

**Prepared in cooperation with the U.S. Centers for Disease Control and Prevention and the Maine Center for Disease Control and Prevention**

# **Methods of Collection and Quality Assessment of Arsenic Data in Well-Water Supplies in Maine, 2001–2 and 2006–7**

Data Series 1125



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By Charles W. Culbertson, James M. Caldwell, Luther F. Schalk,  
Deana Manassaram, Lorraine C. Backer, and Andrew E. Smith

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Data Series 1125

**U.S. Department of the Interior  
U.S. Geological Survey**

**U.S. Department of the Interior**  
DAVID BERNHARDT, Secretary

**U.S. Geological Survey**  
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## Conversion Factors

International System of Units to U.S. customary units

Multiply	By	To obtain
Length		
micrometer ( $\mu\text{m}$ )	0.00003937	inch (in.)
centimeter (cm)	0.3937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
Volume		
cubic meter ( $\text{m}^3$ )	6.290	barrel (petroleum, 1 barrel = 42 gal)
milliliter (mL)	0.03381	ounce, fluid (fl. oz)
Flow rate		
liter per second (L/s)	15.85	gallon per minute (gal/min)
Mass		
milligram (mg)	0.00003527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound, avoirdupois (oz)

Temperature in degrees Celsius ( $^{\circ}\text{C}$ ) may be converted to degrees Fahrenheit ( $^{\circ}\text{F}$ ) as  
 $^{\circ}\text{F} = (1.8 \times ^{\circ}\text{C}) + 32.$

## Datum

Horizontal coordinate information is referenced to the North American Datum of 1983 (NAD 83).

## Supplemental Information

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius ( $\mu\text{S}/\text{cm}$  at  $25^{\circ}\text{C}$ ).

Concentrations of chemical constituents in water are given in milligrams per liter (mg/L) or micrograms per liter ( $\mu\text{g}/\text{L}$ ).

Turbidity is reported in nephelometric turbidity units (NTU).

Concentrations of bacteria are reported in colony forming units per 100 milliliters (CFU/100 mL).

## Abbreviations

EPA	U.S. Environmental Protection Agency
HETL	Health and Environmental Testing Laboratory, Maine Department of Human Services
NWQL	National Water Quality Laboratory
POE	point of entry
POU	point of use
QA/QC	quality assurance and quality control
UL	Underwriters Laboratories Inc.
USGS	U.S. Geological Survey



# Methods of Collection and Quality Assessment of Arsenic Data in Well-Water Supplies in Maine, 2001–2 and 2006–7

By Charles W. Culbertson,<sup>1</sup> James M. Caldwell,<sup>1</sup> Luther F. Schalk,<sup>1</sup> Deana Manassaram,<sup>2</sup> Lorraine C. Backer,<sup>2</sup> and Andrew E. Smith<sup>3</sup>

## Abstract

The U.S. Geological Survey, in cooperation with the U.S. Centers for Disease Control and Prevention and the Maine Center for Disease Control and Prevention, assessed the chemical characteristics and the occurrence, distribution, and oxidation state of inorganic arsenic in drinking water from selected domestic well-water supplies in Maine in 2001–2 and 2006–7.

The data collected provide support for evaluating arsenic-removal efficiencies of household water-purification systems and provide information to State and local officials that can be used in determining a water-treatment approach for the removal of arsenic from drinking water.

## Introduction

Arsenic is toxic to humans and widespread in surface water and groundwater throughout the United States and world (National Research Council, 1999; Welch and others, 1999; Welch and others, 2000; Focazio and others, 2000; Ryker, 2001; Smedley and Kinniburgh, 2002; DeSimone and others, 2009). Arsenic in surface waters and groundwater aquifers has both anthropogenic and natural sources. Anthropogenic sources include current and historical industrial uses, as well as broad-spectrum uses in the lumber, agriculture, and livestock industries (National Research Council, 2001). Arsenic occurs naturally in the Earth's crust, ranking as the 20th most abundant element, with an average crustal abundance of approximately 2 milligrams per kilogram (0.0002 percent). It is common in weathered volcanic and marine sedimentary rocks containing iron and manganese oxides, in fossil fuels, and in a number of minerals, including arsenopyrite (FeAsS), orpiment (As<sub>2</sub>S<sub>3</sub>), scorodite (FeAsO<sub>4</sub>), and realgar (AsS) (Cullen and Reimer, 1989; Korte and Fernando, 1991; Smedley and Kinniburgh, 2002).

Arsenic in groundwater is primarily inorganic, either in the oxidized state as arsenate [As(V)], the form typical in oxic waters, or the reduced state as arsenite [As(III)], the form typical in anoxic waters (Cullen and Reimer, 1989; Smedley and Kinniburgh, 2002).

Roughly half of the population of Maine derives its drinking water from domestic wells, of which about 75 percent are drilled bedrock wells. Arsenic concentrations above the current U.S. Environmental Protection Agency (EPA) drinking-water standard of 10 micrograms per liter (µg/L or parts per billion) (U.S. Environmental Protection Agency, 2001) have been detected in water from drilled bedrock wells throughout Maine (Marvinney and others, 1994, 1995; Peters and others, 1999; Ayotte and others, 1999, 2003, 2006; Lipfert and others, 2006; Yang and others, 2009; Nielsen and others, 2010; Zheng and Ayotte, 2015; Flanagan and others, 2012). Effective removal of arsenic by household water-purification systems is constrained by the chemical form (for example, oxidation state) in which it occurs, as well as the prevailing chemical characteristics of the well water, including pH, and the presence of competing ions, such as phosphate (U.S. Environmental Protection Agency, 2001).

The U.S. Geological Survey (USGS), in cooperation with the U.S. Centers for Disease Control and Prevention and the Maine Center for Disease Control and Prevention, assessed the chemical characteristics and the occurrence, distribution, and oxidation state of inorganic arsenic in drinking water from selected domestic well-water supplies in Maine in 2001–2 and 2006–7.

The data collected provide support for evaluating arsenic-removal efficiencies of household water-purification systems and provide information to State and local officials that can be used in determining a water-treatment approach for the removal of arsenic from drinking water.

## Purpose and Scope

This report describes methods used to collect arsenic and other water-chemistry data from domestic well water in support of studies evaluating arsenic removal efficiencies of household water purification systems. Data consist of water-chemistry results from samples collected over two time periods: 2001–2 and 2006–7 (also two samples collected

<sup>1</sup>U.S. Geological Survey.

