IN-1078
May 1967

MASTER

RETENTION OF IODINE ON SELECTED PARTICULATE FILTERS
AND A POROUS SILVER MEMBRANE BEING CONSIDERED
FOR THE LOFT MAYPACK

J. H. Keller, F. A. Duce, F. O. Cartan

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U. S. ATOMIC ENERGY COMMISSION

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Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

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IN-1078
Issued: May 1967
Reactor Technology
TID-4500

CFSTI PRICES

H.C. \$ 3.00; MN. 65

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ABSTRACT

Data are presented for the retention of iodine by three particulate filters and a porous silver membrane at room temperature. The measured iodine retentions are a function of the weight of iodine incident on the filters. The particulate filters retain a larger percentage of the incident iodine at low iodine exposures. The porous silver membrane, though a good iodine trap, loses its high retention efficiency at low iodine exposures.

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INTRODUCTION

As support for the Loss-of-Fluid-Test (LOFT) program, the performance of the proposed Gas-Particulate sampler is being evaluated. A series of 216 samplers will be installed to monitor fission products released into the atmosphere of the LOFT containment building as a function of time and position.⁽¹⁾ The sampler consists of a hollow aluminum capsule which contains a filter cartridge and a gas void space. The atmosphere will be drawn sequentially through the filter cartridge and the void space by an applied vacuum at a preset time after reactor core meltdown.

The filter cartridge is a modified "Maypack"⁽²⁾, a series of filters arranged to separate the various chemical forms of fission-produced iodine. The proposed cartridge consists of one or more high-efficiency particulate filters, several silver membranes, two chemically impregnated charcoal beds, and a final particulate filter. The filters are assumed to remove the particulate fission products, including iodine sorbed on particles. The silver membrane and the chemically impregnated charcoal are assumed to remove volatile, inorganic iodine compounds and volatile, organic iodine compounds, respectively. After sampling, the "Maypack" is disassembled and the proportions of the various chemical forms of iodine are determined by gamma counting the components.

The proportions obtained from Maypack data frequently do not agree with proportions obtained by other methods such as gas chromatography⁽³⁾. Because the interpretation of Maypack data involves basic assumptions concerning the retention of iodine species on the Maypack components, the validity of these basic assumptions must be established under LOFT conditions.

This report describes tests using molecular iodine on selected particulate filters and a porous silver membrane under conditions that simulate the final stages of the LOFT experiment. These conditions are approximately 100% relative humidity and ambient temperature. Emphasis has been placed on the relationship between the amount of iodine retained and the amount of iodine incident on the components.

APPARATUS AND PROCEDURE

The apparatus (Figure 1) consists of a source of water-saturated air carrying iodine vapor, an air thermostat, flow manifold, the sample test cartridges, and charcoal backup traps.

The air stream is produced by saturating a controlled flow of air with water, then mixing it with iodine carried by a slow helium flow. The air is saturated by bubbling it through a frit immersed in a temperature-controlled water bath. The flow rate is 0.1 cfm., which is the flow rate proposed for the LOFT sampler. The iodine source is a U-tube containing molecular iodine tagged with iodine-131. Details of its preparation are given in the appendix. The weight of iodine loaded into the air stream is controlled by adjusting the helium flow and the temperature of the

U-tube. The helium flow is varied from 1 to 100 cc/min and the U-tube is kept at one of three temperatures, 21°C, 0°C, or -50°C, by suitable baths.

The manifold in the air thermostat is so arranged that the air can be passed through or around the saturator and through or around either of the two test cartridges. This arrangement provides for a preliminary adjustment of the flow and the degree of saturation without exposing the test cartridges.

The test cartridge (Figure 2) consists of a glass entrance section, a two-piece Teflon section which holds the test filters or adsorbers, and a lower glass section which holds the charcoal backup traps. The seals between the sections are O-rings. The test filters or adsorbers are placed into the lower part of the Teflon section and are separated by 0.020-in. Teflon spacers. The exposed diameter of the filters or adsorbers is 7/8-in. (2.2-cm). The charcoal backup traps are two 4.5-g portions of chemically impregnated charcoal separated by filter paper disks.

To make a measurement, the air flow through the water saturator and the test cartridges is adjusted to 0.1 cfm. The helium stream carrying the iodine is turned into the water-saturated air stream. Each of the test cartridges is individually exposed to the mixed stream for a fixed, usually 20-min, period. At the completion of the measurement, the iodine source is isolated in a helium atmosphere and saved for subsequent tests.

The test cartridges are disassembled and the filters, spacers, and the charcoal beds are bagged and the I-131 activities are measured with a calibrated gamma counter. The weight of iodine retained on each component is calculated from the known specific activity of the iodine source. The retention value is calculated as the weight of iodine on a component divided by the total weight of iodine incident on the component.

RESULTS AND DISCUSSION

Figure 3 summarizes the iodine retention of three commercial particulate filters. Each data point represents the retention of the first of a series of three filters. In general, the retentions of the second and third filters are the same as the first filter, which indicates the absence of particulate activity from the iodine source. Information on the composition and size of the filters is given in the appendix.

