagents	1 per sample extraction batch
EPA 515.1 D 5317-93 992.32	1 per extraction batch processed in a work shift or if reagents changed	1 per sample extraction batch
EPA 515.2	1 per extraction batch processed in a work shift or if reagents changed	1 per sample extraction batch

Method	Method Specifications	UCMR Specifications
EPA 515.3	1 per extraction batch or if reagents changed	1 per sample extraction batch
EPA 515.4	Daily or 1 per extraction batch (≤20 samples whichever is greater)	1 per sample extraction batch (≤20 samples)
EPA 524.2 D 5790-95	1 per batch in a work shift	1 per sample analysis batch
SM 6210 D	1 each day	
SM 6200 B	1 each sample batch	
EPA 525.2	1 per extraction batch in a 12 hour shift	1 per sample extraction batch

Note: EPA = EPA Methods, D = ASTM Methods, SM = APHA Standard Methods, 900 series = AOAC Methods. See Table 1.5 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 5.9 UCMR (1999) List 1 Acceptance Criteria for Laboratory Reagent (Method) Blanks

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

Table 5.10 UCMR (1999) List 2 Frequency Requirements for Analyzing Laboratory Reagent (Method) Blanks

Method	Method Specifications	UCMR Specifications
EPA 526	Daily, or 1 per sample batch (≤20 samples), whichever is more frequent	Same as method
EPA 528	Daily, or 1 per sample batch (≤20 samples), whichever is more frequent	Same as method
EPA 532	1 per sample batch (≤20 samples)	Same as method

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method.

Table 5.11 UCMR (1999) List 2 Acceptance Criteria for Laboratory Reagent (Method) Blanks

Contaminant	Minimum Reporting Level, µg/L	Maximum Allowable Background Concentration (≤ ½ MRL), µg/L
1,2-Diphenylhydrazine	0.5	0.25
2-Methyl-phenol	1	0.5
2,4-Dichlorophenol	1	0.5
2,4-Dinitrophenol	5	2.5
2,4,6-Trichlorophenol	1	0.5
Diazinon	0.5	0.25
Disulfoton	0.5	0.25
Diuron	1	0.5
Fonofos	0.5	0.25
Linuron	1	0.5
Nitrobenzene	0.5	0.25
Prometon	0.5	0.25
Terbufos	0.5	0.25

5.4.1 Field Reagent Blank (Shipping or Travel Blank)

In EPA Methods 524.2 and 502.2, and ASTM D 5790-05 (but also recommended for SM 6210 or SM 6200 B and SM 6200 C) are specifications for the preparation, transport, and possible analysis of a field reagent blank (sometimes referred to as a shipping or travel blank) with each group of samples collected from the same general sample site at approximately the same time (§141.40 Appendix A). If no positive is measured for either MTBE or nitrobenzene in the sample analyzed by the respective method, the field reagent blank does not need to be analyzed. This blank is an aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, storage, preservation, and all analytical procedures. The purpose of this blank is to determine if method contaminants or other interferences are present in the field or shipping environment. If any of the contaminants are detected at concentrations equal to or greater than one half the MRL in the field reagent blank, consider all data for the problem contaminant(s) for all samples in the shipping batch invalid and flag the monitoring data as not meeting QC criteria. Data not meeting UCMR QC criteria should not be reported to EPA.

5.5 Quality Control Sample

Most of the UCMR methods recommend that the laboratory analyze a quality control (QC) sample at least quarterly. A QC sample is a solution of method contaminants of known concentration which is either used to spike an aliquot of reagent water or sample matrix or analyzed similar to a calibration standard. Obtain the QC sample from a source external to the laboratory and different from the source of calibration standards. Use the analysis of the QC sample to check the integrity of the calibration standard.

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)(4)).

5.6 Sample Matrix Spike and Matrix Spike Duplicate

Throughout this manual, the terms matrix spike (MS) and matrix spike duplicate (MSD) are routinely used but several of the approved methods refer to these samples as Laboratory Fortified Matrix (LFM) and Laboratory Fortified Matrix duplicate (LFM duplicate), respectively. Keep in mind that these terms are interchangeable and are representative of the exact same type of sample.

One technique that is useful in evaluating a laboratory's precision and accuracy for a method is to determine the precision and accuracy of duplicate analyses. To do this, either replicate samples are collected or, where sufficient volume was collected and target analytes are non-volatile, a sample may be divided into two or more aliquots in the laboratory, and processed and analyzed as separate samples. This technique is most useful when the original sample contains background concentrations of the method contaminants.

To effectively evaluate precision for UCMR contaminants, EPA is requiring preparation and analysis of an MS and MSD (§141.40 Appendix A). A laboratory-spiked MS sample is an aliquot of an environmental sample to which known quantities of the method contaminants are added in the laboratory. A laboratory MSD sample is a second identical fortified sample that has been prepared in exactly the same manner as the MS. For the purposes of the UCMR, EPA is not requiring that the standards used to spike the MS/MSD samples be obtained from a second source. Analyze these MS/MSD samples exactly like a typical monitoring sample; the purpose is to determine whether the sample matrix contributes bias to the analytical results. Determine if there are any measurable concentrations of the contaminants in the unspiked sample matrix in a separate sample analysis of the unfortified field sample.

Laboratories are required to prepare and analyze MS/MSD samples at the frequencies listed in Table 5.12 for UCMR (1999) List 1 methods and Table 5.13 for UCMR (1999) List 2 methods (§141.40 Appendix A). Laboratories are required to perform MS/MSD sample analyses on a minimum of 5% of the UCMR samples (at least one per 20 samples) or with each sample batch, whichever is more frequent (§141.40 Appendix A). For methods that involve extractions, select a sample to use to prepare the MS/MSD pair. For methods which require a 1 L sample aliquot (e.g., EPA 525.2, EPA 526, EPA 528) or the VOC methods (e.g., EPA 524.2), be certain to collect at least triplicate samples of that respective matrix (quadruplicate are strongly recommended since the fourth sample will function as a back-up for reanalysis). The triplicate aliquots will provide sufficient volume for the initial unfortified sample analysis and the MS/MSD pair. For some of the other methods, where multiple aliquots of sample can be drawn from a single sample collection bottle and the contaminants are not volatile (e.g., EPA 314.0, EPA 515.4), the laboratory may separate multiple aliquots from a single sample bottle in order to prepare the unfortified sample and the MS/MSD pair. For methods that require sample extraction, spike both the MS and the MSD with a known concentration of the contaminant(s) prior to extraction. Process the unspiked sample and MS/MSD pair of samples through the entire extraction and analysis process. For methods that do not involve extractions, spike both the MS and the MSD with a known concentration of the contaminant(s) and analyze the unspiked sample and MS/MSD pair of samples in the analysis batch.

