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A Method for Determining Helium in Water

By Philip W. Holland



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CONTENTS

		Page
Intro Ackno Appar Proce Calcu Discu	cact	1 1 3 3 4 4 4 6 7 7 9 9 9 11 12 13 15 16
	ILLUSTRATIONS	
2. C	Diagram of gas extraction apparatus and mass spectrometer inlet system	3 5 10 12
2. H 3. R 4. P	Mass spectrometer operating settings	6 9 11 12 14

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A METHOD FOR DETERMINING HELIUM IN WATER

by

Philip A. Holland 1

ABSTRACT

The Bureau of Mines has developed a method for determining helium in water for use in sampling and analyzing surface and subsurface waters for geochemical survey work. The accuracy of the analytical method is within ± 10 percent for samples containing above $20\times 10^{-8}~{\rm cm}^3$ He (STP)/cm 3 H $_20$. The minimum detectable helium-in-water concentration is calculated to be $2\times 10^{-8}~{\rm cm}^3$ He (STP)/cm 3 water.

Water samples are collected in 500-cm³ stainless steel cylinders. The dissolved gases are extracted into a cylinder of equal volume containing a known quantity of neon. The resulting gas mixture is subsequently analyzed for helium and neon-21 by mass spectrometry. The volume of helium in the sample is calculated from the mass spectrometric determination of the helium-to-neon-21 ratio, the known quantity of neon used, and the extraction efficiency of helium.

INTRODUCTION

Analysis of subsurface waters and soil gases for helium has been demonstrated as a possible exploration technique for uranium and and thorium ore deposits, oil and natural gas accumulations, and geothermal reservoirs (2-3, 7, 10, 12-14).

The Bureau of Mines has published the results of helium surveys that were conducted in the vicinity of the Bureau-owned Cliffside Field near Amarillo, Tex. (5). The reconnaissance surveys were conducted to determine if helium contents of area ground waters and soil gases could be used to indicate the presence of helium in a subsurface natural gas accumulation. The surveys revealed the presence of anomalously high helium concentrations in some of the area ground waters and soil gases.

¹Chemist, Division of Helium Operations, Bureau of Mines, Amarillo, Tex.

²Underlined numbers in parentheses refer to items in the list of references at the end of this report.

Geochemical methods are being developed by the Bureau as a technique for monitoring the helium accumulation in the Cliffside Field. This report describes the method used to analyze ground waters for their dissolved helium content. Although the method is designed for the range of helium-in-water concentrations encountered in the Cliffside Field (about 4 \times 10⁻⁸ to 260 \times 10⁻⁸ cm³ He (STP)/cm³ H₂0), the analytical parameters can be adjusted to extend the range.

Several mass spectrometer methods have been developed for the analysis of helium in surface and subsurface waters for geochemical reconnaissance. Dyck and Pelchat $(\underline{3})$, Reimer $(\underline{11})$, and Martin and Bergquist $(\underline{7})$ assembled helium leak detector mass spectrometers for field measurements of helium in water for uranium reconnaissance.

Pogorski, Quirt, and Blascheck $(\underline{10})$ developed a mass spectrometer system that is equipped with a proprietary helium separator for analysis of helium in waters, soils, and soil gases. Field samples are hermetically sealed in aluminum containers and transported to the laboratory for analysis.

Clarke and Kugler (2) developed a mass spectrometer method for determining helium and other inert gases dissolved in ground waters. Water samples are sealed in annealed copper tubes with special pinch clamps and an evacuated apparatus is used to extract the dissolved gases from the samples. After removal of the noninert gases with a hot titanium getter and adsorption of the argon, krypton, and xenon on activated charcoal at liquid nitrogen temperature, the helium and neon are measured by mass spectrometry. Neon is used as an index of air contamination in the samples.

