

Performance of the Goulden Large-Sample Extractor in Multiclass Pesticide Isolation and Preconcentration from Stream Water

Gregory D. Foster,*† William T. Foreman,‡ and Paul M. Gates‡

Department of Chemistry, George Mason University, 4400 University Drive, Fairfax, Virginia 22030, and Methods Research and Development Program, National Water Quality Laboratory, U.S. Geological Survey, 5293 Ward Road, Arvada, Colorado 80002

The reliability of the Goulden large-sample extractor in preconcentrating pesticides from water was evaluated from the recoveries of 35 pesticides amended to filtered stream waters. Recoveries greater than 90% were observed for many of the pesticides in each major chemical class, but recoveries for some of the individual pesticides varied in seemingly unpredictable ways. Corrections cannot yet be factored into liquid-liquid extraction theory to account for matrix effects, which were apparent between the two stream waters tested. The Goulden large-sample extractor appears to be well suited for rapid chemical screening applications, with quantitative analysis requiring special quality control considerations.

INTRODUCTION

The Goulden large-sample extractor (GLSE), a continuous flow liquid-liquid extractor, has been evaluated in the routine analysis of polychlorinated biphenyls and organochlorine pesticides (Merriman and Metcalfe, 1988; Stevens and Neilson, 1989) in large-volume filtered water samples, and as a result of its high extraction efficiencies, the GLSE has been integrated into water quality monitoring programs. A major benefit of the use of the GLSE is rapid, enhanced field-site isolation and preconcentration of "dissolved-phase" hydrophobic organic contaminants from filtered natural waters. The development of field-site extraction techniques is extremely desirable in pesticide analysis to minimize analyte breakdown during storage and to eliminate the requirement of adding hazardous preservatives to the sample. Field isolation of large volumes of water (10-100 L) can also provide substantially lower analyte method detection levels compared to conventional 1-L sample volumes. The design of the GLSE is made to accommodate dichloromethane as the extraction solvent, which is the recommended solvent for the analysis of most trace organic contaminants in water when liquid-liquid extraction is the favored preconcentration technique (Webb, 1978).

Because the GLSE, in its simplest and most convenient mode of operation, performs a single extraction of the sample, its ability to provide reliable data in multiclass pesticide analysis needs to be demonstrated before it can be used interchangeably with the more widely recognized U.S. Environmental Protection Agency's (EPA) master analytical scheme approach (Pellizzari et al., 1985). The objective of this study was to assess the reliability of the GLSE in pesticide analysis by measuring the recoveries of 35 pesticides amended to 10-L filtered stream water samples and comparing measured recoveries using the GLSE to predicted extraction efficiencies derived from liquid-liquid extraction theory. The latter approach has been previously used (Foster and Rogerson, 1990) to optimize extraction performance and to determine factors important in influencing extraction behavior.

EXPERIMENTAL PROCEDURES

Chemicals. The 35 pesticides were obtained in neat form from the U.S. EPA's Pesticides and Industrial Chemicals Repository (Research Triangle Park, NC). Pesticide residue grade solvents (ethyl acetate, hexane, methanol, and dichloromethane) were purchased from Burdick and Jackson (Muskegon, MI) and used without further purification.

Sample Preparation. Water from Clear Creek in Wheat Ridge, CO, and Boulder Creek in Boulder, CO, was collected in June 1989 directly into 40-L stainless steel milk cans. Stream water was subsequently filtered to remove suspended particulate matter through Whatman (Whatman Inc., Clifton, NJ) GF/F glass fiber filters (0.7- μm nominal pore diameter) held in a 14.2 cm diameter Millipore (Millipore Corp., Bedford, MA) stainless-steel filtration apparatus. An aliquot of the filtered sample was diverted into a 250-mL amber glass bottle for pH, conductivity, hardness, alkalinity, and dissolved organic carbon concentration measurements. A 10-L portion of filtered water sample was extracted with the GLSE to provide ambient level analyte concentrations, when present, which were used for spike recovery data correction. Another 40 L of filtered water in a milk can was fortified with 10 mL of a methanol solution containing 35 pesticides. Ten-liter aliquots of this amended water were extracted by using the GLSE to establish extraction efficiencies. Quantities of pesticides were fortified to the sample to achieve an analyte concentration of 15 ng/L. The pesticides were allowed to equilibrate with the sample for at least 1 h before extraction.

