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CHROMATOGRAPHIC ANALYSIS OF HELIUM
CONTAINING TRACE IMPURITIES

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By

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PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

CHROMATOGRAPHIC ANALYSIS OF HELIUM CONTAINING TRACE IMPURITIES

INTRODUCTION

A high temperature helium gas loop is to be built in the Advanced Test Reactor near Idaho Falls, Idaho. This loop will be used to study irradiation effects on construction materials at temperatures up to 1300 C. Exposure of metals to gas at these temperatures requires very pure helium. In turn, the helium purity requires highly sensitive analytical instruments. Sensitive gas chromatographic instruments offer a good combination of high sensitivity, mechanical reliability, and moderate initial cost. Probable impurities in the helium are hydrogen, oxygen, nitrogen, methane, carbon monoxide, carbon dioxide, and water vapor.

Gas chromatographs using thermal conductivity detectors have had difficulties in achieving high sensitivity for hydrogen when a helium is used as a carrier because of the similar thermal conductivities of helium and hydrogen. Using argon as a carrier, two chromatographs and a complicated H₂-He separation are required.

It has been reported that a beta ionization detector, using a helium carrier, ^(1, 2) is capable of high sensitivity if very pure helium is used. The purpose of this work was to develop the beta ionization concept to the analysis of trace impurities in helium.

SUMMARY AND CONCLUSIONS

A gas chromatograph has been developed that has a lower detection limit of below 1 ppm for hydrogen, oxygen, nitrogen, methane, carbon monoxide, and carbon dioxide. The instrument can detect neon, argon, krypton, and xenon; argon and oxygen interfere with each other.

This chromatograph uses a tritium source ionization detector. When used with ultrapure helium carrier gas this device functions as an extremely sensitive "helium" detector using the metastable helium

atoms to increase response at low ppm levels. Construction and operating details are discussed and a modification is shown that permits total impurity analysis of helium with sensitivities below 1 ppm.

EQUIPMENT

Detector

The detector chosen for this work is the microion cross section detector described by Lovelock.⁽³⁾ The detector uses 260 mCi of tritium deposited on titanium or zirconium coated stainless steel electrodes. Ultrapure helium is used as a carrier gas. Used in this manner the detector becomes a "helium" detector similar in principle to the commonly used argon detector. Very high sensitivity is possible but response factors fluctuate with helium purity.⁽⁴⁾ The high sensitivity results because of the ease by which helium metastables generated in the radiation field can ionize the species to be detected (all impurities except neon have lower ionization potentials). The concentration of helium metastables at any time is an equilibrium between a constant rate of formation and the rate of destruction by interactions with carrier impurities or eluted species. Sensitivity is lost to the extent that carrier gas impurities reduce the concentration of helium metastables. To obtain satisfactory and reproducible response, the system must be extremely leak-tight, the carrier gas ultrapure, and column bleed-off held constant.

Columns

The two columns were 1/8-in. -OD stainless steel tubing packed with 30/60 mesh 13X molecular sieve (for H₂, O₂, N₂, CH₄, Xe, Kr, Ar, Ne) or 30/60 mesh silica gel (for CO₂). The 13X molecular sieve produced sharper peaks than 5A molecular sieve resulting in increased sensitivity. The molecular sieve column was 6 ft long and the silica gel column 3 ft long. A small piece of glass wool was inserted in each end to hold the packing in place. The ends of the 1/8-in. SS tubing were filled with a piece of No. 14 nichrome wire to reduce the dead

volume. The packed section of tube was placed in a 600Ml Glascol heater for activation. The columns were activated and periodically cleaned by heating to 150 C for 3 or 4 hr while flushing with ultrapure helium. It was found that heating over 200 C increased elution times too much for sharp resolution of trace impurities. It was also noted that traces of oxygen could not be eluted off of 5A molecular sieve at 75 C but seemed to be absorbed. After activation the column had to be saturated with oxygen before a trace oxygen analysis could be run. The columns were operated at room temperature to minimize bleed-off contamination.

Piping Arrangement

This work started with a commercially available chromatograph. It was soon found that leaks in the detector, valves, and piping rendered the instrument unsuitable for trace level analysis, especially for oxygen and nitrogen. A new chromatograph was constructed with the detector, valves, columns and all piping in a helium filled box (Figure 1).

FIGURE 1

Schematic of Chromatograph

The original plug-type valves were replaced by linear O-ring valves. However, even the O-ring valves had detectable air inleakage when operated in air. The detector was modified with O-rings to make the housing more leak-tight and all electrical fittings were brought into the box through a sealed fitting. The valve stems were brought through the box with a high-vacuum O-Ring fitting. Placing all the critical components in the helium atmosphere reduced air leaks to below detectable levels.

