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Y-1546
Engineering and Equipment

MASTER

A HIGH-PRESSURE GAS MIXING FACILITY (U)

J. L. Heatherly
W. Garland

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the **ATOMIC ENERGY COMMISSION** *under* **U. S. GOVERNMENT Contract W-7405 eng 26**

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

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ABSTRACT

A high-pressure gas mixing facility has been constructed in the Y-12 Plant. This facility can be used to prepare both binary and multicomponent gas mixtures with the minor component ranging in concentration from 5 ppm to any percent. A high-pressure pump allows mixtures to be prepared up to a volume of 220 cubic feet at a pressure of 2000 psi.

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SUMMARY

High-pressure gas mixtures can now be prepared in the Y-12 Plant. The mixtures may be binary or multicomponent with the minor component ranging in concentration from 5 ppm to any percent. Cylinders may be pressurized to 2000 psi, if so desired. Mass spectrometric measurements of these mixtures indicate that an acceptable degree of accuracy was achieved in the preparation.

INTRODUCTION

Since many different gas mixtures are used in the Y-12 Plant for such applications as: calibrations, standards, welding, radiation counters, and environmental atmospheres, it was obvious that a local gas-mixing facility would reduce the overall cost and provide a fast and flexible service.

DESCRIPTION OF THE FACILITY

EQUIPMENT

The mixing facility consists primarily of four interconnecting manifolds, with the necessary valving arrangements, pressure gages, a mass flowmeter, purification traps, a thermocouple vacuum gage, a high-pressure pump, and vacuum pumps. The facility is shown in Figure 1. A schematic diagram is presented in Figure 2. As listed in Table 1, a variety of pressure gages is used to minimize any error introduced by reading the gage. The gages are interchangeable, depending upon the mixture to be prepared. All gages have been accurately calibrated in the Y-12 Plant. A Hastings mass flowmeter is used when adding components below the 200-ppm concentration level. This flowmeter allows 0 - 50 cc per minute of gas to flow into the evacuated cylinder. A separate calibration curve is used for each gas.

Figure 1. HIGH-PRESSURE GAS MIXING FACILITY.

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PROCEDURE

The cylinder to be filled, the manifold, and all interconnecting lines are first evacuated to 10 microns. The minor component is added first. Above the 200-ppm level

Figure 2. SCHEMATIC OF THE GAS MIXING FACILITY. (All Values are High-Pressure Vacuum Values)

and including the percent level, an appropriate gage is selected so that the reading is taken on the high side of the gage. After the gas is added, the system is allowed to equilibrate until the temperature remains constant.

When the minor component is to be less than 200 ppm, the mass flowmeter is used. This device allows 0 - 50 cc of gas per minute to flow into the evacuated cylinder. The length of time the gas is allowed to flow is determined by the desired concentration level and the reading on the meter. The valve to the cylinder is then closed, the excess pressure vented to the atmosphere, and the manifold evacuated to 10 microns.

The major component is added in a method similar to that of a minor component in the percent level. The appropriate gage is selected and the cylinder is pressurized to the desired pressure. When the temperature of the cylinder remains constant, the temperature and pressure of the cylinder and the barometric pressure are recorded. At pressures of 1000 to 1200 psig, the pressure is obtained by using a supply cylinder of higher pressure and bleeding it down to the desired pressure. At pressures from 1200 to 2000 psig, the high-pressure pump is used.

After all components are added, the gases are mixed by thermal convection or by placing the cylinder in a horizontal position and occasionally rolling. The gases are allowed to mix for a minimum of twenty-four hours. A sample is then taken and analyzed on the mass spectrometer to verify the ratio of the mixture.

DISCUSSION AND RESULTS

The precision and accuracy of this method is determined largely by the accuracy of the pressure gages used for the pressure measurements. As shown in Table 1, the least accurate of these gages is 0.25% full scale. By the proper selection of gages, readings in excess of 50% of full scale can be obtained throughout the range from 10 mm Hg to 2000 psig. The accuracy for an individual measurement is, therefore, $< 0.5\%$.