Extraction and Analysis. The design, operation, and optimization of the GLSE have been previously described (Neilson et al., 1987; Foster and Rogerson, 1990) and are not discussed further. [Note: A recent design modification is the addition of a dichloromethane recovery system (D. J. H. Anthony, Analytical Chemistry Branch, Environment Canada, personal communication). Dichloromethane is removed from the wastewater and returned to the extractor during operation. Without the solvent recovery system, the GLSE generates a substantial quantity of dichloromethane-saturated water that presents a formidable waste disposal problem, especially when used in the field. Use of the solvent recovery system would not change substantially any of the normal operational parameters of the GLSE presented in this paper.] Each extraction was carried out by using 10 L of either pesticide-amended stream water or nonspiked stream water. Additional blanks consisted of extractions performed with 10 L of Milli-Q (Millipore Corp.) treated tap water. Replicate recovery determinations were made on pesticide-amended waters from both Clear Creek ($n = 3$) and Boulder Creek ($n = 4$). The 10-L filtered water samples were extracted by a single pass through the GLSE at a flow rate of 340 mL/min; the extracted water was not processed further.

After each suite of extractions, the milk can reservoir was washed with two 100-mL portions of dichloromethane to remove

* Address correspondence to this author.

† George Mason University.

‡ U.S. Geological Survey.

Table I. General Water Chemistry for Clear Creek and Boulder Creek Water Samples

	Clear Creek	Boulder Creek
pH	8.3	7.5
sp conductance, $\mu\text{S}/\text{cm}$	236	65
alkalinity, mg/L	56	22
Ca, mg/L	19	6.8
Mg, mg/L	4.5	2.0
dissolved organic C, mg/L	2.5	1.0

pesticides sorbed to the container. Methanol was pumped through the sample metering pump and associated Teflon tubing after each individual extraction to eliminate pesticide memory effects. In addition, Milli-Q water blanks were run between some of the extractions to assess pesticide carryover, and no carryover problems were observed.

Dissolved water in the dichloromethane extracts or rinses was removed by using anhydrous sodium sulfate prior to solvent reduction with rotary-flash and nitrogen evaporation. The extracts were reduced to a final volume of approximately 0.3 mL. Dichloromethane was solvent-exchanged with ethyl acetate during volume reduction. Five perdeuterated polycyclic aromatic hydrocarbons (naphthalene- d_8 ; phenanthrene- d_{10} ; fluoranthene- d_{10} ; chrysene- d_{12} ; and perylene- d_{12}) were added to the extracts at 0.4 ng/ μL as internal injection standards for gas chromatographic/mass spectrometric (GC/MS) quantitative analysis.

The GC/MS system consisted of a Hewlett-Packard (HP) Model 5890A GC and a HP Model 5970A mass selective detector/mass spectrometer controlled by an HP Series 200 computer loaded with HP 59970C Chemstation data system software. The GC was equipped with a J&W Scientific Inc. (Folsom, CA) fused-silica DB-5 capillary column (30 m \times 0.25 mm i.d. with a 0.25- μm bonded-phase film thickness). The GC/MS analysis conditions used were exactly the same as those described by Foster and Rogerson (1990). Quantitation was accomplished by the use of internal injection standard calibrations.

Data Treatment. The pesticide recoveries were corrected for the amount of naturally occurring pesticides in the filtered stream water blanks. Theoretically predicted extraction efficiencies were obtained from a liquid-liquid distribution model [see Foster and Rogerson (1990)]. Incorporated estimates of the dichloromethane/water partition coefficients (K_p) were based on an empirical relation between pesticide water solubilities in the liquid state, corrected when necessary for fugacity differences between liquid and solid substances, and K_p values calculated by using a ratio of dichloromethane and water solubilities of the pesticides reported in *The Agrochemicals Handbook* (Hartely and Kidd, 1983). The resulting regression equation was similar to the relation between octanol/water partition coefficient and water solubility determined by Hansch et al. (1968) for low molecular weight organic compounds.

Differences between predicted extraction efficiencies (%E) and average measured percent recoveries (%R) were assessed as percent deviations (%D) according to the relation

$$\%D = ((\%R - \%E) / \%E) \times 100 \quad (1)$$

Negative values of %D indicate measured recoveries were lower than the theoretically predicted extraction efficiencies, and positive values assigned to %D designate measured recoveries were greater than the predicted values.

