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Progress to July I, 1961 ORGANIC MODERATOR-COOLANT IN-PILE IRRADIATION LOOP for the M.I.T. NUCLEAR REACTOR

by E.A.Mason, W.N.Bley, D.T.Morgan DEPARTMENT OF NUCLEAR ENGINEERING MASSACHUSETTS INSTITUTE OF TECHNOLOGY CAMBRIDGE 39, MASSACHUSETTS

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ORGANIC MODERATOR-COOLANT IN-PILE IRRADIATION

LOOP FOR THE MIT NUCLEAR REACTOR

by

E. A. Mason

W. N. Bley

D. T. Morgan

Department of Nuclear Engineering Massachusetts Institute of Technology Cambridge 39, Massachusetts

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1.0 Introduction

An in-pile irradiation loop for studying the irradiation behavior of a wide variety of organic fluids with desirable chemical, physical, and nuclear properties for use in organic cooled nuclear reactors has been designed and constructed for use in the MITR, a heavy water moderated and cooled research reactor. The primary purposes of the loop are for the study of the nature and rate of irradiation degradation of the various organic materials tested and of the effect of the irradiation degradation products on the rate of heat transfer and on the other characteristics pertinent to use of organics as reactor moderator-coolants. The information obtained will be useful in the economic and technical evaluation of various organic materials for use in large scale nuclear power reactors as well as in understanding the degradation processes and in the selection of improved coolants. A description of the loop and the various measurements to be made is presented in this report with experimental data included where available and pertinent.

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2.0 Present Status

This report covers the period from Oct. 1, 1959 to July 1, 1961. The last report⁽¹⁾ issued on this project was dated Jan. 1, 1960 and was submitted to the Atomics International Division of North American Aviation under Contract No. N9-S-514.

Insertion of the in-pile loop was originally scheduled for the spring of 1960. However, delivery of the in-pile assembly was delayed until December, 1960 due to material procurement and fabrication difficulties, and three separate failures of the canned motor pumps, two before receipt of the in-pile section, and one after delivery of the in-pile section, have delayed the insertion of the in-pile loop into the M.I.T.R. approximately one year.

In November 1960 the analytical support program for the in-pile irradiation studies was expanded at M.I.T. to include responsibility for all the physical and chemical measurements on the irradiated coolants. Prior to November 1960, Atomics International was to provide most of these measurements.

Out-of-pile testing of all loop components has now been satisfactorily completed and in-pile operation is expected by the middle of July. Plans for the experimental program are now complete and are presented in this report. Plans for the physical and chemical measurements on the irradiated organic to be made at MIT and the equipment and techniques to be used are complete or in a state of advanced development; arrangements have been completed in all cases for those physical and chemical measurements to be made by outside vendors. The dosimetry phase of the program is now complete and the results have been used to determine the fast neutron and gamma-dose rate in Santowax OMP. Heat transfer measurements have been made during out-of-pile testing of the loop and are in good agreement with the work of previous investigators. The experimental program is presented in more detail in a later section of the report.

3.0 Description of Loop

The loop has been designed to provide a flexible and reliable irradiation facility. The basic design specifications and operating characteristics of the loop are summarized below:

> to 800°F Bulk Temperature to 600 psig Loop Pressure Material of Construction Type 304 and 316 stainless steel 205 cm^3 In-Pile Volume 5990 cm³ Total Loop Volume with 1200 cm³ liquid in surge tank Maximum Heat Flux of Test Heaters 400,000 Btu/hr-ft² to 1000°F Test Heater Wall Temperature to 15 to 20 ff/sec Velocity in Test Heater

In addition to these characteristics, reliable operation was desired so that long-term continuous "feed and bleed" runs at constant %HB could be made.

The loop flow sheet is given in Figure 1 where it can be seen that the filters, flowmeters, pumps, and test heaters are provided in duplicate and valved so that either of the two units for each component can be used. This procedure provides reliable operation for long-term runs since a component which has failed can be immediately replaced by its duplicate and the run continued. The use of two test heaters will be used to provide information on fouling of the heat transfer surfaces. Other components included are a surge tank to provide sampling holdup and to allow for thermal expansion of the organic liquid, an inpile organic holdup capsule positioned in the center of an

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FIGURE | FLOW DIAGRAM

MITR removable plate fuel element with the eight central plates (of sixteen) removed, two "Dowtherm A" reflux coolers for removing the excess heat, a pressurizing system, a safety expansion system for emergency depressurization of the loop, and a feed and dump tank. All out-of-pile components are enclosed in a cabinet and the in-pile section is enclosed in aluminum conduit to reduce the hazard of fire in case of accidental leakage.