Note: As described earlier, an extraction batch is defined as a set of samples prepared/extracted together at the same time by the same person(s) during a work day. Therefore, use the same lot of extracting solvent, internal standard spiking solution, and surrogate standard spiking solution for all samples included in a batch. When applicable, derivatize all samples in a batch with the same batch of derivatizing agent. It is recommended that laboratories include no more than 20 UCMR samples in an extraction batch. Following extraction, the analysis batch is established as the set of samples (or extracts) that are processed as a group during a continuous but limited time period.

Table 5.12 UCMR (1999) List 1 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 analysis batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 502.2	Not required	MS/MSD per 20 samples or per analysis batch, whichever is more frequent[a]; alternate low-, mid-level
SM 6200 C	1 MS/MSD per batch	
EPA 507 D 5475-93	1 per 20 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
991.07	None specified	
EPA 508	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
D 5812-96	10% of samples or 1 per set whichever is greater	
990.06	None specified	
EPA 508.1	1 per sample matrix	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 515.1 D 5317-93	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
992.32	1 per 20 samples	
EPA 515.2	1 per 10 samples or each sample set, whichever is greater	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 515.3	1 per extraction set or 10% of samples whichever is greater	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 515.4	1 per extraction batch ($\leq$20 samples)	MS/MSD per 20 samples or per extraction batch, whichever is more frequent[a]; alternate low-, mid-level
EPA 524.2 D 5790-95	Not required unless matrix effects suspected	MS/MSD per 20 samples or per analysis batch, whichever is more frequent[a]; alternate low-, mid-level
SM 6210 D	Specifies on-going analysis of samples to known additions - no frequency specified	
SM 6200 B	1 per analysis batch	
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 1.5 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 and 515.4, a batch may not contain more than 20 samples.

Table 5.13 UCMR (1999) List 2 Requirements for Performing Spiked Sample Analyses

Method	Method Specifications	UCMR Specifications
EPA 526	1 per extraction batch of 20 samples or less	Same as method
EPA 528	1 per extraction batch of 20 samples or less	Same as method
EPA 532	1 per extraction batch of 20 samples or less	Same as method

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method.

The laboratory must choose a spiking concentration from one of the two concentrations listed in Tables 5.14 and 5.15 for UCMR (1999) List 1 and List 2 contaminants, respectively (§141.40 Appendix A). 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.

Laboratories must report all MS/MSD sample recovery data and all data from the batch of samples processed/analyzed with the MS/MSD sample (§141.35(d)). In addition, laboratories must also report the spiking concentration for MS/MSD samples (§141.35(d)). To facilitate this, data element 16, Spiking Concentration, has been included in the UCMR data elements. Values for this data element should only be reported for MS/MSD samples, and the unit of measure for the value reported should match the unit of measure used to report the sample analytical result reported in data element 7, Analytical Result - Value (this unit of measure is reported in data element 8, Analytical Result - Unit of Measure). Data from MS/MSD samples will be used by EPA to evaluate the quality of the monitoring data. Water systems and laboratories may also use these data to determine the appropriateness of the methodology used to analyze the UCMR samples.

EPA plans to use the data from MS/MSD analyses to provide an estimate of the precision and accuracy of the entire UCMR database as an aggregate. Laboratories will not be required to meet specific percent recovery or precision requirements for the MS/MSD analyses. Monitoring data will not be rejected based on MS/MSD sample recovery data.

The precision of measurements will be evaluated on the basis of values reported for data element 14, Analytical Precision. For the UCMR, analytical precision is defined as the relative percent difference (RPD) between MS and MSD results. For each analytical result obtained, the laboratories must report the RPD for the MS/MSD set analyzed in the same batch of samples as the analytical result being reported (§141.35(d)). Analytical precision is calculated using the formula:

$$RPD = \frac{|r_1 - r_2|}{(r_1 + r_2) \div 2} \times 100\%$$

RPD = Relative Percent Difference
r_1 = matrix spike (MS) analytical result
r_2 = matrix spike duplicate (MSD) analytical result

EPA will also be evaluating the analytical accuracy of measurements reported by examining the values reported for data element 15, Analytical Accuracy. Analytical accuracy describes how close a result is to the true value, and will be measured through the use of spiked matrix samples. For the purposes of the UCMR, analytical accuracy is defined as the percent recovery of the contaminant in the analyzed MS sample. To calculate the analytical accuracy, laboratories should use the formula:

$$Percent\ Recovery = \frac{|r_1 - r_3|}{s} \times 100\%$$

r_1 = matrix spike (MS) analytical result
r_3 = sample analytical result
s = spiking concentration of matrix spike

Table 5.14 UCMR (1999) List 1 Concentrations for Spiking MS/MSD Samples

Contaminant	Low-Level Spike Concentration	Mid-Level Spike Concentration, µg/L
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
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

Table 5.15 UCMR (1999) List 2 Concentrations for Spiking MS/MSD Samples

Contaminant	Low-Level Spike Concentration	Mid-Level Spike Concentration, µg/L
1,2-Diphenylhydrazine	0.5 µg/L ± 20 %	± 20 % of mid-level standard
2-Methyl-phenol	1 µg/L ± 20 %	± 20 % of mid-level standard
2,4-Dichlorophenol	1 µg/L ± 20 %	± 20 % of mid-level standard
2,4-Dinitrophenol	5 µg/L ± 20 %	± 20 % of mid-level standard
2,4,6-Trichlorophenol	1 µg/L ± 20 %	± 20 % of mid-level standard
Diazinon	0.5 µg/L ± 20 %	± 20 % of mid-level standard
Disulfoton	0.5 µg/L ± 20 %	± 20 % of mid-level standard
Diuron	1 µg/L ± 20 %	± 20 % of mid-level standard
Fonofos	0.5 µg/L ± 20 %	± 20 % of mid-level standard
Linuron	1 µg/L ± 20 %	± 20 % of mid-level standard
Nitrobenzene (low level)	0.5 µg/L ± 20 %	± 20 % of mid-level standard
Prometon	0.5 µg/L ± 20 %	± 20 % of mid-level standard
Terbufos	0.5 µg/L ± 20 %	± 20 % of mid-level standard

5.7 Internal Standard

Several of the UCMR methods require or recommend the use of an internal standard (IS) for calibration and quantification purposes. An IS is a pure contaminant that is added to a sample or sample extract in a known amount. It is used to measure the relative responses of other method contaminants and surrogates that are components of the same solution. The IS must be a contaminant that is not a sample component (§141.40 Appendix A). When used, the IS is added to all samples, standards, and QC samples or their extracts.

The methods recommend specific compounds and concentrations for use as ISs. When the method provides flexibility in the selection of the IS or IS concentration, EPA allows the same flexibility for analyses of UCMR samples.