<sup>2</sup>U.S. Centers for Disease Control and Prevention.

<sup>3</sup>Maine Department of Health and Human Services.

in 2008), well characteristics (depth and type), and chemical characteristics of well water from approximately 120 selected domestic wells in known high-arsenic clusters throughout the State of Maine. All data are available in the USGS National Water Information System (NWIS) database, are searchable by USGS site number (U.S. Geological Survey, 2019), and are published in Culbertson and others (2020).

## **Description of Study Area**

The study area includes most of the populated areas in the State of Maine apart from Aroostook County in north-eastern Maine. The samples collected were from single-family dwellings relying on private well water as the primary drinking-water supply; additionally, these households had water-purification systems designed for the removal of arsenic installed either at the well-water point of entry (POE) or point of use (POU). The initial pilot study included 31 households sampled for arsenic and arsenic species in 2001–2, whereas the full-scale study included 120 households sampled in 2006–7. Households were recruited based on their locations in areas of Maine known to have high concentrations of arsenic ( $>10\ \mu\text{g/L}$ ) in groundwater (fig. 1).

## **Methods of Data Collection**

Households with domestic wells included in this study were identified and prescreened prior to both sampling periods by project personnel associated with concurrent studies by the U.S. Centers for Disease Control and Prevention and the Maine Center for Disease Control and Prevention. To be considered for the study, households needed to have well water containing elevated ( $>10\ \mu\text{g/L}$ ) concentrations of arsenic and a previously installed water-purification system designed to remove arsenic; systems used reverse-osmosis, anion-exchange, or adsorption technologies. Most homes tested had point-of-use reverse-osmosis systems installed. Well-water samples (pre-water-treatment system) were collected at the house POE, prior to the pressure tank and household water-treatment system(s), by using the existing domestic well-water pump. Samples collected after water treatment were collected at the kitchen (POU) faucet. A list of constituent names, USGS NWIS parameter codes, and minimum reporting limits for all samples collected for laboratory and field analyses are shown in tables 1 and 2. A summary of field measurements and analytical determinations, along with the entity or entities that analyzed or measured each, is presented in table 3.

## **Measurement of Field Water-Quality Constituents**

Prior to collection of POE well water for arsenic and other chemical analyses, field measurements of temperature, specific conductance, pH, and dissolved oxygen were

measured onsite using a multiparameter water-quality monitor immersed in a flow-through chamber under a steady water flow of approximately 1–1.5 liters per minute. Field measurements and water-chemistry results are published in Culbertson and others (2020). During the 2006–7 sample collection period, low dissolved oxygen readings ( $<1\ \text{mg/L}$ ) were confirmed by an alternate, colorimetric method (CHEMets colorimetric dissolved oxygen test kit, 0–1 part per million; CHEMetrics, Inc., Calverton, Va.). Samples for water chemistry determinations and arsenic analyses were collected after water-quality-monitor readings had stabilized (generally 0.25–0.75 hour). Except for specific conductance, field measurements of the other water-quality constituents in POU water samples were not made because of physical and chemical alterations imparted by the water-treatment systems. Specific conductance, a proxy for total dissolved solids, was measured in both prefiltration and postfiltration water, specifically to assess the performance efficiencies of reverse-osmosis water-treatment systems in removing arsenic (Fravel and Lindsey, 2014).

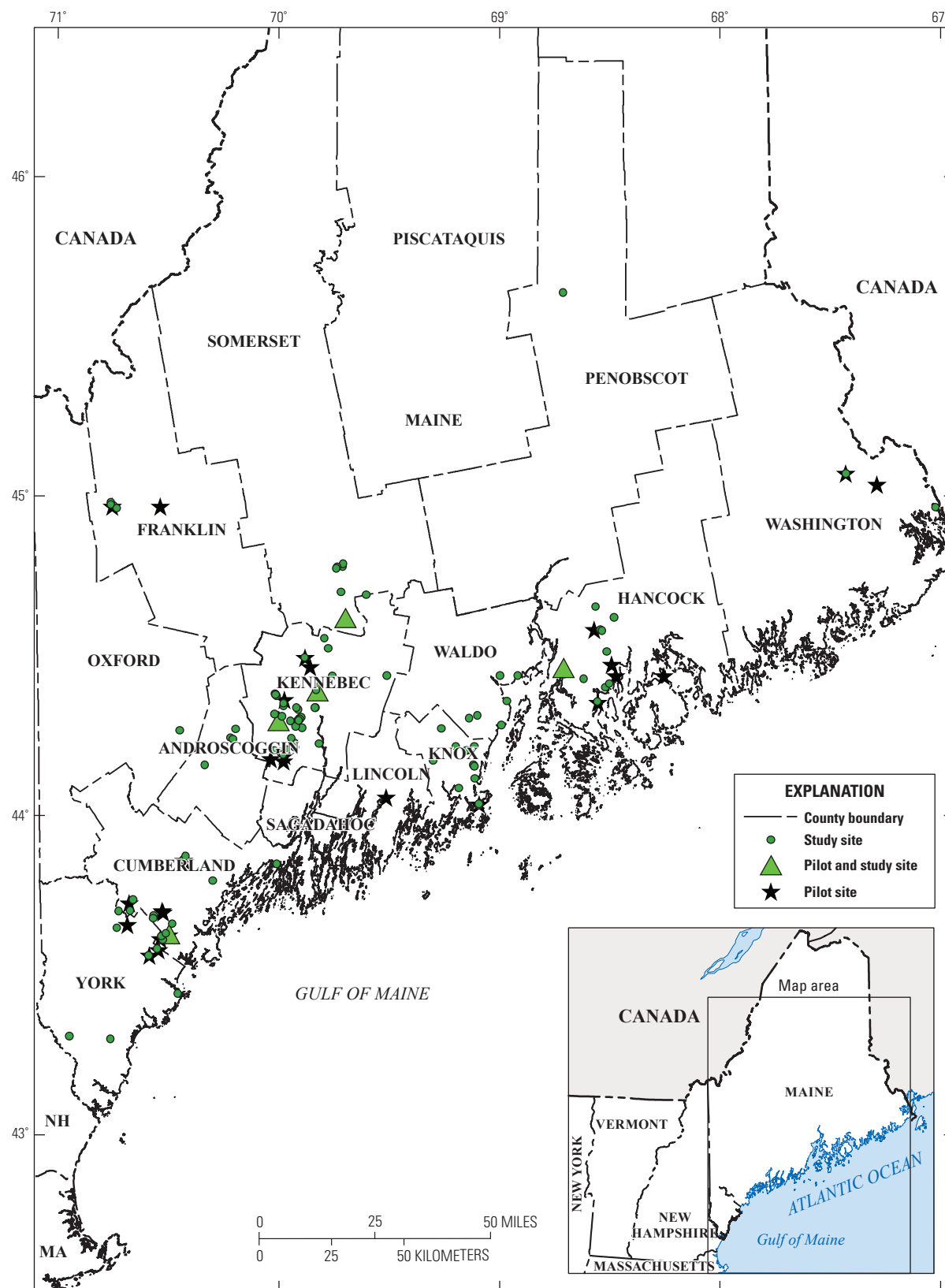
## **Sample Collection for Well-Water Chemical Analyses Performed in the Field**