Another mass spectrometer method for determining dissolved inert gases in thermal waters has been described by Mazor (8). In this method, samples are collected in 1-cm^3 glass tubes with stopcocks. Mazor's apparatus for purifying the extracted inert gases also uses a hot titanium getter. After adsorption of the argon-krypton-xenon fraction on activated charcoal cold traps, the helium and neon are measured by mass spectrometry. Mazor estimates the precision of his helium-in-water data to be ± 8 percent for concentrations above 500×10^{-8} cm³ He (STP)/cm³ H₂O (8).

Lupton (6) has described a method for determining helium and neon dissolved in seawater. Gases are extracted from samples into a high vacuum line and split into known fractions using a calibrated gas splitter. The fractions are spiked with known amounts of helium-3 and neon-22, and mass spectral measurements of the helium-3:helium-4 and neon-20:neon-22 ratios are used to determine the absolute amounts of helium and neon in the sample.

The method described in this report is a modification of a method developed by West $(\underline{16})$. In West's method, dissolved gases are extracted from the sample into a known quantity of neon. Neon-20 is used as the internal standard. The volume of helium in the sample is calculated from the mass spectrometric determination of the helium-to-neon-20 ratio, the known quantity of neon used, and the extraction efficiency of helium. In the present work,

neon-21 rather than neon-20 is used as the internal standard to avoid interference from doubly charged argon in the mass spectral analysis which reduces the minimum detectable limit to $2\times10^{-8}~{\rm cm}^3$ He (STP)/cm³ H₂0.

ACKNOWLEDGMENTS

The author expresses his sincere thanks to Charles A. Seitz, chemist, and David E. Emerson, supervisory chemist, for their technical assistance in development of the sampling and analytical procedures. Both are with the Section of Research and Analytical Services, Division of Helium Operations, Bureau of Mines, Amarillo, Tex.

APPARATUS DESCRIPTION

A diagram of the gas extraction apparatus and inlet system of the mass spectrometer is shown in figure 1. Cylinders \underline{A} and \underline{B} are standard 500-cm^3 stainless steel cylinders with 1/4-inch female pipe openings. Cylinder \underline{A} , the water sample container, is sealed with a pipe plug at one end and fitted with ball valve \underline{I} at the other end. Cylinder \underline{B} , the extraction container, is connected to cylinder \underline{A} through ball valve \underline{I} and fitted with valve \underline{I} at the other end. A portion of the dissolved gases extracted from the water sample is admitted from cylinder \underline{B} to the mass spectrometer inlet system through valve \underline{I} . The gas is dried by the desiccant-filled tube, \underline{I} , as it enters the mass spectrometer. The $12\text{--} \times 1/4\text{-inch}$ ID tube is filled with anhydrous magnesium

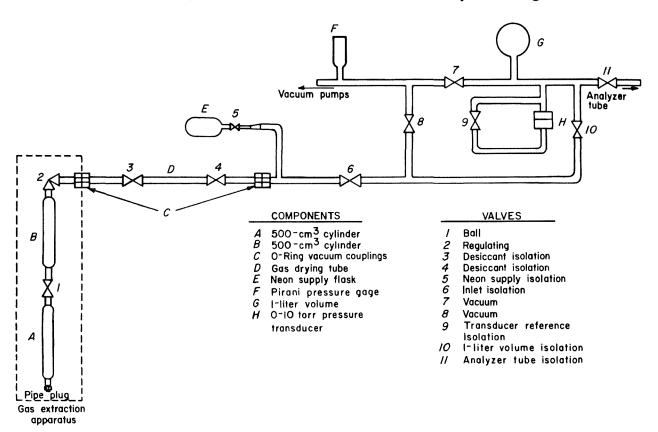


FIGURE 1. - Diagram of gas extraction apparatus and mass spectrometer inlet system.

perchlorate. Valves $\underline{3}$ and $\underline{4}$ are used to isolate the desiccant from the atmosphere when the tube is not in use.

To resist corrosion, all components of the gas extraction apparatus are made of either 304 or 316 stainless steel. However, since ground waters may contain significant quantities of dissolved salts and minerals, the apparatuses used for the extraction of gases from these waters are drained and rinsed with distilled water after each use.