RESULTS AND DISCUSSION

The choice of Clear Creek and Boulder Creek water sources was made to test a range of physicochemical characteristics of stream water, most importantly specific conductance, dissolved organic carbon (DOC) concentrations, and clarity (Table I). Total suspended particle concentrations were not measured, but Boulder Creek water was observably very clear while Clear Creek water was highly turbid, indicating a very large difference in suspended particle concentrations between the two water sources. The chemistry of stream waters is highly variable and depends on the geochemical characteristics of the

basin, eliminating the possibility of selecting a representative or standard surface water to evaluate analytical methods. Variations in water chemistry, including the above constituents, as well as, for example, the presence of naturally occurring pesticides in the water sample, may result in a matrix effect on the performance of the GLSE method. Pesticide recoveries reported below for these two water sources highlight the degree of variability that can be expected in the GLSE from field-site preconcentration.

No naturally occurring pesticides were present in the filtered Boulder Creek water sample above the level of quantification. Six naturally occurring pesticides were present at quantifiable concentrations in the Clear Creek water sample: diazinon (27.8 ng/L), malathion (4.5 ng/L), atrazine (4.2 ng/L), prometon (26.6 ng/L), simazine (3.9 ng/L), and bromacil (8.8 ng/L). These amounts were subtracted from the corresponding pesticide concentrations determined for each Clear Creek matrix spike sample prior to calculation of the spike recoveries.

Percent recoveries measured for the individual pesticides from the spiked samples are shown in Table II. Grand mean (overall) percent recoveries for each pesticide group calculated from the data compiled in Table II for Clear Creek and Boulder Creek water, respectively, were as follows: organochlorines, 79.5, 90.3; organophosphates, 100, 108; triazines, 85.2, 89.5; acetanilides, 89.3, 100; thiocarbamates, 63.8, 67.4; and miscellaneous, 70.6, 79.7. The overall recoveries were quite efficient (i.e., $\geq 85\%$) for the organochlorines, organophosphates, triazines, and acetanilides in at least one of the stream waters. Only the thiocarbamates and miscellaneous pesticides had grand mean recoveries $< 80\%$ in both samples. The results clearly demonstrate the utility of continuous flow liquid-liquid extraction of 10 L of water with 0.2 L of dichloromethane for multiclass pesticide analysis.

Substantial overall recovery differences (i.e., $> 5\%$) were evident between Clear Creek water and Boulder Creek water for the organochlorine, acetanilide, and miscellaneous pesticides. (For this comparison the organophosphates were excluded because it was assumed that grand mean percent recoveries $> 100\%$ equate to 100%.) In most cases, pesticide recoveries were lower in Clear Creek water. Emulsion formation reduces extraction efficiency in the GLSE (Foster and Rogerson, 1990), and emulsions were markedly more pronounced during the extraction of Clear Creek water. The higher DOC concentration and possibly greater abundance of nonfiltered (breakthrough) suspended particles in Clear Creek water may have accounted for the greater degree of emulsions. Leenheer et al. (1991) recently reported that Clear Creek can contain large amounts of poly(ethylene glycol) residues. These surfactant materials can represent up to 20% of the total DOC (J. A. Leenheer, personal communication), and if these residues were also present in our Clear Creek sample, they would have contributed to the observed emulsion formations. If pesticide losses were due entirely to the formation of emulsions in the GLSE, nonselective lowering of recoveries would be observed in Clear Creek water, as more of the solvent containing the preconcentrated pesticides would be swept out in the wastewater.