Upon obtaining stabilized readings of temperature, specific conductance, and pH, well-water samples for field chemical analyses were collected. Field measurements (or analyses) of POE well water included total iron, ferrous iron (FeII), total manganese, reactive phosphorus, sulfate, sulfide, and nitrate. These measurements were made immediately upon sample collection using standard colorimetric methods (American Public Health Association, 1998; Hach Company, 2000). Equipment blanks and sample replicates for quality-control (QC) purposes were collected and analyzed with the field samples. Additionally, during the 2006–7 sample collection period, replicate samples for some constituents were collected for chemical analysis by an alternate laboratory and method (Underwriters Laboratories Inc. [UL], South Bend, Indiana). Results of field chemical and quality-control analyses are published in Culbertson and others (2020).

## **Point-of-Entry and Point-of-Use Sample Collection for Laboratory Chemical Analysis of Well Water**

According to study protocols for the 2006–7 study, two laboratories (Underwriters Laboratories Inc. [UL], South Bend, Indiana, and the Health and Environmental Testing Laboratory [HETL] of the Maine Department of Human Services<sup>4</sup> in Augusta, Maine) were used to measure arsenic concentrations. The laboratory results from these two facilities are published here as a means of comparison of the analytical

<sup>4</sup>Since July 2004, the Maine Department of Health and Human Services.



**Figure 1.** Domestic-well sample locations in Maine, 2001–2 and 2006–7.

**Table 1.** Method information, minimum reporting limits, and U.S. Geological Survey National Water Information System parameter codes for all constituents measured in water samples collected from domestic wells in Maine for laboratory analyses, 2001–2 and 2006–7.

[Samples were collected for laboratory measurements before (point-of-entry, POE) and after (point-of-use, POU) household water-treatment systems. CASRN, Chemical Abstracts Service Registry Number; method reference, indicates the method code number associated with U.S. Environmental Protection Agency analytical methods, Standard Methods for the Examination of Water and Wastewater analytical methods, or U.S. Geological Survey (USGS) National Water Quality Laboratory analytical methods; MCL, maximum contaminant level; SMCL, secondary maximum contaminant level; MRL, minimum reporting limit; N/A, not applicable; mg/L, milligram per liter; CaCO<sub>3</sub>, calcium carbonate; UL, Underwriters Laboratories Inc.; µg/L, microgram per liter; HETL, Maine Department of Human Services Health and Environmental Testing Laboratory; NWQL, National Water Quality Laboratory; As, arsenic; CFU/100 mL, colony forming units (colonies) per 100 milliliters; N, nitrogen; NTU, nephelometric turbidity units]

Constituent	CASRN <sup>1</sup>	Method reference	MCL/SMCL	Unit	MRL	USGS parameter code	USGS method code	Analyzing entity	Analyte description
Alkalinity, total	N/A	2320 B <sup>2</sup>	N/A	mg/L as CaCO <sub>3</sub>	10	29803	TT060	UL	Alkalinity, water, filtered, Gran titration, laboratory, milligrams per liter as calcium carbonate.
Arsenic	7440-38-2	200.8 <sup>3</sup>	10	µg/L	5	01002	PM100	UL	Arsenic, water, unfiltered, micrograms per liter.
Arsenic	7440-38-2	200.8 <sup>3</sup>	10	µg/L	0.5	01002	PLM99	HETL	Arsenic, water, unfiltered, micrograms per liter.
Arsenic	7440-38-2	GF096 <sup>4</sup>	10	µg/L	1.9	01002	GF096	USGS-NWQL	Arsenic, water, unfiltered, micrograms per liter.
Arsenic	7440-38-2	200.8 <sup>3</sup>	10	µg/L	0.5	01000	PLM98	HETL	Arsenic, water, filtered, micrograms per liter.
Arsenite	15502-74-6	200.8 <sup>3</sup>	10	µg/L as As	0.5	62452	PLM98	HETL	Arsenite, water, filtered, micrograms per liter as arsenic.
Arsenate	15584-04-0	200.8 <sup>3</sup>	10	µg/L as As	0.5	62453	PLM98	HETL	Arsenate, water, filtered, micrograms per liter as arsenic.
Fecal coliform	N/A	9222 D <sup>2</sup>	1	CFU/100 mL	1	31616	BAC93	UL	Fecal coliform, M-FC MF (0.45 micrometer) method, water, colony forming units per 100 milliliters.
Fluoride	16984-48-8	300.0 <sup>3</sup>	4	mg/L	0.1	00951	IC075	UL	Fluoride, water, unfiltered, milligrams per liter.
Hardness, total	N/A	2340 B <sup>2</sup>	N/A	mg/L as CaCO <sub>3</sub>	10	00909	TT099	UL	Hardness, water, unfiltered, measured, milligrams per liter as calcium carbonate.
Iron, total	7439-89-6	200.7 <sup>3</sup>	0.3	mg/L	0.02	74010	PLA44	UL	Iron, water, unfiltered, milligrams per liter.
Lead	7439-92-1	200.8 <sup>3</sup>	15	µg/L	5	52298	PM100	UL	Lead, water, unfiltered, micrograms per liter.
Manganese	7439-96-5	200.8 <sup>3</sup>	50	µg/L	5	71883	PM100	UL	Manganese, water, unfiltered, micrograms per liter.
Nitrate	14797-55-8	353.2 <sup>3</sup>	10	mg/L as N	1	00630	CDR25	UL	Nitrate plus nitrite, water, unfiltered, milligrams per liter as nitrogen.
Nitrite	14797-65-0	353.2 <sup>3</sup>	1	mg/L as N	0.1	00615	CDR25	UL	Nitrite, water, unfiltered, milligrams per liter as nitrogen.
pH	N/A	150.1 <sup>3</sup>	6.5–8.5	pH units	N/A	00403	EL022	UL	pH, water, unfiltered, laboratory, standard units.
Sodium	7440-23-5	200.7 <sup>3</sup>	N/A	mg/L	0.1	52290	PLA44	UL	Sodium, water, unfiltered, milligrams per liter.
Sulfate	14808-79-8	300.0 <sup>3</sup>	250	mg/L	10	00946	IC075	UL	Sulfate, water, unfiltered, milligrams per liter.
Turbidity	N/A	180.1 LL <sup>3</sup>	N/A	NTU	0.1	82079	TBD05	UL	Turbidity, water, unfiltered, laboratory, nephelometric turbidity units.
Uranium	7440-61-1	200.8 <sup>3</sup>	30	µg/L	5	63032	PM100	UL	Uranium, water, unfiltered, micrograms per liter.