The position of the organic loop with respect to the MITE is illustrated schematically in Figure 2. Figures 3, 4 and 5 and 6 illustrate the instrument panel, the outof-pile section of the loop (with panels removed), the fuel element assembly and in-pile assembly for insertion in the reactor, and the equipment in place in the reactor building where out-of-pile testing has been completed. A detailed description of this equipment is given in the First Annual Report (1).

At the present time, pre-irradiation testing of both the in-pile and out-of-pile units of the loop are complete and the fuel element assembly is in the central position of the reactor core ready for insertion of the in-pile The main difficulty encountered during testing section. of the loop has been with the canned-motor Chempumps (Model CFHT-3-3/4S) used. It has been necessary to return each pump to the manufacturer three times, once for rebuilding to reduce thrust bearing wear and twice to have the motor stator rewound due to winding shorts. Steps have been taken by the manufacturer to eliminate the motor stator shorts and in-pile loop operation is now expected by the middle of July, 1961.

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FIGURE 2 SCHEMATIC SHOWING LOCATION OF LOOP IN THE M.I.T. NUCLEAR REACTOR



Main Cooler Trace Heater Controls and Ammeter Thermocouple Rotary and Key Switches 8



FIGURE 4 HYDRAULIC CONSOLE



FIGURE 5 FUEL ELEMENT ASSEMBLY AND IN-PILE SECTION THIMBLE ASSEMBLY



FIGURE 6 HYDRAULIC CONSOLE AND INSTRUMENT PANEL IN POSITION ON PLATFORM IN REACTOR ROOM

4.0 Experimental Program

The experimental program has been planned so as to obtain the maximum amount of information from operation of the loop. The information obtained should be useful in the economic and technical evaluation of organic moderated and cooled nuclear reactors using either the old or new and improved coolants. It is also hoped that the detailed chemical measurements to be made will be useful in evaluating the degradation mechanism of organic materials.

4.1 Chemical Changes Induced

The chemical changes induced in the organic material and the rate of appearance or disappearance of various components in the coolant will be measured as completely as possible. The following measurements will be made:

a) Vapor Phase Chromatograph

A Burrell K-7 high temperature vapor phase chromatograph with temperature programming will be the primary method of analyzing the irradiated organic coolant. This instrument containing both flame and electron ionization detectors will be used to give a quantitative analysis of the biphenyl, e-terphenyl, m-terphenyl, and p-terphenyl, using an Apiezon L column operated up to 285°C. The composition of the high boiler will be determined as completely as possible by the use of a silicone rubber column operated up to 400°C. although identification of individual components is limited by lack of availability of pure material for use as standards. The analysis of the material will, however, be quantitative at least for triphenylene and quaterphenyl and changes in the relative composition of the components of the high boilers which do not have too high a boiling point for detection by the gas chromatograph will be followed even though a positive identification of the compound is not available. The gas

composition and low boiling material composition will also be determined by use of this instrument.

In Figure 7 is illustrated a typical analysis of a biphenyl, o-terphenyl, m-terphenyl and p-terphenyl mixture dissolved in benzene and using triphenylmethane as an internal standard. The instrument has been available since January and is now calibrated for analysis of these four materials. Work is continuing on analysis of the high boiler, low boiler, and gases produced by irradiation degradation. In addition to Apiezon L and silicone rubber, other stationary phases are under investigation for possible use. The rate of disappearance or appearance of each component will be correlated with the radiation dose to determine "G" values or molecules formed or degraded per 100 ev absorbed.

(b) Carbon-Hydrogen Content

The carbon-hydrogen content of the coolant will be determined by combustion.

(c) Infrared and Ultraviolet Absorption

Infrared and ultraviolet absorption spectra of the organics before and after irradiation will be measured to provide additional information concerning the chemical changes occurring. However, in view of the complexity of the organic composition, interpretation of the spectra may not be possible.