A modified Consolidated Electrodynamics Corp. (CEC) Model $21-103C^3$ mass spectrometer (a single-focusing, 5-inch-radius, 180° instrument) is used to analyze the extracted gases for helium and neon-21. The original 3-liter glass expansion volume of the spectrometer was replaced with a 1-liter stainless steel volume. A 0- to 10-torr differential pressure transducer was installed in the inlet system as shown in figure 1. With the transducer, sample pressures of 1 torr can be measured with an accuracy of ± 0.5 percent.

PROCEDURES

Sample Collection

To obtain a representative sample, the sample container is partially submerged in a bath of freshly collected sample water, and the filling funnel is inserted into the container as shown in figure 2. The container is then filled to overflowing with the sample water and allowed to stand for 5 minutes. This procedure purges the interior of the container and allows the temperature of the container to approach the temperature of the sample water. The container is then emptied, resubmerged in the water bath, and filled to overflowing with freshly collected sample. The sample water is poured slowly into the funnel to minimize air-water turbulence.

The tip of the funnel delivery tube was designed to allow the container to fill in about I minute to minimize air-water contact and mixing. The funnel is kept full of sample water as the container is filled, which also minimizes air-water mixing.

As soon as the sample container overflows, the funnel is removed and the ball valve closed. The samples are stored at room temperature and analyzed within 48 hours of collection.

Introduction of Internal Standard and Gas Extraction

Prior to extraction of the dissolved gases from a sample, 0.25 torr of neon is introduced into cylinder \underline{B} of the gas extraction apparatus. The analytical sequence used in this procedure is as follows: Cylinder \underline{B} is connected to cylinder \underline{A} as shown in figure 1, desiccant-filled tube \underline{D} is removed, and the apparatus is connected to the mass spectrometer inlet through 0-ring vacuum couplings \underline{C} . With valves $\underline{7}$ and $\underline{9}$ through $\underline{11}$ closed, valves $\underline{2}$, $\underline{6}$, and $\underline{8}$

³Reference to specific manufacturers or equipment is made for identification only and does not imply endorsement by the Bureau of Mines.

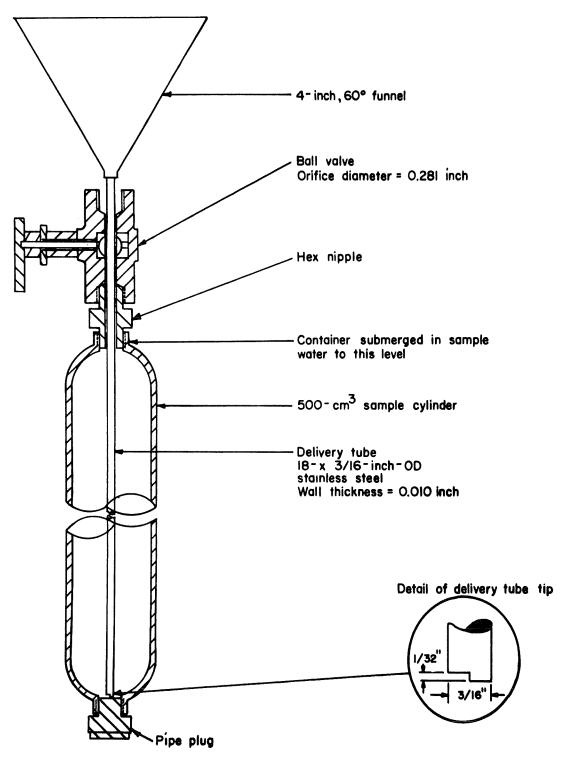


FIGURE 2. - Cutaway of water sample container and filling funnel.

are opened and cylinder \underline{B} is evacuated using the inlet vacuum system of the mass spectrometer. The pressure in cylinder \underline{B} is monitored with Pirani gage \underline{F} . After the pressure in cylinder \underline{B} is below 1 millitorr, valve $\underline{8}$ is closed, valve $\underline{10}$ is opened, and neon from flask \underline{E} is admitted into cylinder \underline{B} and the mass spectrometer inlet system by slowly opening valve $\underline{5}$. The neon pressure is measured using transducer \underline{H} . When the pressure reaches approximately $\underline{0.25}$ torr, valve $\underline{5}$ is closed and the final pressure is measured to the nearest millitorr. The temperature at which the neon pressure measurement is made is also recorded. Valve $\underline{2}$ is then closed and valves $\underline{7}$ and $\underline{8}$ are opened to evacuate volume \underline{G} and the mass spectrometer inlet lines. Valve $\underline{6}$ is then closed and the gas extraction apparatus is disconnected from the mass spectrometer.