The methods vary in their specifications of when the IS is added during the sample processing steps. Some methods require the addition of the IS to the sample prior to extraction (i.e., EPA 525.2), while other methods stipulate the addition to the sample extract prior to instrumental analysis. Laboratories are required to follow the directions in the method when performing analyses for the UCMR (§141.40 Appendix A).

The methods also vary in the criteria used to evaluate the IS recovery, when IS techniques are utilized. In general, monitor the detector response to the IS in each sample; it should be relatively constant during the period in which a batch of samples is analyzed. Specific criteria for evaluating the IS responses are listed in the methods and summarized in Tables 5.16 and 5.17. Compare each sample's IS detector response to the average IS detector response obtained for the calibration curve. The acceptance criteria are given as *percentage recovery* which is determined using the following formula:

$$IS\ \%\ Recovery = \frac{Sample\ IS\ Detector\ Response}{Calibration\ Curve\ Average\ IS\ Detector\ Response} \times 100\%$$

If the IS in a specific sample does not meet the acceptance criteria specified in the method, then consider data from that sample analysis invalid. If possible, re-analyze the sample. If this cannot be done, then the data for that sample are considered invalid for the analysis and should not be reported to EPA.

Table 5.16 UCMR (1999) List 1 Criteria for Internal Standard Response

Method	Method Specifications	UCMR Specifications
EPA 314.0	Not applicable	Same as method
EPA 502.2	IS response should not deviate from the mean IS response established with the calibration curve by more than 20%	
SM 6200 C	IS response should not deviate from the mean IS response established with the calibration curve by more than 30%	
EPA 507 D 5475-93 991.07	IS response should not deviate from the daily CCC IS response by more than 30%	
EPA 508 D 5812-96 990.06	IS response should not deviate from the daily CCC IS response by more than 30%	
EPA 508.1	IS response should not deviate from the most recent CCC IS response by more than 30% or from the mean IS response established with the calibration curve by more than 50%	
EPA 515.1 D 5317-93 992.32	IS response should not deviate from the daily CCC IS response by more than 30%	
EPA 515.2	IS response should not deviate from the daily CCC IS response by more than 30%	

Method	Method Specifications	UCMR Specifications
EPA 515.3	IS response should not deviate from the mean IS response established with the calibration curve by more than 30%	Same as method
EPA 515.4	IS response should not deviate from the mean IS response established with the calibration curve by more than 50%	
EPA 524.2 D 5790-95	IS response should not deviate from the most recent CCC IS response by more than 30% or from the mean IS response established with the calibration curve by more than 50%	
SM 6200 B	IS response should not deviate from the mean IS response established with the calibration curve by more than 30%	
SM 6210 D	No criteria listed in method	
EPA 525.2	For CCCs, IS response should not deviate from the most recent CCC IS response by more than 30% or from the mean IS response established with the calibration curve by more than 50%. (If a recovery standard is added to sample extracts, then IS recovery should be in excess of 70%.)	Same as method - Labs should at a minimum apply the CCC criteria to all analyses

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

Table 5.17 UCMR (1999) List 2 Criteria for Internal Standard Response

Method	Method Specifications	UCMR Specifications
EPA 526	IS response should not deviate from the mean IS response established with the calibration curve by more than 50%	Same as method
EPA 528	IS response should not deviate from the most recent CCC IS response by more than 30% or from the mean IS response established with the calibration curve by more than 50%	
EPA 532	Not applicable	

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method.

5.8 Surrogate Standard

Several of the UCMR methods require the use of surrogate analytes. A surrogate is a pure analyte which is extremely unlikely to be found in any sample. It is added to a sample aliquot in a known amount before the sample is processed, and is measured with the same procedures used to measure other sample target contaminants. The purpose of a surrogate analyte is to monitor

method performance with each sample. When used, the surrogate is added to all samples, standards, and QC samples.

The methods usually recommend specific compounds and concentrations for use as surrogate standards. When the method provides flexibility in the selection of the surrogate standard or its concentration, EPA allows the same flexibility for analyses of UCMR samples.

Tables 5.18 (UCMR (1999) List 1) and 5.19 (UCMR (1999) List 2) summarizes the UCMR methods that require surrogates as well as percent recovery acceptance criteria where specified by the appropriate methods (§141.40 Appendix A).

For EPA Methods 524.2 and 525.2, the surrogate criteria are listed in Section 10.2.6.1 of each method. The surrogate standards for EPA Methods 526, 528, and 532 are located in Sections 9.9, 9.10, and 9.7 of each method, respectively.

There are two techniques for monitoring the surrogate standard. If the method specifies that the same concentration of surrogate standard must be added to all samples, standards and QC samples, then compare the surrogate detector response in each analysis to the average surrogate detector response obtained for the calibration curve. The acceptance criteria are given as *percentage recovery* which is determined using the following formula:

$$\textit{Surrogate \% Recovery} = \frac{\textit{Sample Surrogate Detector Response}}{\textit{Calibration Curve Average Surrogate Detector Response}} \times 100\%$$

Some methods recommend preparing a calibration curve for the surrogate standard similar to the preparation of a curve for each of the method contaminants. In those cases, the acceptance criteria are given as *percentage recovery* which is determined using the following formula:

$$\textit{Surrogate \% Recovery} = \frac{\textit{Measured Surrogate Concentration}}{\textit{Expected Surrogate Concentration}} \times 100\%$$

If the surrogate in a specific sample does not meet the acceptance criteria, re-analyze the sample if possible. If this cannot be done, then the data for that sample are considered suspect for the analysis in question, and should not be reported to EPA.

EPA recognizes that failure to meet the surrogate standard recovery criteria could be the result of matrix interferences in a small number of instances. Even if this is the reason for failure, the data are suspect for all the contaminants in the analysis.