(d) Average Molecular Weight of High Boiler Material

The average molecular weight of the high boiler material will be measured by the cryoscopic method using diphenyl ether. This measurement will be of particular importance in the long-term "feed and-bleed" runs in indicating the behavior of the high boiling materials over a long period of operation such as would occur in a





large scale nuclear power reactor. Over a long period of operation, the solubility limit of some higher boiling materials could be reached if the molecular weight increases continually during reactor operation; should this happen fouling of the heat transfer surfaces might result.

(e) Micro-Distillation

As a check on the VPC analysis and to provide high boiler material for the average molecular weight determinations, a concentric-tube multi-stage micro-distillation column has been set-up at MIT. The column has a holdup of 0.2 ml. and a procedure for distilling the irradiated material is presently being developed.

(f) Gas Content

The amount of gas dissolved in the organic material will be measured in an apparatus constructed and tested at MIT. Samples of this gas as well as samples taken from the gas space above the liquid level in the surge tank will be analyzed for chemical composition by mass spectrometry. Determination of the total gas in the loop and correction for gas leakage should provide the gas evolution rate in molecules per 100 ev absorbed.

(h) Activation Analysis

Measurement of the coolant gamma activity will be made at MIT using a NaI crystal and 256 channel analyzer. This measurement indicates the buildup of particular impurities in the coolant. In the present case, use of stainless steel construction should result in a minimum coolant activation. Correlation of the coolant activity with ash content will be attempted.

(g) Ash Content

To determine the inorganic impurity level in the coolant, the ash content will be measured. In addition, emission spectrography will be used to determine the chemical composition. of the ash. The inorganic content of the coolant appears to be important in fouling of heat transfer surfaces and is the primary source of radioactivity in the coolant.

4.2 Physical Properties

Various physical properties, particularly those important in heat transfer correlations, will be measured and are summarized below.

(a) Viscosity and Density

The viscosity and density of the organic fluids are important in correlating heat transfer data and in estimating pressure drops in the engineering design of reactors. These measurements will be made at MIT over the temperature range of 400-800°F and a high temperature salt bath controllable to $\pm 1^{\circ}F$ has been set-up for this purpose. The viscosity will be measured using a Cannon semi-micro Ostwald-type viscometer; the density will be measured by using a pycnometer in which the volume of a known mass of fluid will be determined by measuring the liquid height in two capillary tubes connected to a small reservoir of fluid. At the present time, several viscosity measurements have been made and work is beginning on the density measurements. Nitrogen pressure is used to prevent boiling of the organic and the pressurizing system is arranged so that a differential gas pressure can be put across the viscometer for measurements at different shear rates.

(b) Heat capacity and Thermal Conductivity

Heat capacity and thermal conductivity are needed for correlation of heat transfer data. The heat capacity will be measured at about every 5-8% increase in the high boiler concentration whereas the thermal conductivity will be measured every 10-15% increase due to the large sample size required. A drop calorimeter will be used for the heat capacity measurement and a method similar to that of D. W. McCready (WADC Tech. Report 58-405) will be used to measure the thermal conductivity.

(c) Melting Point

The melting point of the irradiated organic material is helpful from an operational point of view and will be measured using the Fisher-Johns technique.

5.0 Heat Transfer

As indicated in the loop description, two test heaters have been provided for heat transfer measurements with organic velocities up to 20 ft/sec and heat fluxes up to 400,000 Btu/hr-ft². Each test heater consists of a 1/4" 0.D x 0.020" wall stainless steel tube which is heated internally by using the tube as a conductor for large A.C. currents at low voltage. The heater construction and thermocouple placement is indicated in Figure 8.

Heat transfer measurements have been made during out-of-pile testing of the loop and in Figure 9, a plot of the experimental temperatures used in determining the heat transfer coefficient is presented. As can be seen, there is a rather large scatter of temperatures about the mean and steps are being taken to reduce this scatter. A calibrating furnace for the test heater thermocouples has been ordered which should increase the accuracy of the heat transfer measurements on future test heaters.

In Figure 10 values of h measured are compared with one of the basic heat transfer correlations with good agreement.