The particulate filter presently proposed for the LOFT filter cartridge is the Flanders F-700. This glass fiber filter was chosen on the basis of strength, water resistance, and some preliminary iodine retention measurements. Unfortunately, these preliminary retention measurements were made with large amounts of iodine. With small amounts of incident iodine, Figure 3 shows that a F-700 filter removes a large fraction of the iodine vapor.

The particulate filter assembly in the proposed LOFT filter cartridge consists of three F-700 filters. As an example, a sample of the LOFT containment atmosphere containing 0.5 μg of iodine would lose approximately 50% of the iodine when passed through the filter assembly. This large loss is inconsistent with the function of the particulate filter assembly. This function is the retention of particulate matter activity only.

This undesired iodine retention of particulate filters probably is surface controlled. A comparison (Figure 3) of the iodine retentions of the F-700 filter and the F-600 filter tends to confirm this. The F-600 and F-700 filters are identical except that the F-700 contains a binder and a waterproofing agent. The lower iodine retention of the F-700 indicates that the addition of the binder or waterproofing agent reduces the number of sites available for sorption. The high iodine retentions of these glass fiber filters at low iodine exposures probably are typical of other glass fiber filters.

The low iodine retention of the F-500 filter compared with the F-700 and F-600 filters, is further evidence of the importance of the type of surface. The F-500 filter is made from Fiberfrax (magnesium aluminum silicate) and asbestos fibers.

Figure 4 summarizes the iodine retention of a commercial porous silver membrane. The membrane is described in the Appendix. Each data point represents the retention on the first of a series of three silver membranes. These data indicate that a single silver membrane is not a satisfactory iodine trap at low amounts of incident iodine. The retentions obtained from the second and third membranes are not consistent with the values from the first membrane. This behavior may be caused by the conversion of iodine to other forms on the surface of the membranes. Also, at low iodine exposures, the purity of the iodine reaching the test cartridges is questionable. During the passage of the iodine vapor from the source to the test cartridge or on the filter itself, there may be a partial conversion to other chemical forms, such as hydrogen iodide. The different sorption characteristics of these forms could explain the observed scatter of the data points.

The humidity of the air stream affects the iodine retention. Figure 5 shows retention data for the F-500 particulate filter using both dry and water-saturated air streams at 21°C. The mechanism responsible for the increased retention at high humidity is not yet established.

Additional work is planned as follows:

1. The test air streams will be analyzed to determine whether chemical forms other than molecular iodine are present at low iodine concentrations.
2. The measurements will be repeated at 90° to 100°C.
3. The retentions of other chemical forms of iodine on the LOFT filter cartridge components will be measured at room temperature and near 100°C.

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APPENDIX

Description of Filters, Adsorbers, and Iodine Preparations

A. Adsorption Materials

Silver Membrane	Selas Flotronics silver membrane discs, 1.062-in. diam, 0.002-in. thick, 5- μ pore size. Exposed diam was 7/8-in.
F-600 particulate filter	Flanders Filter Inc. all-glass high-efficiency filters were supplied in 12- x 12-in. sheets, 0.016-in. thick. Discs, 1.0625-in. diam, were punched from the sheets. Exposed diam was 7/8-in.
F-700 particulate filter	Flanders Filter Inc. glass-fiber, high-efficiency filters with a latex binder and a silicone waterproofing agent were supplied in 12- x 12-in. sheets, 0.016-in. thick. Discs, 1.0625-in. diam, were punched from the sheets. Exposed diam was 7/8-in.
F-500 particulate filter	Flanders Filter Inc. Fiberfrax-asbestos high-temperature filters were supplied in 12- x 12-in. sheets, 0.040-in. thick. Disc, 1.0625-in. diam, were punched from the sheets. Exposed diam was 7/8-in.

B. Reagents

Molecular iodine tagged with iodine 131.	Carrier-free I-131 (Isotopes Division of ORNL) was evaporated to dryness under a stream of helium. Weighed amounts of reagent grade potassium iodide were added to the dry spike. The dry solids were covered with powdered reagent grade potassium dichromate. The solids were heated under a slow stream of helium until fusion and oxidation occurred. The tagged iodine was trapped in a U-tube in an ice bath.
Impregnated charcoal	Pittsburgh Activated Carbon coconut charcoal, PCB 12 x 30 mesh, was impregnated with 2% by weight of reagent grade potassium iodide. The potassium iodide was dissolved in a

volume of water equal to one-half the volume of the dry charcoal. The solution was added to the charcoal with vigorous stirring. The wet solid was air dried and finally dried in a vacuum oven at 120°C.

Figure 1 Schematic Diagram of Test Apparatus

Figure 2 Pictorial Diagram of Test Cartridge

Figure 3 Iodine Retention on Particulate Filters at 21°C and 99% Relative Humidity

Figure 4 Iodine Retention on a Silver Membrane
at 21°C and 99% Relative Humidity

Figure 5 Effect of Humidity on Iodine Retention
of F 500 Filter at 21°C