Table 5.18 UCMR (1999) List 1 Requirements for Surrogate Standard Analyses

Method	Method Specifications	UCMR Specifications
EPA 314.0	Not Applicable	Same as method
EPA 502.2	Surrogate recovery should be 80 - 120% of true value (9.3.3 and 9.7)	
SM 6200C	Surrogate recovery should be within ± 30% of true value	
EPA 507 D 5475-93 991.07	Surrogate recovery should be 70 - 130% of true value (9.5)	
EPA 508 990.06	Surrogate recovery should be 70 - 130% of true value (9.5)	
D 5812-96	No criteria for surrogate recovery	
EPA 508.1	Surrogate recovery should be 70 - 130% of true value (9.3.5)	
EPA 515.1 D 5317-93 992.32	Surrogate recovery should be 70 - 130% of true value (10.5)	
EPA 515.2	Surrogate recovery should be 60 - 140% of true value (9.5)	
EPA 515.3	Surrogate recovery should be 70 - 130% of true value (9.8)	
EPA 515.4	Surrogate recovery should be 70 - 130% of true value (9.8)	
EPA 524.2	Surrogate response within 30% of mean response measured in initial calibration curve (10.3.5)	
D 5790-95	Surrogate recovery between 70 - 130% of true value (14.3.3 - IDC requirement)	
SM 6200 B	Surrogate recovery should be within ± 30% of true value	
SM 6210 D	No criteria listed in method	
EPA 525.2	Surrogate recovery should be 70 - 130% of true value (9.3.3 - IDC requirement)	

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

Table 5.19 UCMR (1999) List 2 Requirements for Surrogate Standard Analyses		
Method	**Method Specified Surrogate Recovery**	**UCMR Specifications**
EPA 526	Surrogate recovery between 70 - 130% (9.9)	Same as method
EPA 528	Surrogate recovery between 70 - 130% except for 2,4,6-tribromophenol which is between 60 - 130% (9.10)	
EPA 532	Surrogate recovery between 70 - 130% (9.7)	

Note: EPA = EPA Methods. See Table 1.6 for the full reference for each analytical method.

5.9 Confirmation

5.9.1 Gas Chromatographic Methods

Preliminary identification of contaminant compounds using EPA Methods 507, 508, 508.1, 515.1, 515.2, 515.3 and 515.4, as well as in the approved alternative methods (see Table 1.5), is performed by comparison of the target contaminant retention time to the retention time of a standard reference chromatogram. If the retention time of an unknown contaminant corresponds, within standard acceptable limits (known as retention time windows), to the retention time established during calibration of a standard reference compound, then identification is presumed positive. The UCMR requires analytical confirmation by gas chromatographic/mass spectrometry (GC/MS) for positive identification (§141.40 Appendix A).

The length of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. A suggested window size for a particular contaminant can be calculated using three times the standard deviation of a retention time for that particular contaminant. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

Identification requires expert judgement when sample components are not resolved chromatographically. When peaks obviously represent more than one sample contaminant (i.e., a broadened peak with shoulder[s] or a valley between two or more maxima), or any time when doubt exists over the identification of a peak on a chromatogram, use appropriate alternative techniques to help confirm peak identification. Positive identification of contaminants is required for results by GC/MS (§141.40 Appendix A) except for EPA 314.0 and EPA 532. False positives can be minimized by the use of a second dissimilar chromatography column. Primary columns and suggested alternative dissimilar confirmation columns are described in each of the methods. GC/MS analysis for confirmation is not necessary if positive results from the primary column cannot be confirmed by the secondary dissimilar column or if the primary analysis uses GC/MS as the determinative step.

5.9.2 Gas Chromatography/Mass Spectrometry Confirmation

The UCMR requires that any contaminant detected above the MRL when using a gas chromatography method must be confirmed using GC/MS (§141.40 Appendix A). Analytes

detected using ion chromatography do not need to be confirmed (Section 5.9.4) and analytes detected using HPLC must be confirmed by analysis using a dissimilar second chromatographic column (Section 5.9.5). Laboratories have the option of confirming the presence of an analyte using a second chromatography column prior to submitting the sample for GC/MS analyses, or may go directly from the primary column analyses to GC/MS confirmation. If the contaminant detection is confirmed by the secondary column, then reconfirm the contaminant by GC/MS using three specified ion peaks for contaminant identification. Recommended ion peaks for identification purposes are listed in Tables 5.20 and 5.21. The UCMR allows single point calibration of the GC/MS system for confirmation purposes only as long as the calibration standard is at a concentration within ± 50% of the concentration determined by the initial analysis. As an option, laboratories may prefer to perform a three-point calibration, bracketing the measured concentration, with single point daily calibration verification of the GC/MS system regardless of whether that verification standard concentration is within ± 50% of sample response. If GC/MS analysis confirms the initial contaminant detection, report the results determined from the initial analysis.

Table 5.20 UCMR (1999) List 1 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, mono- and di-acid	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.

Table 5.21 UCMR (1999) List 2 Recommended Confirmation Ions

Contaminant	Recommended Confirmation Ions
1,2-Diphenylhydrazine	77, 51
2-Methyl-phenol	108, 79
2,4-Dichlorophenol	63, 164
2,4-Dinitrophenol	107, 132, 196
2,4,6-Trichlorophenol	
Diazinon	137, 304
Disulfoton	89, 97
Fonofos	109, 137
Nitrobenzene	51, 123
Prometon	168, 183
Terbufos	153, 125
Linuron	Not Applicable
Diuron	Not Applicable

5.9.3 Mass Spectrometry Methods

Perform identification and confirmation of a contaminant using EPA Methods 524.2, 525.2, 526 and 528 by comparison of the contaminant's mass spectrum (after background subtraction) to a reference spectrum in the user-created database. The GC retention time of the contaminant should be within three standard deviations of the mean retention time of the reference contaminant in the calibration mixture.

In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample and should agree within ± 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample (contaminant) spectrum should be in the range of 10-50%. Some ions, particularly the molecular ions, are of special importance and should be evaluated even if they are below 10% relative abundance.

Identification requires expert judgment when sample contaminants are not resolved chromatographically and produce mass spectra containing ions that are contributed by more than one contaminant. When GC peaks obviously represent more than one sample contaminant (i.e., a broadened peak with shoulder[s] or a valley between two or more maxima), select appropriate contaminant spectra and background spectra by examining plots of characteristic ions for tentatively identified contaminants. When target contaminants co-elute (i.e., when only one GC peak is apparent for two or more contaminants), the identification criteria can be met, but each

contaminant spectrum will contain extraneous ions contributed by the co-eluting contaminants. Because purgeable organic compounds (such as those for some UCMR contaminants) are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most contaminants determined according to EPA Method 524.2.

Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC or retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

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 retention time window 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 5.22 displays an example of the 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 5.22 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. This may result in an unacceptable revised laboratory MRL which is above the statutory MRL of 4 µg/L.

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 are between the MRL and the highest calibration standards.

5.9.5 High Performance Liquid Chromatography (HPLC) with UV Detection

EPA Method 532 utilizes HPLC to analyze for linuron and diuron. All positive results using HPLC are to be confirmed by using a second, dissimilar HPLC column (§141.40 Appendix A).

5.10 Additional Quality Controls

The laboratory should examine the samples when they arrive in the laboratory to determine if the proper shipping procedures were used and the required shipping conditions were maintained. Samples requiring storage at 4˚C (± 2˚), or 10˚C or below for UCMR (1999) List 2 samples should arrive at the laboratory packed in ice or frozen cold packs. Samples should not be analyzed if they were not shipped properly and/or if they did not arrive in the required temperature. As the UCMR specifies that resampling is required within 14 days of the observance of a sampling error (§141.40(a)(5)(ii)(F)), the laboratory should immediately contact the water system and arrange for resampling. If resampling cannot be performed within this time period, then the water system should attempt to recollect these samples as soon as possible and indicate in their official file why recollection was not possible within 14 days and why the original samples were invalidated.

