LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

Price $0.75 . Available from the Office of Technical Services, Department of Commerce, Washington 25, D. C.
ORGANIC REACTOR WASTE GAS ANALYZER

BY

H. M. GILROY
R. J. EDWARDS

ATOMICS INTERNATIONAL
A DIVISION OF NORTH AMERICAN AVIATION, INC.
P.O. BOX 309 CANOGA PARK, CALIFORNIA

CONTRACT: AT(11-1)-GEN-8
ISSUED: OCTOBER 15, 1958
DISTRIBUTION

This report has been distributed according to the category "Radioactive Waste" as given in "Standard Distribution Lists of Unclassified Scientific and Technical Reports" TID-4500 (13th Ed., Rev.), February 15, 1958. A total of 595 copies was printed.
TABLE OF CONTENTS

Abstract .................................................. 4
I. Introduction ............................................. 5
II. Selection of Instruments ............................... 6
III. Major Components ...................................... 7
IV. Accessories ............................................. 11
V. Safeguards .............................................. 12
VI. Operational Procedure ................................. 14
VII. Test Operation .......................................... 17
VIII. Supplementary Data ................................... 18
IX. Summary ................................................ 19

LIST OF FIGURES

1. Photograph of Console (10-3-57-7500-5505B) ............ 9
2. Simplified Flow Diagram .................................. 10
3. Pressure Relief and High Level Radiation
   Alarm System ............................................ 13
4. Total Flow Diagram ...................................... 15
ABSTRACT

The design of a waste-gas treatment system for organic moderated reactors requires a knowledge of reactor waste-gas composition, generation rate, and radioactivity. To obtain data on these variables a continuous stream analyzer was constructed to analyze the waste gas from the Organic Moderated Reactor Experiment (OMRE). The major components of this analyzer were: a vapor-phase chromatograph for gas composition, a vibrating reed ionization chamber for radioactivity measurements, a wet test meter for generation rate, and cold traps for condensable materials.
I. INTRODUCTION

During the operation of an Organic Moderated Reactor (OMR), waste gases are formed as a result of pyrolytic and radiolytic decomposition of the organic coolant. These gases must be removed to maintain constant reactor pressure and a system for treatment of these gases is required, where the possibility of radioactive or fission gases exists. Considering the removal of waste gases, provisions must be made for organic coolant vapors; water vapors, if a steam generator is present; blanket gas, if used; decomposition gases; and radioactive or fission gases.

Preliminary to the design of a waste gas treatment system information must be obtained on the radiolytic gas composition, generation rate, the specific radioactivity of the waste gas and the solubility of the gases in the coolant at operating conditions. These data can be obtained by analysis of the waste gases from the OMRE now in operation at National Reactor Testing Station. Information obtained during operation of the OMRE at various temperatures, power levels, and percent high-boiler residue will supply valuable data on waste gas from both the reactor head-space purge and purification system. This information can then be applied to the design of a waste-gas treatment system for the OMR.
II. SELECTION OF INSTRUMENTS

Analysis of gases from the OMRE waste gas and purification systems require analytical instruments capable of continuous sampling, with broad coverage of the components expected. Information obtained from small-scale capsule experiments and from in-pile loop irradiation of diphenyl and terphenyl showed that the major components to be expected are hydrogen, lower aliphatic hydrocarbons through butane with traces of hexane, benzene, and toluene. The OMRE reactor head space is continuously purged with nitrogen gas to dilute the waste gas formed resulting in an off-gas of high nitrogen content. This dilution of the waste gas requires analytical instruments capable of high accuracy. The instrument considered to meet these requirements best, for composition determination, is a vapor phase chromatograph which is capable of analyzing both liquid and gaseous samples.

The gas released from the OMRE must be metered to determine total off-gas rate and waste-gas generation rate. For continuous operation at the flow rates expected, a wet test meter was selected which integrates the unsteady-state waste gas flow.

Measurement of total waste gas radioactivity on a continuous basis is necessary for comparison of waste gas activity with changes in composition and generation rate. For this reason an in-line ionization chamber, capable of operation at elevated temperatures, with associated vibrating reed electrometer was selected.
III. MAJOR COMPONENTS

These instruments, together with auxiliary components and accessories, were assembled in the form of a portable console, as shown in Figure 1. As shown in the simplified flow diagram, Figure 2, the system consists of the following major components:

1) Gas Chromatograph, Beckman, Model GC-2.

   The chromatograph was modified to contain two columns in the heating chamber, and to have appropriate valves for operation of one column at a time. A hexadecane column for analysis of aliphatic hydrocarbons from methane through butane, and a molecular sieve column for analysis of nitrogen, hydrogen, and methane were selected for analysis of expected waste gas from the reactor head-space purge system. A silicone column was selected for analysis of aromatic compounds from benzene to propyl benzene expected in the purification system waste gas.

   A strip chart recorder was selected to record the signal from the chromatograph detector.

2) Talbert-Cary Ionization Chamber, 250 Ml capacity.

   This chamber was installed in-line to monitor the total waste gas flow after passage through one cold trap.

3) Vibrating Reed Electrometer, Cary Model 31.

   The electrometer was connected to the ion chamber by an electrical cable. A strip chart recorder was selected to record electrometer readings.

4) Wet Test Meter, Precision, 50 scfh.

   The wet test meter was installed at the analyzer-console discharge to integrate the waste gas flow rate. Complete removal of coolant by two cold traps upstream from the wet test meter eliminates the possibility of contamination of the water used in the meter.
5) Cold Traps, Pyrex, 1 Liter, Ice-Cooled.