To perform the extraction, valve \underline{l} is opened and the apparatus is inverted to allow the water sample to \underline{flow} from cylinder \underline{A} to cylinder \underline{B} . After cylinder \underline{A} is emptied, the apparatus is reinverted, allowing the contents of cylinder \underline{B} to return to cylinder \underline{A} . The apparatus is inverted a total of 10 times to achieve equilibration and is then placed in a vertical position for 5 minutes to allow the residual water in cylinder \underline{B} to drain into cylinder \underline{A} . Valve \underline{l} is then closed and the gaseous mixture in cylinder \underline{B} is analyzed for helium and neon-21 by mass spectrometry.

Analysis of the Extracted Gas

The mass spectrometer operating settings for analysis of helium and neon-21 are listed in table 1. The settings of 1-torr inlet sample pressure and 180 microamperes ionizing current were selected to provide adequate sensitivity.

	Helium scan	Neon-21 scan
Magnet currentmilliamperes	87.0	257.5
Ion accelerating voltagevolts	1,250 to 1,200	1,900 to 1,850
Ionizing currentmicroamperes	180	180
Analyzer slit widthmillimeters	0.76	0.76
Ionizing voltagevolts	70	70
Scan ratevolts per second	(position $3)$ 1.5	(position 3) 1.5
Recorder attentuationmillivolts	(full scale) 50	(full scale) 50
Recorder chart speed		
centimeters per minute	5.0	5.0
Inlet sample pressuretorr	1	1

TABLE 1. - Mass spectrometer operating settings

Before each sample is scanned, a scan of the mass 4 and mass 21 background is made. The presence of residual \underline{n} -heptane and various heavier aliphatics in the mass spectrometer produces \underline{m} etastable ions with a mass-charge ratio of 21.7 (15). These ions are not resolved from neon-21 in the sample analysis. No detectable contribution to mass 21 was observed when airequilibrated distilled water and ground water samples without the neon added were analyzed.

The background is scanned by analyzing a sample of high-purity nitrogen that contains less than the minimum detectable limits of helium and neon-21,

which are 0.5 and 1 ppm (part per million), respectively. A sample inlet pressure of 1 torr is used for the scan. After the background scan is completed, the inlet system and analyzer tube of the mass spectrometer are evacuated for 10 minutes.

A gas sample from cylinder \underline{B} of the extraction apparatus is admitted to the mass spectrometer as follows: The apparatus is connected as shown in figure 1. With valves $\underline{7}$ and $\underline{9}$ through $\underline{11}$ closed, valves $\underline{3}$, $\underline{4}$, $\underline{6}$ and $\underline{8}$ are opened to evacuate to valve $\underline{2}$. The pressure is monitored with Pirani gage \underline{F} . When the pressure is less than 1 millitorr, valve $\underline{8}$ is closed, valve $\underline{10}$ is opened, and sample from cylinder \underline{B} is admitted into volume \underline{G} by slowly opening valve $\underline{2}$. The pressure in volume \underline{G} is monitored with transducer \underline{H} . After the pressure reaches 1 torr, valve $\underline{2}$ is closed and the sample is isolated in volume \underline{G} by closing valve $\underline{10}$.

Sample is admitted to the analyzer of the mass spectrometer by opening valve $\underline{11}$. Scans of masses 4 and 21 are then made using the established scan rate. After the scan is completed, valve $\underline{6}$ is closed, valves $\underline{7}$ through $\underline{10}$ are opened, and the inlet system and analyzer tube are allowed to evacuate for 10 minutes before the next sample is admitted.