The laboratory also must invalidate samples that were collected in improper sampling containers (e.g., plastic bottles instead of glass) or that were improperly filled (e.g., half-filled bottles for samples that are required to be completely full) (§141.40(a)(5)). As above, the laboratory should immediately contact the water system and arrange for resampling within the required 14 days (§141.40(a)(5)(ii)(F)).

Finally, the laboratory must ensure each sample is analyzed within the required holding time (§141.40 Appendix A). A list of applicable holding times is presented in Tables 3.1 and 3.2. When appropriate, EPA standardized the holding times across analytical methods for the same contaminant group.

If a UCMR sample is not extracted or analyzed within these holding times, then the data for the sample should not be reported and the sample should be recollected. As above, the laboratory should immediately contact the water system and arrange for resampling within the required 14 days (§141.40(a)(5)(ii)(F)).

There are other problems, of course, that may invalidate a sample. Some additional comments on sampling frequency deviations are identified in 2.2.1.

5.10.1 Laboratory Fortified Blank

A laboratory fortified blank (LFB) analysis must be performed if it is a requirement in the method (§141.40 Appendix A). Each analysis should be performed to the specifications and acceptance criteria of each respective method.

Specifically, EPA Method 314.0 requires the LFB to be 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 extremely 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, 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 the laboratory chooses to dilute, their revised MRL must still be lower than the statutory MRL of 4 µg/L for perchlorate.

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

EPA Methods 515.1 and 515.2, as well as their equivalent methods from voluntary consensus organizations (D 5317-93 and 992.32), have been approved for use in measuring the DCPA mono- and di-acid degradates under the UCMR (see Table 1.5). However, EPA is specifically requiring that the solvent wash listed in Sections 11.1.4 and 11.1.5 of EPA Methods 515.1 and 515.2 (Sections 12.1.4 and 12.1.5 of D 5317-93 and Section F(a) of 992.32) be performed (§141.40(a)(5)). The use of this wash is being emphasized in the QC requirements because it washes away the parent compound, DCPA.

If this wash is not performed, the data generated will reflect not just the total of the mono- and di-acid degradates of DCPA, but will include the concentration of the parent compound in addition to the concentration of the two degradates. As noted previously, because the approved methods do not allow for the identification and quantification of the individual acids, the single analytical result obtained from these methods should be reported as total DCPA mono- and di-acid degradates.

Please note that the hydrolysis step listed immediately prior to the solvent wash step (i.e., Section 11.1.3 of the EPA methods) is not strong enough to hydrolyze the parent compound (DCPA) to either the mono- or di-acid degradate if it is performed as described in the EPA Methods.

6.2 Recommendations for EPA Method 524.2 and the Approved Equivalent Methods

Although EPA is not altering the method specifications for EPA Method 524.2 for use with the UCMR, EPA emphasizes that laboratories should use the three stage trap listed in Section 6.2.2 of the Method [Section 7.2.2.1 of D 5790.95, Section 3 of SM 6210D, and Section 2(a)(2) of SM 6200B]. Alternate traps may not be capable of retaining adequate concentrations of nitrobenzene, therefore, nitrobenzene can not be detected at reasonable concentrations in either standards or samples. Thus, the data generated for nitrobenzene with this method will not be capable of meeting the quality control requirements specified in the UCMR and described in this Manual.

6.3 Clarifications Concerning EPA Method 515.3

EPA Method 515.3 was approved for the analysis of DCPA mono- and di-acid degradates under the UCMR (see Table 1.6). However, it must be noted that because EPA Method 515.3 does not require a solvent wash step to remove the DCPA parent, there are conditions which must be met if Method 515.3 is to be used (§141.40(a)(3)). If DCPA mono- and di-acid degradates are detected at concentrations greater than or equal to the MRL, then a duplicate sample must be analyzed within the method specified holding time, or a replacement sample must be collected and analyzed within the same month as the original sample, using one of the other approved methods (§141.40(a)(3)). Sampling results greater than the MRL using Method 515.3 should not be reported to EPA, only the results of the subsequent analysis.

6.4 Additional Notes on the Analysis of 2,4- and 2,6-Dinitrotoluene by Method 525.2

Since promulgation of the UCMR, some laboratories have reported that they have observed low recoveries for 2,4- and 2,6-dinitrotoluene. Reasons for low recoveries of these compounds include: 1) breakthrough from the solid phase sorbent, or 2) loss during evaporation of the excess solvent. Of these two potential problems, breakthrough may be the most common. This may be caused by variations between different manufacturers of C18 cartridges or variations in lots that have been produced.

What can be done to avoid breakthrough?

1. Breakthrough occurs when the analyte is not sufficiently retained on the sorbent. This can be strongly influenced by the speed at which the sample is pulled through the sorbent. Slowing down the flow rate will increase the contact time between the analyte and the sorbent, allowing for better retention.

2. Breakthrough is related to the capacity of the SPE device. Fortifying LFBs with lower amounts of the analytes can improve recovery. A suggested concentration is 1 µg/L each of 2,4- and 2,6-dinitrotoluene. If you are getting poor recoveries, spike less, not more.

3. All SPE products are not created equal. Some brands are more effective than others. Shopping around can help. Once you find a product you like, stay with that product and always track lot numbers. Recently, a product has been developed and is expected to be commercially available by July 2001 which is specifically designed to help minimize this dinitrotoluene breakthrough (JT Baker, BAKERBOND *Speedisk*® Extraction Disks C18: High Capacity, product # 8055-07, or equivalent).

What if breakthrough is not the problem?

1. The other area where losses may occur is during the solvent evaporation step. 2,4- and 2,6-dinitrotoluene are some of the more volatile analytes in the method. To evaluate this step, fortify a 50/50 mixture of ethyl acetate and methylene chloride with your analytes. Use a solvent volume that is representative of your extract volume. Evaporate this mixture to 1 mL by your typical procedure. If recoveries are low, slow down your evaporation step. A suggested evaporation rate is 0.2 mL/min at approximately 40°C.

I get poor recoveries of the internal standards in Method 525.2.

There are multiple causes of poor recovery for these compounds. Some are listed here:

1 These internal standards are extremely hydrophobic. They would rather be on the glass bottle than dissolved in water. Therefore all the glassware that the sample touches must be rinsed with extraction solvent and added to the extract. The sodium sulfate drying column must also be well rinsed and the solvent added to the extract.