<sup>1</sup>This report contains CAS Registry Numbers, which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRNs through CAS Client Services.

<sup>2</sup>Analytical method code from “Standard Methods for the Examination of Water and Wastewater” (American Public Health Association, 1998).

<sup>3</sup>Analytical method code from the U.S. Environmental Protection Agency.

<sup>4</sup>Analytical method code from the USGS National Water Quality Laboratory.

**Table 2.** Constituents, U.S. Geological Survey National Water Information System parameter codes, minimum reporting limits, and method information for all water samples collected from domestic wells in Maine for analyses performed in the field, 2001–2 and 2006–7.

[USGS, U.S. Geological Survey; MRL, minimum reporting limit; mg/L, milligram per liter; CASRN, Chemical Abstracts Service Registry Number; N, nitrogen; wu, water, unfiltered; NEWSC, New England Water Science Center; PO4, phosphate; °C, degree Celsius; NA, not applicable; µS/cm at 25 °C, microsiemens per centimeter at 25 °C, microsiemens per centimeter at 25 °C, percent saturation; std units, standard units; gal/min, gallon per minute]

Constituent	USGS parameter code	Units	MRL (mg/L)	CASRN <sup>1</sup>	Analytical		USGS method code	USGS method name	Method description
					method number	reference <sup>2</sup>			
Field chemistry parameters									
Nitrate, water, unfiltered, field, milligrams per liter as nitrogen	99124	mg/L as N	0.1	14797-55-8	8039	CDR23	Nitrate, wu, field, colorimetry, Hach test kit (NEWSC)	Field determination of nitrate in unfiltered water by cadmium reduction colorimetry method with Hach Test Kit.	
Sulfate, water, unfiltered, field, milligrams per liter	99127	mg/L	1	14808-79-8	8051	CL203	Sulfate, wu, field, colorimetry, Hach test kit (NEWSC)	Field determination of sulfate in unfiltered water with Hach Test Kit, SulfaVer 4 method.	
Orthophosphate, water, unfiltered, milligrams per liter as PO4	99126	mg/L	0.01	7723-14-0	8048	ASC20	Phosphate (orthophosphate), wu, field, colorimetry, Hach test kit (NEWSC)	Field determination of phosphate in unfiltered water with Hach Test Kit, PhosVer 3 (ascor- bic acid) method.	
Iron(II), water, unfiltered, field, milligrams per liter	99128	mg/L	0.01	15438-31-0	8146	PHEN5	Iron(II), wu, field, Hach colorimetry	Iron(II) in unfiltered water determined in the field by the 1,10-phenanthroline method with portable colorimeter using Hach AccuVac ampules. Range up to 3.00 mg/L.	
Iron, water, unfiltered, field, milligrams per liter	99138	mg/L	0.01	7439-89-6	8008	CL204	Iron, total, wu, field, colo- rimetry, Hach test kit (NEWSC)	Field determination of total iron in unfiltered water with Hach Test Kit, FerroVer method. Range up to 3.00 mg/L.	
Manganese, water, unfil- tered, field, milligrams per liter	53570	mg/L	0.1	7439-96-5	8034	CL205	Manganese, wu, field, colorimetry, Hach test kit (NEWSC)	Field determination of manganese in unfiltered water with Hach Test Kit, Periodate Oxida- tion method. Range up to 20.00 mg/L.	
Sulfide, water, unfiltered, field, milligrams per liter	99119	mg/L	0.001	18496-25-8	8131	MB002	Sulfide, field, by Hach methy- lene blue colorimetric method test kit	Sulfide, field, by Hach methylene blue colori- metric method test kit.	

**Table 2.** Constituents, U.S. Geological Survey National Water Information System parameter codes, minimum reporting limits, and method information for all water samples collected from domestic wells in Maine for analyses performed in the field, 2001–2 and 2006–7.—Continued

[USGS, U.S. Geological Survey; MRL, minimum reporting limit; mg/L, milligram per liter; CASRN, Chemical Abstracts Service Registry Number; N, nitrogen; wu, water, unfiltered; NEWSC, New England Water Science Center; PO4, phosphate; °C, degree Celsius; NA, not applicable; µS/cm at 25 °C, microsiemens per centimeter at 25 degrees Celsius; ppm, part per million; % saturation, percent saturation; std units, standard units; gal/min, gallon per minute]