Table 1
PRESSURE GAGES

Pressure Range	Specified Accuracy ⁽¹⁾
<u>Absolute Pressure Indicator⁽²⁾</u>	
0 - 20 mm abs	0.1
0 - 100 mm abs	0.1
0 - 400 mm abs	0.1
400 - 800 mm abs	0.1
<u>Test Gage⁽³⁾</u>	
0 - 15 psi	0.25
0 - 30 psi	0.25
0 - 100 psi	0.25
0 - 300 psi	0.25
0 - 600 psi	0.25
0 - 1000 psi	0.25
0 - 3000 psi	0.25

(1) Percent of full scale.

(2) Manufactured by Wallace and Tiernan.

(3) Manufactured by Ashcroft Laboratory.

Because of the nonideal behavior of gases at high pressures, a correction factor is necessary. In most cases the Van der Waals equation is used. The constants used in this equation are taken from Lange's Handbook.^(a) While the equation is admittedly an approximation, the error is negligible since the correction is only around 10 percent for most of the gases at less than 2000 psi. Whenever the component is a minor constituent, this correction is insignificant. The correction factor is calculated by dividing the molar volume of an ideal gas by the molar volume obtained from the Van der Waals equation. Examples of the correction factor for argon, helium, and nitrogen are presented in Figures 3, 4, and 5. Similar correction curves are prepared for each gas used.

(a) Lange, N. A., Editor; Handbook of Chemistry, 10th Edition, pp 1510 - 1513; McGraw-Hill Book Company, Inc (1961)

Figure 3. ARGON CORRECTION CURVE.

Figure 4. HELIUM CORRECTION CURVE.

The purity of the gases obtained in the Y-12 Plant is such that it is seldom necessary to purify them further. When a mixture containing a component in the low concentration range is desired, an analysis is first made of the balance gas prior to preparing the mixture. If the component is present in the balance gas, the amount necessary to bring the component to the desired level is then added.

A comparison of the mixtures requested and the mass spectrometer analysis is reported in Table 2.

Figure 5. NITROGEN CORRECTION CURVE.

Table 2
MASS SPECTROMETER ANALYSES OF GAS MIXTURES

Gas	Mixture Requested	Analysis
He	97.0%	97.1%
H ₂	3.0%	2.9%
He	98.0%	98.1%
H ₂	2.0%	1.9%
He	95.0%	95.3%
H ₂	5.0%	4.7%
He	85.0%	85.2%
H ₂	15.0%	14.8%
CH ₄	64.4%	64.6%
CO ₂	32.5%	31.5%
N ₂	3.1%	3.9%
O ₂	75.0 ppm	75.0 ppm
N ₂	Balance	Balance
N ₂	95.0%	95.0%
H ₂	5.0%	5.2%
N ₂	95.0%	95.0%
H ₂	5.0%	4.4%
N ₂	95.0%	95.0%
H ₂	5.0	4.3%
O ₂	75.0 ppm	75.0 ppm
N ₂	Balance	Balance
CH ₄	600 - 1000 ppm	932 ppm
He	Balance	Balance
H ₂	5.0%	5.2%
Ar	95.0%	94.8%
H ₂	5.0%	4.3%
Ar	95.0%	94.7%
N ₂	15 ppm	3 Cylinders { 18 ppm
Ar	Balance	
N ₂	85 ppm	90 ppm
Ar	Balance	Balance
He	97.0%	97.1%
H ₂	3.0%	2.9%
He	97.0%	97.0%
H ₂	3.0%	3.0%
He	97.0%	97.1%
H ₂	3.0%	2.9%
O ₂	75 ppm	75 ppm
N ₂	Balance	Balance
O ₂	75 ppm	85 ppm
N ₂	Balance	Balance

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