2. If the sample was not properly dechlorinated, and the sample still contains free chlorine at the time the internal standards are spiked, the internal standards react with the chlorine and will not be recovered.

3. The internal standards can be oxidized during the cartridge or disk drying step (just prior to elution). Do not dry the sorbent too long. Ten minutes is enough, and longer drying times can cause oxidation of the internal standards.

4. The internal standards can photodegrade. Be aware of this and limit exposure of samples and extracts to direct light. Use amber sample bottles and extract vials if possible. Some examples for controlling light are: 1) if you process samples in the hood, leave the hood light off and the room lights on, 2) if you process samples in a room with windows, consider leaving the room lights off, 3) if the lab has separate banks of lights on separate switches, leave the lights off on one side of the room. How this can be handled depends on the physical set-up of the laboratory. However, please do not create safety issues by trying to work in near darkness. That degree of light control is not required to get good recoveries.

6.5 Additional Notes on the Analysis of Nitrobenzene by Method 524.2

Since beginning preparation for UCMR, some laboratories have reported that they cannot meet MRL requirements for nitrobenzene. Nitrobenzene does exhibit poor purging efficiency, however adequate data can be obtained if certain guidelines are followed.

1. The purge and trap unit must contain plumbing that is all glass lined or made of Silcosteel®. Many older purge and trap units are glass-lined. Newer ones may be made of Silcosteel® or may just be stainless, or have sections of plumbing that are stainless. If you are having trouble with nitrobenzene, your first step should be to find out what the plumbing in your purge and trap unit is made of. Units that are not glass lined or Silcosteel® may be retrofitted.

2. Purge a 5 mL sample in a 5 mL purge vessel. Purging efficiency can drop dramatically when you purge a larger sample, *i.e.* 25 mL. Never purge a 10 mL sample in a 25 mL purge vessel. This "modification" seems to be gaining popularity in many laboratories. This is not currently a legal modification and it has a negative effect on purging efficiency. When the purging efficiency is already low, method modifications that make it even lower are disastrous. The lower the purging efficiency, the higher the detection or reporting limit.

3. Contact the manufacturer of your purge and trap unit. Some manufacturers are already aware of instrument parameters or minor modifications that will help you to get acceptable data for nitrobenzene.

6.6 Additional Notes on Methods 526 and 528

Some laboratories have raised concerns regarding some of the requirements of Methods 526 and 528. These are both solid phase extraction methods that are similar in many ways to Method 525.2. One way that these methods differ from Method 525.2 is that the internal standards are added at the end of the sample processing.

Other questions that have arisen are:

Why can't I use a SPE disk for Method 528?

The solid phase sorbent used for Method 528 is not available in disk format. Phenols are highly polar and water soluble, and are not well retained on other sorbents.

Do I need to use all three internal standards in Method 526 if no analytes are being calculated from the third internal standard?

No, you do not need to use the third internal standard, if the second surrogate can be correctly quantitated off the second internal standard.

Should I use a packed or open liner if I do PTV injection for Method 526?

During the method development an open 0.5 mm i.d. liner was used. In general, packing materials add to the potential for active sites.

Why does Method 526 have so many additives added to the sample at the time of collection?

One of the common properties among the analytes in Methods 526 is their tendency to be unstable in water. In order to preserve the analytes from time of collection to time of analysis, the analytes need to be protected from hydrolysis (both pH and metal catalyzed), chlorination from free chlorine, and microbial degradation. As a result, the method calls for a buffer to standardize and hold the pH, EDTA to complex metal ions, ascorbic acid to eliminate free chlorine, and a microbial inhibitor.

When I extract LFBs for Method 528, I get little or no recovery of 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol and pentachlorophenol. The other analytes are well recovered. What's wrong?

The three compounds listed are strong acids. They can only be extracted if the water sample is acidified. This includes LFBs.

When I try to elute the cartridges for Method 528, I have a hard time pulling the methylene chloride through. My extracts also have a large water layer. Sometimes my recoveries are poor.

The cartridges must be dry before they are eluted. You can tell that they are fully dried because the color will change from a dark brown to a light tan. Drying times will vary because of the strength of the vacuum source and the number of cartridges being processed at one time. Drying the cartridges too long is not an issue for this method. During method development samples were dried under vacuum for several hours with no analyte loss.

Section 7. Reporting

7.1 Public Water Systems Reporting to EPA

The appropriate and consistent reporting of UCMR monitoring data from the participating PWSs is critical to EPA's efforts to evaluate the data for future regulatory development. The UCMR monitoring data are reported according to specific "data elements" through the Safe Drinking Water Accession and Review System (SDWARS/UCMR) for inclusion in the National Drinking Water Contaminant Occurrence Database (NCOD).

The PWSs are required to report the UCMR monitoring data to EPA for evaluation (§141.35(a)). The UCMR contaminant occurrence data will be included in the NCOD and will be made available to the public. For a detailed description of reporting responsibilities and requirements, refer to the *Unregulated Contaminant Monitoring Regulation Reporting Guidance* (EPA-815-R-01-029), the *Unregulated Contaminant Monitoring Regulation Guidance for Operators of Public Water Systems Serving 10,000 or Fewer People* (EPA 815-R-01-002), and the *Reference Guide for the Unregulated Contaminant Monitoring Regulation* (EPA 815-R-01-023), all available from the EPA Water Docket, telephone (202) 260-3027, Docket Number W-98-02 or from the internet at http://www.epa.gov/safewater/ucmr.html.

EPA is taking a "one-entry" approach to the electronic reporting process to improve reporting quality, reduce reporting errors, and reduce the time involved in investigating and correcting errors at all levels (laboratory, system, State and EPA), thereby making the data more useful earlier. The SDWARS/UCMR electronic reporting system became fully operational on October 1, 2001. A PWS should instruct the agent or organization that conducts the testing and laboratory analysis for the unregulated contaminants to enter the data into the UCMR electronic reporting system. EPA will provide electronic forms via its Internet website or via "batch" electronic data transfer following a format specified by EPA. The PWS can instruct the laboratory to enter the UCMR results directly into the electronic template, after which the PWS can review the results on-line and electronically indicate its approval to submit the data to EPA. PWSs can also require their laboratories to receive their approval before the UCMR results are posted on the EPA electronic posting system.

Results of each analysis must be reported to EPA within 30 days following the month the PWS receives them from the laboratories (§141.35 (c)). For UCMR monitoring data received at the PWS from the laboratory in 2001, a reporting deadline of April 30, 2002 is being strongly considered and will likely be promulgated in supplemental UCMR Federal Register notice early in the 2002 calendar year (for the latest information, closely monitor the UCMR web site at: www.epa.gov/safewater/ucmr.html). For small systems, EPA will enter and report the results directly to its electronic reporting system through its contract laboratories. EPA will place all data received from laboratories and PWSs into the National Drinking Water Contaminant Occurrence Database (NCOD), which will be updated four times a year, after a 60-day quality control review.