Constituent	USGS parameter code	Units	MRL (mg/L)	CASRN <sup>1</sup>	Analytical method number reference <sup>2</sup>	USGS method code	USGS method name	Method description
Field water-quality parameters								
Temperature, water, degrees Celsius	00010	° C	NA	NA	NA	THM01	Temperature, water, wu, field, thermistor, multiparameter sonde (NEWSC)	Field determination of water temperature (° C) in unfiltered water with multiparameter sonde.
Specific conductance, water, unfiltered, microsiemens per centimeter at 25 degrees Celsius	00095	µS/cm at 25 °C	NA	NA	NA	SC001	Specific conductance, wu, field, electrometry, multiparameter sonde (NEWSC)	Field determination of specific conductance in unfiltered water by electrometry with multiparameter sonde.
Dissolved oxygen, water, unfiltered, milligrams per liter	00300	mg/L	0.01	7782-44-7	NA	LUMIN	Dissolved oxygen, wu, field, luminescence sensor, multiparameter sonde (NEWSC)	Field determination of dissolved oxygen in unfiltered water by luminescence-based sensor with multiparameter sonde.
Dissolved oxygen, water, unfiltered, milligrams per liter	00300	ppm	0.01	7782-44-7	NA	RHODA	Dissolved oxygen, wu, field, CHEMetrics 0–1 ppm kit (NEWSC)	Field determination of dissolved oxygen in unfiltered water with CHEMetrics indigo carmine test kit, 0–1 ppm (mg/L).
Dissolved oxygen, water, unfiltered, percent of saturation	00301	% saturation	0.1	7782-44-7	NA	CAL35	Dissolved oxygen solubility, barometric pressure, temperature, calculation, multiparameter sonde (NEWSC)	Dissolved oxygen solubility in natural waters, calculation, multiparameter sonde.
pH, water, unfiltered, field, standard units	00400	std units	NA	NA	NA	PROBE	pH, wu, field, electrometry, multiparameter sonde (NEWSC)	Field determination of pH in unfiltered water by electrometry with multiparameter sonde.
Flow rate, instantaneous, gallons per minute	00059	gal/min	NA	NA	NA	G0009	Instantaneous flow rate, gallons per minute	Field measured flow rate; timed measurement of volume.

<sup>1</sup>This report contains CAS Registry Numbers, which is a Registered Trademark of the American Chemical Society. CAS recommends the verification of the CASRN through CAS Client Services.

<sup>2</sup>Hach DR/2010 Spectrophotometer Procedures Manual, Hach Company (2000).

**Table 3.** Constituents, field parameters, arsenic speciation, and measuring entities for water samples collected from domestic wells in Maine, 2001–2 and 2006–7.

[USGS, U.S. Geological Survey; Maine HETL, Maine Department of Human Services Health and Environmental Testing Laboratory, Augusta, Maine; Underwriters Laboratories, Underwriters Laboratories Inc., South Bend, Indiana; NWQL, National Water Quality Laboratory, Denver, Colorado; DO, dissolved oxygen]

Constituent	USGS parameter code	Laboratory or measuring entity			
		Maine HETL 2001–2; 2006–7	Underwriters Laboratories 2006–7	USGS- NWQL 2001–2	USGS field measurements 2001–2; 2006–7
Analytical determinations					
Alkalinity	29803		X		
Arsenic, total (unfiltered)	01002	X	X	X	
Arsenic, dissolved (filtered)	01000	X			
Fecal coliform	31616		X		
Fluoride	00951		X		
Iron, total	74010/99138		X		X
Iron, ferrous (Fe II)	99128				X
Iron, ferric (Fe III)	51279				X
Lead	52298		X		
Manganese	71883/53570		X		X
Nitrate	00630/99124		X		X
Nitrite	00615		X		
pH (lab)	00403		X		
Phosphate	99126				X
Sodium	52290		X		
Sulfate	00946/99127		X		X
Sulfide	99119				X
Total hardness	00909		X		
Turbidity	82079		X		
Uranium	63032		X		
Field parameters					
Dissolved oxygen	00300				X
Percent DO saturation	00301				X
pH (field)	00400				X
Specific conductance	00095				X
Temperature	00010				X
Arsenic speciation—field preparation					
Total arsenic, raw well water					X
Filtered arsenic, raw well water					X
Arsenite [As(III)], raw well water; field ion exchange					X
Arsenate [As(V)], raw well water; field ion exchange					X
Filtered arsenic, well water, post-household filtration system					X
Arsenite [As(III)], well water, post-household filtration system					X

results for many of the same constituents, and to help inform the evaluation of the water treatment system performance in both studies.

POE and POU samples were collected for laboratory chemical analysis by following standard USGS protocols (U.S. Geological Survey, variously dated) and that of the Underwriters Laboratories Inc. (UL) DrinkWell well-water testing system. Once stable readings for temperature, specific conductance, and pH were achieved (0.25–0.75 hour), POE samples were collected. Source water for POU samples (kitchen faucet) was run continuously for approximately 20 minutes prior to sample collection. The faucet aerator on the POU faucet was removed to minimize sample aeration. Faucets were disinfected with alcohol (isopropanol) wipes prior to sample collection. Bottle types and preservatives varied depending on the analysis to be performed. A complete list of constituents and minimum reporting limits is shown in table 1. All samples collected for analysis at UL were chilled to 4 degrees Celsius and shipped overnight on the day of collection. Samples were analyzed within appropriate, analyte-specific holding times, according to the laboratories' standard operating procedures. Published arsenic results are labeled as either "pre-household filtration" (POE) or "post-household filtration" (POU) samples (Culbertson and others, 2020).

### **Point-of-Entry and Point-of-Use Sample Collection for Laboratory Arsenic and Arsenic Speciation (Valence) Analysis**

Household well-water samples for unfiltered arsenic, filtered arsenic (0.45- $\mu$ m filter), and filtered As(III), and As(V) were collected at the POE and processed onsite prior to laboratory analysis. Laboratory analyses of arsenic, for both the 2001–2 and 2006–7 sampling periods, were performed by the Health and Environmental Testing Laboratory (HETL) of the Maine Department of Human Services<sup>5</sup> in Augusta, Maine, and are published in Culbertson and others (2020). A subset of sample replicates (paired samples) were collected during the 2001–2 sampling period and analyzed by both the HETL and the USGS National Water Quality Laboratory (NWQL). Replicate results are identified as such in Culbertson and others (2020) by the "SampleType" field. In both sampling periods, the effectiveness of household water-treatment systems in removing arsenic was assessed by comparing POU samples (post-arsenic treatment system, collected at the kitchen faucet) to raw, untreated POE samples. Throughout the 2006–7 sampling period, replicate (paired) samples were collected for pre- and post-arsenic treatment system assessment and analyzed by both UL and HETL. During preliminary stages of the 2006–7 sampling period, post-household treatment system (POU) samples were analyzed for filtered arsenic as well as As(III); however, there was concern that any As(III) present could be oxidized by interaction with the treatment system and thus be

reported as As(V). Consequently, the sample collection protocol was modified mid-study, whereby, an additional replicate sample for As(III) determination on the raw (POE) well water was collected. Thereafter, instead of two sample replicates, As(III) determinations on raw POE well water were performed in triplicate. All samples for As(III) determinations on raw water were collected in triplicate during the 2001–2 sample period. Except where noted, the only arsenic determination on post-household filtration (POU) water was for filtered arsenic.