Cold trap A is required to remove coolant vapors which could cause plugging of lines, interfere with chromatographic analysis and radioactivity determination, and contaminate the water used in the wet test meter.

Cold trap B is used to insure complete removal of coolant vapors and to indicate the efficiency of cold trap A.
Figure 1. Photograph of Console
Figure 2. Simplified Flow Diagram
IV. ACCESSORIES

Accessories in the waste gas analyzer console include:

1) Flowmeters for high and low flow rates of waste gas and for chromatograph carrier gas.

2) Manometers for measurement of waste gas pressure and chromatograph column pressure.

3) A demister at the inlet of the analyzer console system to remove entrained liquid from the waste gas.

4) A sediment strainer at the inlet of the analyzer console to prevent entry of foreign matter which could plug the system.

5) Valves to control flow and enable by-passing of the various components

6) Powerstat transformers, heating wires, heating mantles, temperature recorder, and thermocouples for controlled heating of all lines, valves and other components to prevent plugging with solidified coolant.

7) Vacuum pumps for operation at reduced pressure, where radiation level of analyzed gas might prohibit any outward leakage. When vacuum operation is required, one pump is used to maintain the sample gas system at reduced pressure while the other pump brings the chromatograph column discharge to below atmospheric pressure.

8) Two voltage regulators, one for the chromatograph and the other for the remainder of the console instruments, were also used.

9) All joints were silver soldered and all lines, valves, and components up to the chromatograph were insulated to maintain stable temperature in the entire system.
V. SAFEGUARDS

For protection of operating personnel and equipment the following safeguards are included as part of the analyzer console (Figure 3):

1) High level radiation alarm and shut off system.

This system consists of a Geiger-Muller detector tube mounted on the outside of the waste gas line upstream from the analyzer and a three-way solenoid valve at the inlet of the console. If the waste gas should become radioactive, above a point set on the detector, the three-way solenoid valve will close the sample line to the console, divert the waste gas to the stack through the pressure relief system, and purge the analyzer console system with helium. At reduced pressure, the detector circuit would also shut off the system vacuum pump.

2) Pressure Relief System.

The pressure relief system consists of a pressure sensing switch installed in the waste gas line upstream from the analyzer console, and a normally closed solenoid valve in a by-pass line to the stack. A pressure increase beyond the limit of the pressure sensing switch opens the solenoid valve and vents the waste gas to the stack.

3) Flame Arrestor.

Included in the analyzer console system is a copper mesh flame arrestor.
Figure 3. Pressure Relief and High Level Radiation Alarm System
VI. OPERATIONAL PROCEDURE

As shown in Figure 4, the waste gas analyzer console is designed to function as follows: the waste gas is monitored by a Geiger-Muller tube and, if the activity level of the gas is below a preset level, the gas is admitted to the analyzer system. The pressure of the gas must also be below a preset limit or the gas cannot enter the system. A sediment strainer removes any particulate matter which could cause plugging in any part of the system and a demister removes any entrained liquid particles. The gas goes through an ice-cooled trap to remove condensable materials, is then directed through either the high or low range flowmeter, depending on the flow rate of the waste gas. The metered gas then goes through a flame arrestor, steel wool filter column, and into the in-line ionization chamber. The gas can then be analyzed by the chromatograph or sent through another ice-cooled trap to the wet-test meter, as is done during instrument warm-up periods or during chromatograph column changes. When chromatographic analysis is being performed, the gas flows through a sampling valve loop where a constant volume is made available for analysis, then through another flowmeter, and the wet test meter; it is finally returned to the OMRE stack for disposal. When vacuum operation is required, the system vacuum pump is used after the second cold trap and before discharge to the stack.
Figure 4. Total Flow Diagram
VII. TEST OPERATION

Test operation of the analyzer required a source of coolant-saturated gases simulating conditions expected at the OMRE. A synthetic gas mixture, similar to that expected during operation, was bubbled through a tank of terphenyl maintained at 500°F. The resulting vapors and gases were passed into the analyzer console and conditions of temperature, pressure, and flow were selected to give optimum performance from the traps, separators, and chromatograph. Chromatographic analysis of the synthetic gas mixture with and without organic coolant showed no interference from organic vapors.

The vibrating reed ionization chamber, combination was calibrated with Krypton-85 sealed in the ion chamber. Normal operation of all components was observed. The direct reading from the ionization chamber will be used as a relative measure of waste gas activity and will be related to specific activity through gamma spectra and beta absorption curves.

The entire analyzer console system was vacuum tested satisfactorily.
VIII. SUPPLEMENTARY DATA

To supplement the data obtained from the vapor phase chromatograph and the vibrating reed electrometer, samples will be taken at random intervals for mass spectrometer analysis and radio assay. Information from mass spectrometer analyses will be used to confirm the analyses obtained with the chromatograph and will aid in calibration of the chromatograph for trace components. Vibrating-reed electrometer data will be interpreted by correlation of $\gamma$ spectrum and $\beta$ absorption curves with continuous records of waste gas activity and in-situ decay curves.
IX. SUMMARY

A waste-gas analyzer console has been designed, constructed, and test operated and will be installed at the OMRE to analyze waste gases for composition and radioactivity and determine waste gas generation rate. Waste gases will be passed through the analyzer console and returned to the OMRE stack for disposal. Determination of generation rate and radioactivity will be continuous and frequency of sampling for composition analysis will be dependent on the length of time required for a complete analysis.