Determination of the Helium-to-Neon-21 Sensitivity Ratio

To calculate the volume of helium in the sample, the helium-to-neon-21 sensitivity ratio must be known. The helium and neon-21 sensitivities are determined by analyzing standards of helium and neon in nitrogen. The concentrations of helium and neon-21 in the standards approximate those from a typical sample extraction. The standards are analyzed using the same operating settings and scan rate as the background and sample scans.

The helium-to-neon-21 sensitivity ratio is defined in equation 1 as

$$S_{R} = \frac{S_{He}}{S_{Ne-21}},\tag{1}$$

where S_R = ratio of the helium-to-neon-21 sensitivities,

 S_{He} = sensitivity of helium, divisions per millitorr of helium,

and S_{Ne-21} = sensitivity of neon-21, divisions per millitorr of neon.⁴

CALCULATIONS

Using Henry's law, West $(\underline{16})$ derived the equation describing the extraction efficiency of the permanent gases from water with the described

⁴The neon-21 sensitivity is based on the neon pressure since the neon used to prepare the neon-in-nitrogen standard is the same as that introduced into the gas extraction apparatus. Thus, the isotopic composition of the neon does not have to be known.

extraction apparatus. The extraction efficiency for any slightly soluble gas is calculated lated using equation 2.

$$E_{\times} = \frac{27300}{273 + A_{R}} (K_{\times} T), \qquad (2)$$

where E_x = the extraction efficiency of component x, percent,

A = volume of cylinder \underline{A} of the extraction apparatus, cubic centimeters.

B = volume of cylinder \underline{B} of the extraction apparatus, cubic centimeters.

 K_{\times} = Henry's law constant for component x at the temperature of extraction, cubic centimeters at STP/per cubic centimeters of water - atmosphere,

and T = temperature of extraction, K.

The volume of helium at STP in a water sample is calculated from the mass spectral data, the extraction efficiency of helium, the efficiency of neon recovery, and other experimental parameters shown in equation 3. The efficiency of neon recovery is also based on Henry's law and is calculated in the same manner as the extraction efficiency of helium using equation 2. It is assumed that neon-21 and neon-20 have the same solubility.

where V_{He} = volume of helium in the water sample, cubic centimeters of helium at STP,

 S_R = ratio of the helium-to-neon-21 sensitivities,

 P_{Ne} = pressure of pure neon introduced into cylinder \underline{B} of the extraction apparatus, atmosphere,

B = volume of cylinder \underline{B} of the extraction apparatus, cubic centimeters,

T = temperature of the pure neon at the time of pressure measurement, K.

 E_{Ne} = efficiency of neon recovery from the system, percent,

and E_{He} = extraction efficiency of helium from water with the extraction apparatus, percent.

The concentration of helium in the water sample is then calculated using equation 4.

$$C_{He} = \frac{V_{He}}{A} , \qquad (4)$$

where C_{He} = concentration of helium in the water sample, cubic centimeters of helium at STP per cubic centimeter of water,

 V_{He} = volume of helium in the water sample, cubic centimeters of helium at STP,

and A = volume of cylinder \underline{A} of the extraction apparatus (volume of water sample), cubic centimeters.

DISCUSSION OF RESULTS

Helium-to-Neon-21 Sensitivity Ratio Determination

The mass spectrometric helium-to-neon-21 sensitivity ratio, S_R , was determined by scanning samples of primary standards (9) containing 77.2 ± 0.3 ppm helium in nitrogen and 1.936 ± 0.002 percent neon (approximately 58 ppm neon-21) in nitrogen. Ten samples from each standard were scanned using sample inlet pressures of 1 torr. The results are shown in table 2. The neon-21 sensitivities are based on the neon pressures.