### **Arsenic Sample Preparation and Preservation**

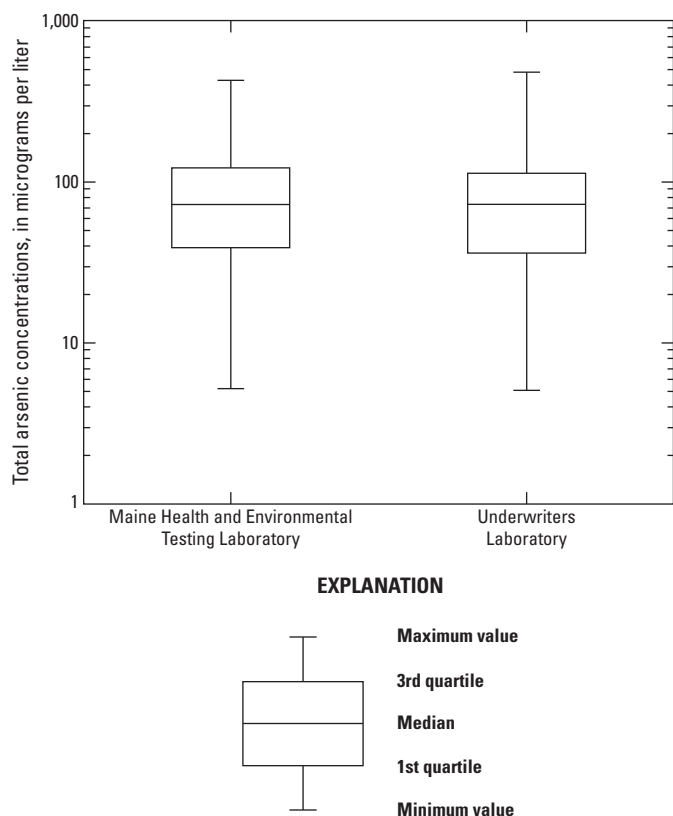
Sample preparation for arsenic analysis included acidification to pH <2 (Ultrex, ultra-high-purity nitric acid [HNO<sub>3</sub>], J.T. Baker Chemical Company, Phillipsburg, N.J.) for filtered arsenic sample analysis, and filtration through 0.45  $\mu$ m nylon syringe filters, followed by acidification (5 percent [volume per volume] sulfuric acid [0.05 percent <sub>final</sub> H<sub>2</sub>SO<sub>4</sub>], Baker Insta-Analyzed, J.T. Baker Chemical Company) for filtered arsenic sample analysis and arsenic speciation (valence) analysis. Samples for As(III) and As(V) analysis were prepared onsite by eluting aliquots of the filtered/acidified samples through anion-exchange chromatography columns according to the method described by Ficklin (1983). Once aliquots for arsenic valence analysis were collected, the samples for filtered arsenic analysis were further acidified to pH <2 with HNO<sub>3</sub> (Ultrex). This step was necessary to prevent HNO<sub>3</sub> damage to the anion-exchange resin. The H<sub>2</sub>SO<sub>4</sub> step was necessary to adjust the pH of the source water to approximately pH 4. At this pH, the anion-exchange columns retain the negatively charged As(V) species while allowing the neutral As(III) species to pass through in the filtrate (Ficklin, 1983; Edwards and others, 1998; Wilkie and Hering, 1998). The filtrates were collected and further acidified to pH <2 with HNO<sub>3</sub>. Any arsenic measured in these samples is interpreted to be As(III) (table 3). As(V) was determined as the difference between filtered arsenic and As(III). Total particulate arsenic was determined as the difference between unfiltered and filtered arsenic samples.

Arsenic analyses were performed by three laboratories: (1) the Maine Department of Human Services HETL, Augusta, Maine, (2) UL, South Bend, Ind., and (3) the USGS NWQL, Denver, Colorado (table 3). Interlaboratory comparisons are displayed in figures 2 and 3 and are available in the published data (Culbertson and others, 2020).

### **Preparation of Field Anion-Exchange Chromatography Columns for Arsenic Speciation Analysis**

Anion-exchange chromatography columns for the separation of As(III) and As(V) in the field were prepared in the laboratory prior to well-water sample collection. AG 1 anion-exchange resin (50–100 mesh; Bio-Rad Laboratories,

<sup>5</sup>Since July 2004, the Maine Department of Health and Human Services.

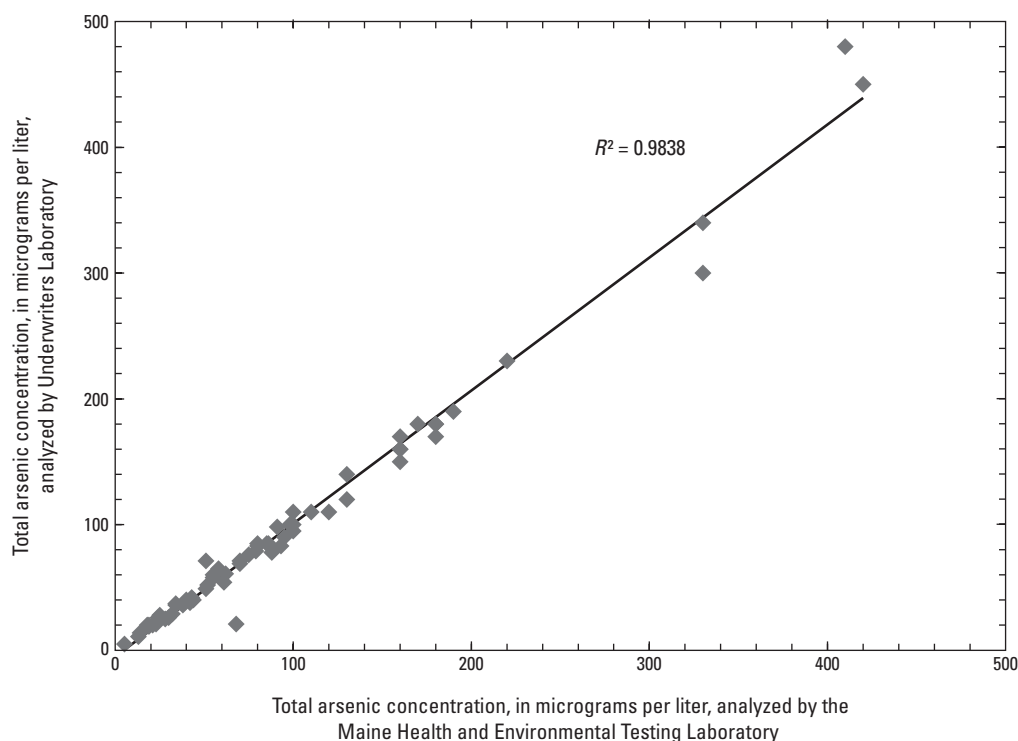


**Figure 2.** Distribution of unfiltered arsenic concentrations in paired samples analyzed by the Maine Department of Human Services Health and Environmental Testing Laboratory (HETL) and Underwriters Laboratories Inc. (UL), 2006–7. Modified from Nielsen and others (2010).