The sample loop was a 24-in. piece of 1/8-in. -diameter stainless steel tubing and had a volume of about 2.7 cm^3 .

Electrical Wiring

The electrometer from the original chromatograph was used although the rechargeable batteries caused some inconvenience. Since this type of detector has a fairly high noise level (approximately 10^{-13} amps) any good quality chromatographic electrometer would be suitable. The only special wiring requirement is careful shielding of the leads between the detector and electrometer. These leads are vibration sensitive and the shielded cable was enclosed in a copper tube conduit for rigidity and additional shielding.

The output of the electrometer was run through an automatic attenuator to a 0-1 millivolt potentiometric recorder.

PROCEDURES

Operating Parameters

The beta ionization detector is rather insensitive to temperature and pressure changes so that no special use of thermostats was required for intermittent chromatography. However, for an automatic process stream instrument a constant temperature would be desirable to minimize base line drift.

The carrier gas pressure at the inlet was 50 psi. This made possible a helium flow rate of 150 cc/min through the molecular sieve column and 200 cc/min through the silica gel column. It is desirable to elute the peaks as rapidly as consistent with complete separation.

The longer the impurity components remain on the column, the more random diffusion broadens the peak. This effect has been recently demonstrated by Kargor and Cooke.⁽⁵⁾

The carrier gas used was obtained from Matheson Corp. as "ultrapure" helium. A typical analysis is given in Table I. This gas has a total impurity of 14 ppm including 11 ppm neon.

Choice of Columns

Either the molecular sieve column or the silica gel column is selected by a column switch valve (Figure 1). This method was chosen in preference to arranging the two columns in series. If the silica gel column is placed ahead of the molecular sieve column the transit time of H_2 , O_2 , N_2 , CH_4 , and CO is increased and sensitivity is reduced. Also when the silica gel column is switched directly to the detector the higher bleed-off rate of silica gel causes a severe base line shift for about 15 min. It was preferable to switch the columns and wait until the bleed-off stabilized rather than attempting a two-column separation. These base-line problems were most severe when detecting samples at low ppm levels.

Operating Procedure

The quantity of sample gas was unlimited for this work and was piped from a manifold where either standard gas or unknown gas could be selected. After the sample loop was purged the sample was injected into the instrument by using the gas sample valve. The output of the detector appeared as successive peaks on the recorder. Automatic attenuation was used to keep the peaks on scale. However, some difficulty was encountered with the hydrogen peak that was so sharp that the response time of the automatic attenuator was insufficient. Manual attenuation was then used. Automatic integration was not found to be necessary.

Calibration

The peak heights of the various impurities to be measured were calibrated against known standards. These standards were prepared in the facility shown in Figure 2. A stainless steel gas cylinder was evacuated to below 1μ . Impurity gases were then added as measured with a mercury manometer or McLeod gauge accurate to about $\pm 1\%$. Helium was then added to a total pressure of approximately 1000 psi as measured on a precision pressure gauge. Both the standard gas and the

FIGURE 2

Schematic of Facility for Make-up of Standard Gases

makeup helium were analyzed on the chromatograph and the results corrected for the original impurities in the helium. Typical calibration mixtures are given in Table I. To provide more dilute mixtures, standards were blended with ultrapure helium (Table II) in ratios 10/1, 5/5, 2/10, etc. Dilution of primary standards to overlap the next lower mixture provides a method for intercomparison of mixtures.

TABLE I
COMPOSITION OF GAS STANDARDS

	Standard 1			Standard 2			Standard 3	
	Partial Pressure, Torr	ppm Added	ppm Total	Partial Pressure, Torr	ppm Added	ppm Total	Partial Pressure, Torr	ppm Total
H ₂	1.96	78	78	0.227	4.6	4.6	21.7	868
O ₂	0.43	17	18	0.0242	0.5	1.9	5.58	223
N ₂	1.71	68	74	0.0768	1.4	7.4	22.3	852
CH ₄	0.89	36	36	0.0810	1.6	1.7	----	---
CO	2.63	105	105	0.141	2.8	2.8	18.0	720
CO ₂	1.64	65	65	---	---	---	21.0	840
He	491 psi x 51.91* x 0.981** = 25,000 Torr			991 psi x 51.91 x 0.965 = 49,642 Torr			491 psi x 51.91 x 0.981 = 25,000 Torr	

* Conversion factor psi to Torr

** Corrosion factor nonideal behavior of helium

TABLE II
COMPOSITION OF ULTRAPURE HELIUM
(Vender's Analysis)

CO ₂	0.1 ppm
O ₂	0.1
H ₂	0.2
N ₂	0.3
Ar	0.0
CH ₄	0.0
H ₂ O	2.9
Ne	10.9

RESULTS

Calibrations--Lower Detection Limit

The results of the calibration runs are given in Figure 3. Assuming that a peak twice the noise level was detectable, the lower detection limits were extrapolated as given in Table III. All components are detectable below 1 ppm and oxygen and methane approach a 0.1 ppm detection limit. It should be emphasized that these results are for the chromatograph as a whole, rather than just detector sensitivity, and the results were obtained with actual helium gas samples.