TABLE 2. - Helium-to-neon-21 sensitivity ratio values 1

	Net helium	Net neon-21	Helium sensitiv-	Neon-21 sensitiv-	
Date	peak,	peak,	ity, divisions	ity, divisions	SR
	divisions	divisions	per millitorr	per millitorr	
5-1-80	44.7	59.4	579	3.06	189
5-1-80	44.3	59.5	574	3.07	187
5-1-80	44.5	59.1	576	3.05	189
5-1-80	45.0	59.1	583	3.05	191
5-1-80	44.5	59.6	576	3.07	188
5-7-80	44.4	59.0	575	3.04	189
5-7-80	43.9	58.6	569	3.02	188
5-9-80	44.8	59.4	580	3.06	190
5-9-80	44.6	59.5	578	3.07	188
6-4-80	44.8	58.9	580	3.04	191

Average = 189; standard deviation = 1.

Equilibration Study

Equilibrium is established in the extraction apparatus by inverting the apparatus a sufficient number of times to allow the contents of the water sample container (cylinder \underline{A} in fig. 1) to equilibrate with the contents of the extraction container (cylinder \underline{B}). The number of inversions required to achieve equilibration was determined experimentally.

A helium-in-water standard solution was prepared by bubbling a certified standard of helium in air through distilled water using the apparatus shown in figure 3. A standard containing 110 ± 1 ppm helium in air was bubbled through 3 liters of water at 400 cm³/min for 4 hours. The temperature of the water and atmospheric pressure were 25° C and 0.879 atm, respectively. Using Henry's law, the concentration of helium in the solution was calculated to be 83.1 \pm 1.7 \times 10 $^{-8}$ cm 3 He (STP)/cm 3 H₂O.

After bubbling was completed, the ball valve was closed, the regulating valve opened, and the solution was allowed to stand for 5 minutes. Five 500-cm³ aliquots were then obtained from the apparatus. An aliquot was first drained into a beaker, then transferred into a sample container using the techniques previously described. Each sample container was connected to an extraction cylinder and the five apparatuses were designated as L, M, N, O, and P.

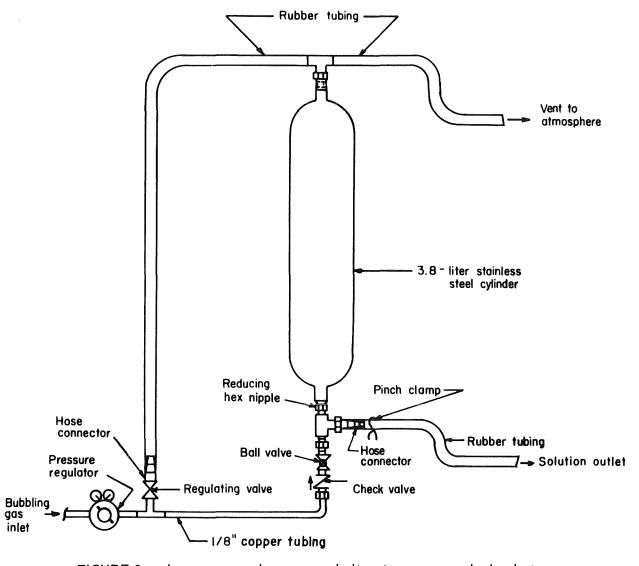


FIGURE 3. - Apparatus used to prepare helium-in-water standard solutions.

After evacuation of the extraction cylinders and addition of the neon, the dissolved gases were extracted by inverting each apparatus a predetermined number of times. Apparatus \underline{L} was inverted 2 times; apparatus \underline{M} , 4 times; apparatus \underline{N} , 6 times; apparatus \underline{O} , 8 times; and apparatus \underline{P} , 10 times. A sample of the extracted gas from each apparatus was scanned for helium and neon-21, and the helium-in-water contents were calculated.

When an apparatus was inverted six or more times, the analyzed helium content equaled the calculated content within the experimental error as shown in table 3. Thus, it is concluded that equilibrium is established in the extraction apparatus after a minimum of six inversions.