Hercules, Calif.) was obtained in the chloride form and converted to the acetate form prior to column packing (Wilkie and Hering, 1998). Poly-Prep 0.8 x 4-centimeter polypropylene chromatography columns (Bio-Rad Laboratories, Hercules, Calif.) were slurry-vibra-packed with approximately 2 milliliters of the converted anion-exchange resin and excess deionized water to keep the resin moist (vibra-packing of the resin slurry was used to eliminate channeling in the resin bed). Columns were capped and refrigerated until use in the field. The anion-exchange columns were prepared in batches of 100; column efficiency tests were performed on each batch. Representative anion-exchange column efficiencies are shown in tables 4 and 5.

## Quality Assessment of Laboratory Arsenic Analysis

Arsenic in all samples was determined by inductively coupled plasma-mass spectrometry (EPA Method 200.8; U.S. Environmental Protection Agency, 1994) by the HETL (table 1). As a QA check of samples analyzed by the HETL, replicate samples, collected during 2001–2, were analyzed for unfiltered arsenic by an alternate method: USGS Method GF096: “Arsenic Determination by Hydride Generation/Atomic Absorption Spectrometry” by the USGS NWQL (table 1).



**Figure 3.** Arsenic concentrations in paired samples analyzed by the Maine Department of Human Services Health and Environmental Testing Laboratory and Underwriters Laboratories Inc., 2006–7. Modified from Nielsen and others (2010).

**Table 4.** Representative efficiency of anion-exchange chromatography resin for the field separation of As(III) and As(V) for single arsenic species recovery.

[As, arsenic; µg/L, microgram per liter As(III), arsenic-3 or arsenite; As(V), arsenic-5 or arsenate]

Replicate number	As added (µg/L)	As recoverable (µg/L)
As(III)		
Replicate 1	62	61
Replicate 2	62	61
Replicate 3	62	57
As(V)		
Replicate 1	67	<0.5
Replicate 2	67	<0.5
Replicate 3	67	<0.5

**Table 5.** Representative efficiency of anion-exchange chromatography resin for the field separation of As(III) and As(V) for As(III)+As(V) mix recovery.

[As, arsenic; As(III), arsenic-3 or arsenite; µg/L, microgram per liter; As(V), arsenic-5 or arsenate]

Replicate number	As(III) added (µg/L)	As(V) added (µg/L)	As recoverable (µg/L)
Replicate 1	33	34	31
Replicate 2	33	34	32

An assessment of the HETL and UL arsenic analyses was derived from a 2006–7 comparison of arsenic determinations reported by each laboratory for replicate (paired) samples. Sixty-four samples from wells with concentrations of arsenic ranging from 5 to 420 micrograms per liter (µg/L) were replicated (sampled at the same time and from the same source, preserved in the same way, and analyzed by using EPA Method 200.8). The results of the analyses for unfiltered arsenic from the two laboratories were compared to assure quality in the analytical process (figs. 2 and 3; Nielsen and others, 2010; Culbertson and others, 2020).

In general, the average difference in concentration for the 64 sample pairs analyzed by HETL and UL, expressed as the absolute value of the relative percent difference between the samples, was about 7.6 percent ( $(|[\text{HETL}-\text{UL}]|/([\text{HETL}+\text{UL}]/2)) \times 100$ ). A comparison of concentrations for the paired samples (fig. 3) shows that, for the most part, this difference is primarily at the upper end of the range in concentrations of arsenic (with one exception at the lower end of the range). The range in relative percent difference is 0 to 105 percent, although only two samples have a relative percent difference greater than 16.7 percent. According to a one-way analysis of variance of the datasets, the concentration means are not significantly different at the  $p=0.05$  level (Nielsen and others, 2010).

**References Cited**

American Public Health Association, 1998, Standard methods for the examination of water and wastewater (20th ed.): Washington, D.C., American Public Health Association, American Water Works Association, and Water Environment Federation, 1,325 p.

Ayotte, J.D., Montgomery, D.L., Flanagan, S.M., and Robinson, K.W., 2003, Arsenic in groundwater in eastern New England—Occurrence, controls, and human health implications: *Environmental Science & Technology*, v. 37, no. 10, p. 2075–2083.

Ayotte, J.D., Nielsen, M.G., Robinson, G.R., and Moore, R.B., 1999, Relation of arsenic, iron, and manganese in ground water to aquifer type, bedrock lithogeochemistry, and land use in the New England Coastal Basins Study Unit: U.S. Geological Survey Water-Resources Investigations Report 99–4162, 61 p.

Ayotte, J.D., Nolan, B.T., Nuckols, J.R., Cantor, K.P., Robinson, G.R., Jr., Baris, D., Hayes, L., Karagas, M.R., Bress, W., Silverman, D.T., and Lubin, J.H., 2006, Modeling the probability of arsenic in groundwater in New England as a tool for exposure assessment: *Environmental Science & Technology*, v. 40, no. 11, p. 3578–3585.

Culbertson, C.W., Caldwell, J.M., Schalk, L.F., and Medalie, L., 2020, Arsenic datasets and other physical and chemical measurements for selected domestic well-water supplies in Maine—2001–2 and 2006–7: U.S. Geological Survey data release, <https://doi.org/10.5066/P9X5HVDF>.

Cullen, W.R., and Reimer, K.J., 1989, Arsenic speciation in the environment: *Chemical Reviews*, v. 89, no. 4, p. 713–764.

DeSimone, L.A., Hamilton, P.A., and Gilliom, R.J., 2009, Quality of water from domestic wells in principal aquifers of the United States, 1991–2004—Overview of major findings: U.S. Geological Survey Circular 1332, 48 p.