Since a nonselective lowering of recoveries was not evident for Clear Creek samples, it is possible that unextractable DOC and/or nonfilterable particles facilitated the loss of hydrophobic pesticides from the GLSE through sorptive processes. Sorption of hydrophobic pesticides to small particles is a well-known process, but it also has been demonstrated that natural aquatic colloids bind

Table II. Recoveries of Pesticides from Clear Creek and Boulder Creek Water Using the Goulden Large-Sample Extractor

pesticide	% E ^a	Clear Creek			Boulder Creek		
		% R ^b	(% RSD) ^c	rinse ^d	% R	(% RSD)	rinse
organochlorines							
4,4'-DDD	100	79.4	(5.5)	5.5	95.4	(1.3)	2.8
4,4'-DDE	100	51.6	(7.3)	2.6	70.7	(1.8)	3.0
4,4'-DDT	100	83.8	(12.8)	nd ^e	77.7	(14.3)	10.7
dieldrin	100	81.0	(2.3)	nd	99.1	(2.5)	nd
endosulfan I	100	83.6	(2.7)	nd	87.2	(0.3)	nd
endosulfan II	100	82.9	(1.7)	nd	91.3	(1.3)	nd
δ-HCH	100	62.6	(14.9)	nd	65.5	(4.5)	nd
γ-HCH (lindane)	100	88.9	(9.2)	nd	119	(3.2)	nd
organophosphates							
chlorpyrifos	100	106	(7.0)	nd	107	(0.7)	nd
demeton S	99.8	113	(6.2)	nd	102	(15.3)	nd
diazinon	99.9	60.4	(18.4)	4.8	109	(2.5)	nd
dimethoate	65.0	65.2	(6.1)	nd	95.3	(9.8)	nd
ethion	100	113	(4.9)	nd	121	(2.9)	nd
malathion	99.7	105	(15.5)	nd	90.4	(4.8)	nd
methyl parathion	99.7	119	(10.7)	nd	109	(3.9)	nd
parathion	99.9	na ^f			129	(6.4)	nd
triazines							
atrazine	96.3	67.1	(3.8)	nd	79.3	(2.3)	nd
cyanazine	87.6	121	(3.1)	nd	101	(3.3)	nd
metribuzin	70.6	107	(7.4)	nd	102	(7.6)	nd
prometon	100	63.5	(10.1)	nd	85.6	(2.0)	nd
prometryn	97.9	92.5	(3.4)	nd	98.5	(3.9)	nd
propazine	97.7	80.3	(2.5)	nd	89.2	(0.7)	nd
simazine	98.7	69.6	(18.6)	nd	73.1	(4.0)	nd
terbuthylazine	99.0	80.4	(2.6)	nd	89.6	(0.6)	nd
acetanilides							
alachlor	98.8	94.4	(4.4)	nd	106	(1.7)	nd
butachlor	99.9	99.5	(3.6)	nd	93.6	(3.6)	nd
metolachlor	98.8	85.8	(4.1)	nd	104	(2.9)	nd
propachlor	93.5	77.4	(10.6)	nd	96.2	(2.9)	nd
thiocarbamates							
butylate	99.8	65.1	(7.6)	nd	68.8	(12.9)	nd
EPTC	98.7	62.4	(7.3)	nd	66.0	(15.8)	nd
miscellaneous							
bromacil	99.4	54.3	(15.3)	nd	70.1	(8.6)	nd
linuron	98.8	68.2	(9.1)	nd	31.9	(12.6)	nd
cis- + trans-permethrin	100	48.9	(10.3)	16.2	65.5	(10.6)	26.3
propargite	100	75.4	(21.8)	nd	117	(4.0)	nd
trifluralin	100	106	(4.7)	nd	114	(2.9)	nd

^a % E, theoretically predicted extraction efficiency for a 10-L water sample [see Foster and Rogerson (1990)]. ^b % R, mean percent recovery of total spike. ^c % RSD, percent relative standard deviation. ^d Percent of total spike recovered in reservoir rinse. ^e nd, not detected. ^f na, not analyzed.

hydrophobic organic compounds (Wijayarathne and Means, 1984; Caron et al., 1985; Chiou et al., 1986). It would be anticipated that those pesticides with large *n*-octanol/water partition coefficients (K_{ow} , especially $>10^5$) would experience sorption to DOC during the time the pesticides were being mixed with stream water. A fraction of the sorbed pesticide may pass unextracted through the GLSE.

The presence of naturally occurring pesticides may also selectively influence recoveries. Recovery corrections were made for the six naturally occurring pesticides detected in the Clear Creek water sample. However, application of this correction may still be inadequate, and positive or negative biases may result, particularly when the naturally occurring pesticide is present at concentrations similar to or above the spike concentration (15 ng/L), as was observed for the six pesticides detected in the Clear Creek sample.