Apparatus Number of inversion		Calculated helium- in-water content, cm ³ He (STP)/cm ³ H ₂ O	Analyzed helium-in- water content, cm ³ He (STP)/cm ³ H ₂ O	
L	2	$83.1 \pm 1.7 \times 10^{-8}$	$65 \pm 4 \times 10^{-8}$	
M	4	83.1 $\pm 1.7 \times 10^{-8}$	$77 \pm 4 \times 10^{-8}$	
N	6	83.1 $\pm 1.7 \times 10^{-8}$	$82 \pm 4 \times 10^{-8}$	
0	8	83.1 $\pm 1.7 \times 10^{-8}$	$84 \pm 4 \times 10^{-8}$	
P	10	83.1 $\pm 1.7 \times 10^{-8}$	$82 \pm 4 \times 10^{-8}$	

TABLE 3. - Results of equilibration study

All subsequent sample extractions in this study were performed using 10 inversions. This number of inversions was found to be sufficient for samples containing from about 4×10^{-8} to 245×10^{-8} cm³ He (STP)/cm³ H₂O.

Loss of Helium During Sampling

In the geochemical survey conducted at the Cliffside Field, ground water samples are collected from wells that are pumped by windmills. The water is drawn from an aquifer that ranges from about 80 to 250 feet below the surface (1). The ground water is in contact with the atmosphere an estimated 2 to 3 minutes before a sample is sealed in a sample container. To quantitatively estimate the amount of helium that is lost to the atmosphere during sampling, the following experiment was conducted.

A 2-liter helium-in-water standard containing $170 \pm 8 \times 10^{-8}$ cm³ He (STP)/cm³ H₂O was prepared. The solution was drained from the bubbling apparatus into a 4-liter plastic bucket and samples of the solution were collected in sample containers at the end of 3, 9, and 15 minutes. The samples were extracted and analyzed on the mass spectrometer. The experimentally determined helium concentrations are plotted against the time of collection as shown in figure 4. From the curve it can be seen that no detectable loss of helium occurred during the first 3 minutes. Therefore, it is concluded that no significant loss of helium should occur during the 2- to 3-minute period that samples of ground water are exposed to the atmosphere.

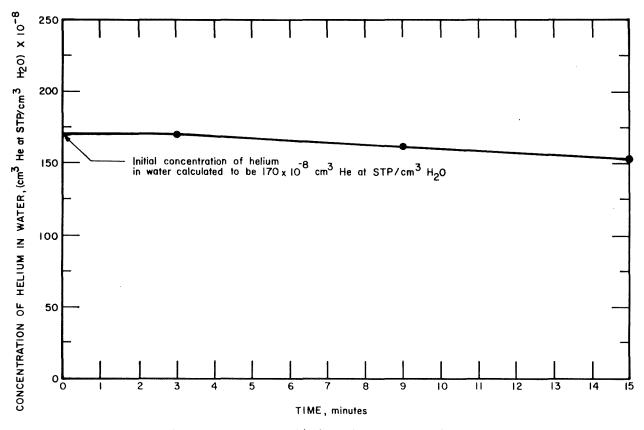


FIGURE 4. - Loss of helium from water with time.

Precision of Ground Water Analysis

The precision of ground water analysis was checked by analyzing water samples obtained from two windmill-pumped water wells in the Cliffside Field. Two sets of five samples each were obtained from each well. Each five-sample set was collected on a different day within a 45-minute period. Each sample was analyzed within 48 hours of collection and the results are given in table 4. The precision of analysis is within ± 5 percent.

TABLE 4. - Precision of helium-in-ground-water analysis, cm³ He (STP)/cm³ H₂O

	We1	1 1	Well 2		
Sample	1st set of	2d set of	lst set of	2d set of	
	samples	samples	samples	samples	
1	127×10^{-8}	128×10^{-8}	7.6×10^{-8}	8.4×10^{-8}	
2	114×10^{-8}		8.0×10^{-8}	7.6×10^{-8}	
3		130×10^{-8}	7.4×10^{-8}	8.2×10^{-8}	
4	124×10^{-8}	120×10^{-8}	7.8×10^{-8}	8.0×10^{-8}	
5	117×10^{-8}	126×10^{-8}	8.1×10^{-8}	8.7×10^{-8}	
Average	120×10^{-8}	126×10^{-8}	7.8×10^{-8}	8.2×10^{-8}	
Standard deviation	5×10^{-8}	4×10^{-8}	0.3×10^{-8}	0.4×10^{-8}	