FIGURE 3
Typical Calibration Curve

TABLE III
LOWER DETECTION LIMITS OF IMPURITIES IN HELIUM

(Based on Figure 3; Extrapolation to 2 x Noise Level)

H ₂	0.3 ppm
O ₂	0.1
N ₂	0.2
CH ₄	0.05
CO	0.2
CO ₂	0.4

Typical Chromatograms

Typical chromatograms for various impurities are given in Figures 4 and 5. Sharp separations were obtained even at a few ppm. Typical elution times are given in Table IV.

TABLE IV

TYPICAL ELUTION TIMES

At Room Temperature Off of Column Using 30/60 13X Molecular Sieve
(Length-6 ft, Diameter-1/8 in., Flow-150 ml/min)

Ne	11 sec	Kr	48 sec
H ₂	16	CH ₄	64
O ₂	24	CO	116
Ar	23	Xe	214
N ₂	41		

At Room Temperature Off of Silica Gel Column
(Length-3 ft, Diameter 1/8 in., Flow-150 ml/min)

CO ₂	100 sec
-----------------	---------

Response to Noble Gases

The elution times for neon, argon, krypton, and xenon were determined (Figure 6) and are also given in Table IV. Neon elutes just before hydrogen; the argon peak is coincident with oxygen; krypton elutes just after nitrogen and xenon causes no interference, eluting far after carbon monoxide.

FIGURE-4

Typical Chromatogram of Impurities Added to Helium
6 ft Molecular Sieve (13X) Column, Room Temperature

FIGURE 5

Typical Chromatogram of CO₂ 3 ft Silica Gel Column
at Room Temperature

FIGURE 6
 Typical Chromatogram of Noble Gases in Helium
 6 ft Molecular Sieve (13X) Column, Room Temperature

The neon peak is negative because the neon ionization potential is higher than metastable helium (Figure 6). A peak reversal occurs at high neon concentrations (Figure 6) probably because of offsetting effects of depression of the base line caused by the higher ionization potential of neon and the positive peak from direct ionization of neon.

Linearity of Response

The response of the chromatographs was checked for linearity by the dilution method described. Results (Figure 7) indicate reasonable linearity up to about 20-100 ppm. As concentrations increase further,

FIGURE 7

Linearity of Oxygen and Nitrogen Peak Height

The relative response per ppm decreases and establishes a new linear relationship between 100 and 1000 ppm. This nonlinearity is to be expected with this type of detector, because in the low ppm region the response is the result of the helium metastable reaction plus direct ionization. At higher concentrations the sample impurity itself suppresses the metastables leaving only direct ionization.

Detector Voltage

A 67 1/2 v battery was used for collector voltage in all this work. The effect of varying detector collector plate voltage on response factors was determined as shown in Figure 8. These results indicated response was independent of collector voltage over the range of 40 to 100 v.

FIGURE 8

Relative Detector Response as a Function of
Detector Voltage

Around 200 v both response and noise began to increase rapidly. It appeared that the signal-to-noise ratio for oxygen was especially enhanced. However, for other components such as nitrogen the ratio was less favorable. The difference in response factors versus voltage was thought to offer a means of analyzing oxygen in the presence of argon. This was tried, but initial results were unsuccessful because of extreme variability and unstable response at 200 v.

TOTAL IMPURITY ANALYZER

The changes in background current produced by variations in carrier gas purity suggest application of the detector as a total impurity analyzer of extremely high sensitivity. There is a need for such a device in the ATR Gas Loop to continuously monitor gas purity and give an immediate alarm if purity changes for any reason. Such a total impurity analyzer can be built as shown in Figure 9.

FIGURE 9
Schematic of Total Impurity Analyzer

Tests showed the feasibility of using this detector as a total impurity analyzer. Contaminated helium produced the response in Figure 10. The analyzer is sensitive to less than 1 ppm change in gas purity. An additional advantage is gained over thermal conductivity total impurity analyzers in that hydrogen produces a positive response with the ionization detector, whereas hydrogen cancels some of the response in thermal conductivity detectors.

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FIGURE 10
Response of Total Impurity Analyzer
to Contaminated Helium

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