Actual performance of the analytical method in relation to theoretical extraction efficiencies is illustrated by the %D values calculated for Clear Creek and Boulder Creek sample extractions by using eq 1 (Figure 1). The %D values provide some insight on the magnitude of matrix effects. Although the model does not incorporate analyte loss from extract workup (solvent desiccation, volume reductions, and transfers), it does predict the ideal analytical result and serves as the basis of comparison. Measured recoveries include error associated with losses

incurred during all sample preparation and extraction steps, factors not completely accounted for in the model. However, uncertainties arising from the workup of the 0.2-L dichloromethane extracts apply equally to all samples regardless of the water source. Even under more ideal conditions liquid-liquid extraction theory does not necessarily provide a good prediction of the measured result. It has been demonstrated that recoveries of organics from water using separatory funnel-batch extractions also deviate substantially from theory (Baker et al., 1987), suggesting that these types of comparisons do need to be interpreted with a degree of caution.

The greatest systematic error in the analysis, apart from extraction, arises from the solvent evaporation steps. Propagated uncertainties associated with volume measurements, spike additions, and fluid deliveries are random and make up only 2% of the total error. Solvent evaporation uncertainties were estimated to be approximately 10% of %D from mass balance determinations made after solvent volume reductions were carried out with a subset of the pesticides listed in Table II (Foster and Rogerson, 1990). If acceptable %D values are arbitrarily assigned $\geq -20\%$ to additionally account for matrix-related variables, then the model would be expected to have reasonable predictability.

Three of the eight organochlorine pesticides evaluated

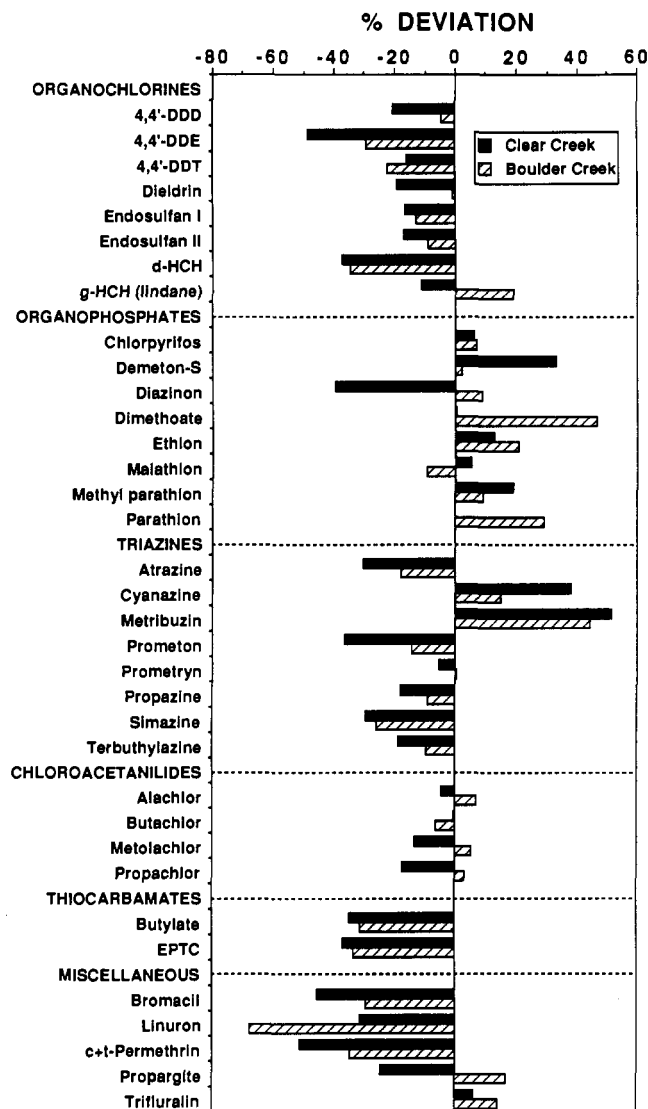


Figure 1. Percent deviation (% D) of average measured pesticide recoveries from predicted extraction efficiencies in GLSE extractions of Clear Creek and Boulder Creek spiked water samples calculated by using eq 1.

had %D values $< -20\%$ (Figure 1): 4,4'-DDD recovery in Clear Creek was also included after consideration was made for the amount of DDD found in the can rinse. However, a matrix effect was evident because seven of the eight pesticides showed lower recoveries in Clear Creek water. Positive %D values were assumed to indicate optimum recoveries. All of the organochlorines had predicted extraction efficiencies (%E) near 100% for 10-L sample volumes (Table II).