Edwards, M., Patel, S., McNeill, L., Chen, H., Frey, M., Eaton, A.D., Antweiler, R.C., and Taylor, H.E., 1998, Considerations in As analysis and speciation: *Journal American Water Works Association*, v. 90, no. 3, p. 103–113.

Ficklin, W.H., 1983, Separation of arsenic(III) and arsenic(V) in ground waters by ion-exchange: *Talanta*, v. 30, no. 5, p. 371–373.

Flanagan, S.M., Ayotte, J.D., and Robinson, G.R., Jr., 2012, Quality of water from crystalline rock aquifers in New England, New Jersey, and New York, 1995–2007 (ver. 1.1, April 2018): U.S. Geological Survey Scientific Investigations Report 2011–5220, 104 p., accessed October 29, 2019, at <https://doi.org/10.3133/sir20115220>.

- Focazio, M.J., Welch, A.H., Watkins, S.A., Helsel, D.R., and Horn, M.A., 2000, A retrospective analysis on the occurrence of arsenic in ground-water resources of the United States and limitations in drinking-water-supply characterizations: U.S. Geological Survey Water-Resources Investigations Report 99–4279, 21 p.
- Fravel, H.G., Jr., and Lindsey, K., 2014, Understanding salt passage vs. salt rejection in reverse osmosis systems: American Membrane Technology Association blog, August 29, 2014, accessed August 2017 at <https://www.amtaorg.com/understanding-salt-passage-vs-salt-rejection-in-reverse-osmosis-systems>.
- Hach Company, 2000, Procedures manual, DR2010/DR2400 Spectrophotometer (7th ed., October 2000): Loveland, Colo., Hach Company, 872 p.
- Korte, N.E., and Fernando, Q., 1991, A review of arsenic (III) in groundwater: Critical Reviews in Environmental Science and Technology, v. 21, no. 1, p. 1–39.
- Lipfert, G., Reeve, A.S., Sidle, W.C., and Marvinney, R.G., 2006, Geochemical patterns of arsenic-enriched ground water in fractured crystalline bedrock, Northport, Maine, USA: Applied Geochemistry, v. 21, no. 3, p. 528–545.
- Marvinney, R.G., Loiselle, M.C., Hopeck, J.T., Braley, D., and Krueger, J.A., 1994, Arsenic in Maine ground water—An example from Buxton, Maine, *in* Proceedings of the 1994 Focus Conference on Eastern Regional Ground Water Issues, Burlington, Vt., October 3–5: National Ground Water Association, p. 701–715.
- Marvinney, R.G., Loiselle, M.C., Hopeck, J.T., Braley, D., and Krueger, J.A., 1995, Arsenic in Maine groundwater—An example from Buxton, Maine [abs.]: Geological Society of America Abstracts with Programs, v. 27, p. 67.
- National Research Council, 1999, Arsenic in drinking water: Washington, D.C., National Academy Press, 330 p.
- National Research Council, 2001, Arsenic in drinking water—2001 update: Washington, D.C., National Academy Press, 244 p.
- Nielsen, M.G., Lombard, P.J., and Schalk, L.F., 2010, Assessment of arsenic concentrations in domestic well water, by town, in Maine, 2005–09: U.S. Geological Survey Scientific Investigations Report 2010–5199, 68 p. [Also available at <https://doi.org/10.3133/sir20105199>.]
- Peters, S.C., Blum, J.D., Klaue, B., and Karagas, M.R., 1999, Arsenic occurrence in New Hampshire drinking water: Environmental Science & Technology, v. 33, no. 9, p. 1328–1333.
- Ryker, S.J., 2001, Mapping arsenic in ground water: Geotimes, v. 46, no. 11, p. 34–36.
- Shapiro, S.S., and Francia, R.S., 1972, An approximate analysis of variance test for normality: Journal of the American Statistical Association, v. 67, no. 337, p. 215–216.
- Smedley, P.L., and Kinniburgh, D.G., 2002, A review of the source, behavior and distribution of arsenic in natural waters: Applied Geochemistry, v. 17, no. 5, p. 517–568.
- U.S. Environmental Protection Agency, 1994, Method 200.8—Determination of trace elements in waters and wastes by inductively coupled plasma-mass spectrometry (rev. 5.4): U.S. Environmental Protection Agency method, 57 p.
- U.S. Environmental Protection Agency, 2001, National primary drinking water regulations—Arsenic and clarifications to compliance and new source contaminants monitoring [final rule]: Federal Register, v. 66, no. 14, p. 6975–7066.
- U.S. Geological Survey, 2019, USGS water data for the Nation: U.S. Geological Survey National Water Information System database, accessed May 14, 2019, at <https://doi.org/10.5066/F7P55KJN>.
- U.S. Geological Survey, [variously dated], National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1–A10, accessed April 4, 2017, at <https://pubs.water.usgs.gov/twri9A>.
- Welch, A.H., Helsel, D.R., Focazio, M.J., and Watkins, S.A., 1999, Arsenic in ground water supplies of the United States, *in* Chappell, W.R., Abernathy, C.O., and Calderon, R.L., eds., Arsenic exposure and health effects: Burlington, Mass., Elsevier, p. 9–17.
- Welch, A.H., Westjohn, D.B., Helsel, D.R., and Wanty, R.B., 2000, Arsenic in ground water of the United States—Occurrence and geochemistry: Ground Water, v. 38, no. 4, p. 589–604.
- Wilkie, J.A., and Hering, J.G., 1998, Rapid oxidation of geothermal arsenic(III) in streamwaters of the Eastern Sierra Nevada: Environmental Science & Technology, v. 32, no. 5, p. 657–662.
- Yang, Q., Jung, H.B., Culbertson, C.W., Marvinney, R.G., Loiselle, M.C., Locke, D.B., Cheek, H., Thibodeau, H., and Zheng, Y., 2009, Spatial pattern of groundwater arsenic occurrence and association with bedrock geology in greater Augusta, Maine, USA: Environmental Science & Technology, v. 43, no. 8, p. 2714–2719.
- Zheng, Y., and Ayotte, J.D., 2015, At the crossroads—Hazard assessment and reduction of health risks from arsenic in private well waters of the northeastern United States and Atlantic Canada: Science of the Total Environment, v. 505, p. 1237–1247.



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