Monitoring data for all contaminants must be reported according to contaminant type and must include the 16 specific data elements for each contaminant (§141.35 (d)(3)). Please note that EPA has reserved data element 17, since it is specifically related to microbiological contaminants. These data elements, with their revised definitions, are listed in Table 7.1. If a PWS chooses to report multiple results for a particular contaminant (for example, because more than one laboratory analyzed the samples collected under §141.40, or because multiple samples

were collected during the monitoring period at the same sampling point), only the highest reported value will be used as the official result. PWSs retain final responsibility to ensure that all monitoring data is reviewed, approved and reported to the EPA.

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

The analytical methods approved under the UCMR for measuring the DCPA mono- and di-acid degradates do not, as approved, allow for the identification and quantification of the individual acids. To provide for the consistent reporting of results to the NCOD and to avoid confusion, EPA is specifying that the single analytical result obtained from these methods should be reported as total DCPA mono- and di-acid degradates. As a result, data element 5, Contaminant/Parameter, will not have as acceptable values "DCPA mono-acid degradate" or "DCPA di-acid degradate." Instead, the appropriate acceptable value for this data element will be "Total DCPA mono- and di-acid degradates."

7.1.2 Reporting of Data Below the Specified Minimum Reporting Level

Under the final UCMR (1999) List 2 Rule (66 *FR* 2273), laboratories can no longer report results obtained below the specified MRL of each contaminant.

7.1.3 Reporting of Water Quality Parameter Data

Because EPA has eliminated pH as a water quality parameter for chemical contaminants, there is no longer a need to report these data with chemical contaminant results at this time.

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 Manual and includes 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 the matrix conductance data are not reported to EPA since they were not promulgated as specific data reporting elements. In the rare situation in which the field sample selected for the MS/MSD pair required pretreatment, these QC data would be reported to SDWARS/UCMR for that respective analysis batch.

7.1.5 Reporting of Presence/Absence

The Presence/Absence data element is being reserved for potential future use, as all of the contaminants currently being monitored can be accurately and precisely quantified. Therefore, the presence or absence of a contaminant does not need to be reported. This data element is being reserved for contaminants and not deleted in order to permit the use of this data element if warranted in future regulations.

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

The results of UCMR monitoring will be reportable through the Consumer Confidence Reports (CCR), as required by SDWA §1441(c)(4)(B) and the Consumer Confidence Reports regulation (63 *FR* 44512), as well as through the revised Public Notification Rule (PNR) (65 *FR* 25982). It is anticipated that reporting through the PNR and CCR rules would satisfy the notification provision for unregulated contaminants. Failure to monitor for unregulated contaminants required through the UCMR would be reportable under the PNR.

The results reported through the PNR and CCR rules should be based on the same monitoring data EPA and the States will receive. UCMR data will be submitted to SDWARS for inclusion in the NCOD. Information in the NCOD will be available to the public.

Reporting of unregulated contaminants not on the UCMR (1999) List is not required by EPA under the CCR or PNR, except as directed by the State. Therefore, any "emerging" contaminants of local or State concern alternatively could be reported voluntarily to the NCOD. This reporting would assist in the determination of whether an emerging contaminant is a problem of national extent and should be considered for health-based standards or advisories.

Large PWSs may also wish to report previously collected data on the occurrence of UCMR (1999) List 1 contaminants in lieu of participating in Assessment Monitoring. Grandparenting data is allowed but in order to ensure the quality of data included in the NCOD, EPA is requiring that any data previously collected and submitted in lieu of UCMR monitoring must meet *all* analytical method and quality control requirements specified in the UCMR (§141.35(g)).

Table 7.1 Unregulated Contaminant Monitoring Reporting Requirements

Data Element	Definition
1. Public Water System (PWS) Identification Number	The code used to identify each PWS. The code begins with the standard two-character postal State abbreviation; the remaining seven characters are unique to each PWS.
2. Public Water System Facility Identification Number - Sampling Point Identification Number and Sampling Point Type Identification	The sampling point identification number and sampling point type identification must either be static or traceable to previous numbers and type identifications throughout the period of unregulated contaminant monitoring. The sampling point identification number is a three-part alphanumeric designation, made up of: a. The Public Water System Facility Identification Number is an identification number established by the State, or at the State's discretion the PWS, that is unique to the PWS for an intake for each source of water, a treatment plant, a distribution system, or any other facility associated with water treatment or delivery and provides for the relationship of facilities to each other to be maintained; b. The Sampling Point Identification Number is an identification number established by the State, or at the State's discretion the PWS, that is unique to each PWS facility that identifies the specific sampling point and allows the relationship of the sampling point to other facilities to be maintained; and c. Sampling Point Type Identification is one of following: SR - Untreated water collected at the source of the water system facility. EP - Entry point to the distribution system. MD - midpoint in the distribution system where the disinfectant residual would be expected to be typical for the system such as the location for sampling coliform indicator bacteria as described in 40 CFR 141.21. MR - point of maximum retention is the point located the furthest from the entry point to the distribution system which is approved by the State for trihalomethane (THM) (disinfectant byproducts (DBP)) and/or total coliform sampling. LD - location in the distribution system where the disinfectant residual is the lowest which is approved by the State for THM (DBP) and/or total coliform sampling.
3. Sample Collection Date	The date the sample is collected reported as 4-digit year, 2-digit month, and 2-digit day.
4. Sample Identification Number	An alphanumeric value of up to 15 characters assigned by the laboratory to uniquely identify containers or groups of containers containing water samples collected at the same time and sampling point.
5. Contaminant/ Parameter	The unregulated contaminant or water quality parameter for which the sample is being analyzed.

Data Element	Definition
6. Analytical Results - Sign	An alphanumeric value indicating whether the sample analysis result was: (a) (<) "less than" means the contaminant was not detected or was detected at a level "less than" the MRL. (b) (=) "equal to" means the contaminant was detected at a level "equal to" the value reported in "Analytical Result - Value."
7. Analytical Result - Value	The actual numeric value of the analysis for chemical and microbiological results, or the Minimum Reporting Level (MRL) if the analytical result is less than the specified contaminant's MRL.
8. Analytical Result - Unit of Measure	The unit of measurement for the analytical results reported [e.g., micrograms per liter, (µg/L); colony-forming units per 100 milliliters, (CFU/100 mL), etc.].
9. Analytical Method Number	The identification number of the analytical method used.
10. Sample Analysis Type	The type of sample collected. Permitted values include: a. RFS - Raw field sample - untreated sample collected and submitted for analysis under this rule. b. RDS - Raw duplicate field sample - untreated field sample duplicate collected at the same time and place as the raw field sample and submitted for analysis under this rule. c. TFS - Treated field sample - treated sample collected and submitted for analysis under this rule. d. TDS - Treated duplicate field sample - treated field sample duplicate collected at the same time and place as the treated field sample and submitted for analysis under this rule.