All of the organophosphorus pesticides had %D values $> -10\%$ except for diazinon in Clear Creek, which showed a substantially lower than predicted recovery (Figure 1). The organophosphorus pesticide recoveries were much less affected by the type of water than were the organochlorines. If, as before, positive deviations were assumed to indicate optimum recoveries, predictability of the GLSE performance was excellent. The range of predicted extraction efficiencies for the organophosphates was 100% for chlorpyrifos to 65% for dimethoate (Table II).

The triazine herbicides showed a trend similar to the organochlorines in the directional magnitude of recoveries, with recoveries from Boulder Creek water being higher for six of eight triazines (Figure 1). For the recoveries from Boulder Creek water only simazine had a %D value

$< -20\%$, while in Clear Creek water simazine, atrazine, and prometon deviations were lower than that value. The range of predicted recoveries for the triazines was 100% (prometon) to 71% (metribuzin) (Table II).

The acetanilide herbicides as a group showed the most predictable extraction behavior in comparison to all of the other pesticide groups (Figure 1). All four of the acetanilides had %D values $> -20\%$. In contrast, the two thiocarbamate herbicides had much lower than expected recoveries in both stream waters (Figure 1). Even for these two classes of moderate-polarity herbicides, the predicted recoveries in the GLSE ranged between 100% (butachlor) and 93% (propachlor) (Table II).

As expected, the five miscellaneous pesticides had the largest variability as a group (Figure 1). Notable were the low recoveries of *cis*- + *trans*-permethrin (estimated $\log K_p = 5.5$), owing partly to substantial sorption to the reservoir surface (Table II). In all examples except linuron, %D values were less negative in Boulder Creek water. Predicted recoveries for these five pesticides were $> 98\%$.

Although the type of water higher in specific conductance, DOC, suspended particle concentration, and number of quantifiable naturally occurring pesticides (Clear Creek) showed lower pesticide recoveries overall, it was not a predictable phenomenon. Extraction efficiencies previously measured for atrazine, linuron, methyl parathion, metribuzin, and *cis*- + *trans*-permethrin had %D values $> -5\%$ when recoveries were measured in distilled water or tap water (Foster and Rogerson, 1990). The nature of the processes affecting extraction in stream waters is inadequately understood, and additional research in this area is needed.

Due to the diversity of stream waters and the unpredictable nature of matrix effects, it is recommended that any application of field isolation and preconcentration of pesticides in water using the GLSE make use of a suite of surrogate standards introduced into the sample before extraction. The surrogates should be selected in such a manner that one member from each major pesticide class be represented in the analysis. In GC/MS analysis, this can best be accomplished by using isotopically labeled homologues.

In addition to analysis of organochlorine pesticides and polychlorinated biphenyls, the GLSE appears to be well suited to the analysis of other classes of pesticides, including organophosphorus pesticides and triazine and acetanilide herbicides, especially in chemical screening applications. The GLSE may be more susceptible to some matrix effects that the typical separatory funnel extraction routine because of the nature of the extraction process: the filtered sample is extracted rapidly in a flow-through mechanism, preventing an efficient separation of emulsions; only a one-pass extraction is performed on the sample; and the loss of small solvent droplets in the wastewater cannot be monitored efficiently. As a result, additional QC procedures specific to the GLSE may need to be established for routine quantitative analysis of pesticides in stream waters.

ABBREVIATIONS USED

DOC, dissolved organic carbon; GC/MS, gas chromatography/mass spectrometry; GLSE, Goulder large-sample extractor; K_{ow} , *n*-octanol/water partition coefficient; K_p , dichloromethane/water partition coefficient; %D, percent deviation defined by eq 1; %E, percent predicted extraction efficiency; %R, average measured percent recovery.

ACKNOWLEDGMENT

Support for this project was provided by the U.S. Geological Survey's Office of Water Quality, the National Water Quality Assessment Program, and the National Water Quality Laboratory.

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Received for review January 24, 1991. Accepted May 3, 1991. The use of trade names is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.