Accuracy of the Analytical Method

To evaluate the accuracy of the analytical method, four standard solutions containing from about 4×10^{-8} to 245×10^{-8} cm³ He (STP)/cm³ H₂O were prepared and analyzed. This concentration range was selected for study because the helium-in-ground-water concentrations in the Cliffside Field were known to be in approximately the same range ($\underline{5}$). Two of the solutions were prepared by bubbling certified standards of helium in nitrogen through 1-liter quantities of distilled water. A third solution was prepared by bubbling a certified standard of helium in air through 1 liter of distilled water. The standards were bubbled through the 1-liter volumes of water at 400 cm³/min for 4 hours. A fourth solution was prepared by allowing 1 liter of distilled water to equilibrate with ambient air, which contains 5.239 ±0.004 ppm helium ($\underline{4}$). Henry's law was used to calculate the concentration of helium in each solution.

A sample from each standard solution was collected and analyzed within 48 hours of collection. The composition of the gaseous standards used to prepare the solutions, the equilibrium conditions, the calculated and analyzed helium-in-water contents, and accuracy are shown in table 5. Table 5 shows that the accuracy of the analytical method is within ± 10 percent for solutions containing above 20×10^{-8} cm³ He (STP)/cm³ H₂O. The minimum detectable concentration of helium in water is calculated to be 2×10^{-8} cm³ He (STP)/cm³ H₂O.

TABLE 5. - Evaluation of accuracy of analytical method

Composition of gaseous standard used to prepare solution	Equili- bration tempera- ture, ° C	Henry's law constant at equilibration temperature, l cm ³ He (STP)/cm ³ H ₂ O, atm	Equili- bration pres- sure, atm	Calculated helium- in-water content, cm ³ He (STP)/cm ³ H ₂ O	Analyzed helium- in-water content, ² cm ³ He (STP)/cm ³ H ₂ O	Accu- racy, per- cent
5.239 ±0.004 ppm He in air. 3	23	8.60×10^{-3}	0.872	$3.93 \pm 0.08 \times 10^{-8}$	$4.5 \pm 0.9 \times 10^{-8}$	±20
26.6 ±0.3 ppm He in nitrogen.	29	8.53 × 10 ⁻³	.879	19.9 ±0.4 × 10 ⁻⁸	20 ±2 × 10 ⁻⁸	±10
<pre>111 ± ppm He in nitrogen.</pre>	19	8.68 × 10 ⁻³	.879	84.7 ±1.7 × 10 ⁻⁸	85 ±4 × 10 ⁻⁸	±5
329 ±7 ppm He in air	28	8.53 × 10 ⁻³	.872	245 ±7 × 10 ⁻⁸	240 ±12 × 10 ⁻⁸	±5

¹Constants were obtained from Handbook of Chemistry and Physics, 42d ed., Chemical Rubber Publishing Co., 1960, p. 1708.

²The reported accuracies of the analyzed helium-in-water contents are based on the root-mean-square addition of the uncertainties in the experimental parameters used to determine each concentration.

³Concentration of helium in air reported by Gluekauf (4).

CONCLUSIONS

The procedure described in this report provides a precise and reliable method for determining helium in water. Analysis of prepared helium-in-water standards containing from approximately 4 \times 10⁻⁸ to 245 \times 10⁻⁸ cm³ He (STP)/cm³ H₂O showed the accuracy of the analytical method to be within ±10 percent for solutions containing above 20 \times 10⁻⁸ cm³ He (STP)/cm³ H₂O. The minimum detectable concentration is calculated to be 2 \times 10⁻⁸ cm³ He (STP)/cm³ H₂O.

When collecting samples of prepared solutions in the open air, there was no detectable loss of helium from solution when samples were sealed in containers within 3 minutes.

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