Data Element	Definition
11. Sample Batch Identification Number	The sample batch identification number consists of three parts: a. Up to a 10-character laboratory identification code assigned by EPA; b. Up to a 15-character code assigned by the laboratory to uniquely identify each extraction or analysis batch. Within SDWARS/UCMR, this variable is known as the "Batch ID." c. The date that the samples contained in each extraction batch extracted or in an analysis batch were analyzed, reported as an 8-digit number in the form 4-digit year, 2-digit month, and 2-digit day. FOR EXAMPLE: Sample Batch ID#: **KY99999123xyz524220010326** **a. KY99999** Fictitious lab in KY that was assigned this number after participating in a PE study several years ago. **b. 123xyz5242** Just a completely fictitious example of a Method 524.2 analytical batch. It could be anything alphanumeric, 15 characters or less used by your lab to refer to a batch of samples. This would likely be the number used by your LIMS and is the origin of the 15 character or less reference. **c. 20011115** Analysis date of 11/15/2001
12. Minimum Reporting Level	Minimum Reporting Level (MRL) refers to the lowest concentration of an analyte that may be reported. Unregulated contaminant monitoring (UCM) MRLs are established in §141.40 monitoring requirements for unregulated contaminants.
13. Minimum Reporting Level Unit of Measure	The unit of measure to express the concentration, count, or other value of a contaminant level for the Minimum Reporting Level reported [e.g., micrograms per liter, (µg/L); colony-forming units per 100 milliliters, (CFU/100 mL), etc.].

Data Element	Definition
14. Analytical Precision	Precision is the degree of agreement between two repeated measurements and is monitored through the use of duplicate spiked samples. For purposes of the Unregulated Contaminant Monitoring Rule (UCMR), Analytical Precision is defined as the relative percent difference (RPD) between spiked matrix duplicates. The RPD for the spiked matrix duplicates analyzed in the same batch of samples as the analytical result being reported is to be entered in this field. Precision is calculated as Relative Percent Difference (RPD) of spiked matrix duplicates from the mean using: RPD = absolute value of $[(X_1 - X_2) / ((X_1 + X_2)/2)] \times 100\%$ where: X_1 is the concentration observed in spiked field sample minus the concentration observed in unspiked field sample X_2 is the concentration observed in duplicate spiked field sample minus the concentration observed in unspiked field sample
15. Analytical Accuracy	Accuracy describes how close a result is to the true value measured through the use of spiked field samples. For the purposes of the UCMR, accuracy is defined as the percent recovery of the contaminant in the spiked matrix sample analyzed in the same analytical batch as the sample result being reported and calculated using: % recovery = [(amount found in Spiked sample - amount found in sample) / amount spiked] x 100%
16. Spiking Concentration	The concentration of method analytes added to a sample to be analyzed for calculating analytical precision and accuracy where the value reported uses the same unit of measure reported for Analytical Results.
17. Presence/Absence	Reserved

Appendix A

Abbreviations and Acronyms

2,4-DNT	- 2,4-dinitrotoluene
4,4'-DDE	- 4,4'-dichloro dichlorophenyl ethylene, a degradation product of DDT
AOAC	- Association of Official Analytical Chemists
APHA	- American Public Health Association
ASTM	- American Society for Testing and Materials
˚ C	- degrees Celsius
CAS	- Chemical Abstract Service
CCL	- Contaminant Candidate List
CCR	- Consumer Confidence Reports
CFR	- Code of Federal Regulations
CFU	- colony forming unit
CFU/100 mL	- colony forming units per 100 milliliter
CWS	- community water system
DCPA	- dimethyl tetrachloroterephthalate, chemical name of the herbicide dacthal
DCPA mono- and di-acid degradates	- degradation products of DCPA
DDT	- dichloro diphenyl trichloroethane, a general insecticide
DPD	- *N,N*-diethyl-*p*-phenylenediamine, free chlorine colorimetric indicator
EDL	- estimated detection limit
EDTA	- ethlenediaminetetraacetic acid
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
FR	- Federal Register
GC	- gas chromatography, a laboratory method
GC/MS	- gas chromatography/mass spectrometry, a laboratory method
HPLC	- high performance liquid chromatography, a laboratory method
ICCS	- initial calibration check standard
IPC	- instrument performance check
IS	- internal standard
LFB	- laboratory fortified blank
LFM	- laboratory fortified matrix

MCL	- maximum contaminant level
MCT	- matrix conductivity threshold
MDL	- method detection limit
mL	- milliliter
MRL	- minimum reporting level
MS	- sample matrix spike
MSD	- sample matrix spike duplicate
MTBE	- methyl tertiary-butyl ether, a gasoline additive
N	- normality
NCOD	- National Drinking Water Contaminant Occurrence Database
NPDWS	- National Primary Drinking Water Standards
NTIS	- National Technical Information Service
NTNCWS	- non-transient non-community water system
OGWDW	- Office of Ground Water and Drinking Water
OMB	- Office of Management and Budget
PAH	- Polycyclic aromatic hydrocarbon
$PD_{A/H}$	- percent difference (perchlorate peak area to height ratio)
PNR	- Public Notification Rule
PT	- performance testing, synonymous with proficiency testing
PTFE	- polytetrafluoroethylene
PTV	- programmable temperature vaporizing, type of GC sample injection
PWS	- Public Water System
QC	- quality control
RPD	- relative percent difference
SDWA	- Safe Drinking Water Act
SDWARS	- Safe Drinking Water Accession and Review System
SM	- Standard Methods
SPE	- solid phase extraction, a laboratory method
SRF	- State Revolving Fund
SW	- surface water
TDS	- total dissolved solids
UCMR	- Unregulated Contaminant Monitoring Regulation/Rule
VOC	- volatile organic compound
µg/L	- micrograms per liter

Appendix B

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.

Listed contaminant means a contaminant identified as an analyte in Table 1, 141.40(a)(3) of the Unregulated Contaminant Monitoring Rule (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 may 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 could 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 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 and Screening Survey(s)) 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 may 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.

Appendix C

Procedure for Determination of Detection Limits

EPA has removed the reference to the 40 CFR Part 136 Appendix B definition of method detection limit (MDL) in the Appendix to § 141.40. Under the revised UCMR (66 *FR* 2273), laboratories should refer to the detection limit calculations listed in each respective method for the analyte under consideration.