The experimental program has been planned to obtain both the heat transfer rates through the fluid film and fouling of the heat transfer surface. Two approaches will be made for detecting fouling of the heat transfer surface. First of all, at the beginning of an experimental run, two new and unfouled heaters will be placed in the loop. Heat transfer measurements will be performed on both heaters at the beginning of an irradiation experiment. One of the heaters will be valved off, allowed to cool, and will not be used for the remainder of that experiment





q/A = 176,400 Btu/hr - Ft²



FIGURE 10 COMPARISON OF EXPERIMENTAL HEAT TRANSFER DATA WITH STANDARD HEAT TRANSFER CORRELATION

whereas the other will be used continuously for heat transfer measurements and as a heat source for operation of the loop. At the conclusion of the experiment, which may last a few months, heat transfer measurements will again be made on both test heaters and **the results** compared. This procedure should cancel out the effect of physical property changes in the irradiated organic liquid and should indicate any appreciable fouling of the heater which was in continual operation.

The second method involves application of the "Wilson Method" for determining fouling resistances using the test heater which is operated continuously. Since for the present case, the effective heat transfer coefficient based on the inside wall temperature and the bulk fluid temperature is measured, the heat transfer coefficient, U, includes a fouling resistance, R_d , and a fluid resistance R_e where $\frac{1}{U} = R_d + R_e$. Now, if a series of heat transfer measurements are made at any time at a given bulk fluid temperature, the liquid physical properties remain constant and the Dittus-Boelter correlation reduces to:

 $h_e = \frac{1}{R_e} = CV^{0.8}$

Hence, $\frac{1}{U} = R_d + \frac{1}{CV^{0.8}}$

If U is measured at several different velocities and $\frac{1}{U}$ is plotted vs. $\frac{1}{\sqrt{0.8}}$, a straight line should be obtained which on extrapolation to $\frac{1}{\sqrt{0.8}} = 0$ should give R_d directly since R_d is not a function of velocity.

It is felt that the two methods to be used for fouling studies should indicate any significant fouling of the heat transfer surfaces where fouling occurs in the absence of irradiation at the heat transfer surface. Before applying the data to the standard heat transfer correlations, this fouling resistance, if any, will be subtracted from the data.

6.0 Dosimetry

Since it now appears that the G(-coolant) value (molecules decomposed per 100 ev of energy absorbed) for fast neutrons is greater than that for gamma photons⁽²⁾, (3) an important phase of the program at MIT has been the measurement of the fast neutron and the gamma dose rate in the organic material being exposed to the radiation field in the MITR core. With this objective in mind, calorimetric measurements of the dose rate in several materials have been completed in the No. 1 fuel element position of the MITR and, based on these results, the dose rates due to fast neutron interactions and due to gamma interactions in Santowax OMP have been determined.

Due to space limitations, it was not possible to perform the calorimeter measurements with the in-pile section in place. However, to correct for perturbations in the dose rate due to insertion of the in-pile section, measurements were made in both an aluminum thimble and in a stainless steel thimble which closely approximates the reactivity and flux depression effect of the in-pib section filled with organic. Additional information will be obtained by the use of monitor wire activation and ionization chamber measurements. The activation and ionization chamber measurements will be made with the in-pile section in place utilizing a small diameter $(0.242" I_D)$ monitor tube which extends from the reactor top to the bottom of the irradiation capsule. These measurements will also be used to determine changes in the dose rate at any time during the irradiations.

6.1 Calorimetry

The method of calorimetry used was measuring the adiabatic rate of temperature rise in several samples having different neutron scattering characteristics. The samples used and their pertinent properties are given in Table 1. By consideration of the gamma and fast neutron interactions taking place in the various materials, the dose rate was separated into the fraction due to gamma radiation and the fraction due to fast neutron scattering. The earlier calorimetry results as well as a description of the calorimeter and technique used in all measurements are described by Fischer⁽⁴⁾.

Six different sets of calorimeter measurements were made over a period extending from March 25, 1960 to March 27, 1961. The last two measurements (March 20 and 27, 1961) were made in a stainless steel thimble (instead of Al) whose reactivity was approximately that of the in-pile section and which was calculated to give the same thermal flux depression as the in-pile section filled with organic material. Measurements were made at reactor power levels of O(background), 50kw, 100kw, 200 kw, and 500 kw with the major portion of the measurements performed at 50 and 100 kw. All measurements, when extrapolated linearly to a power level of 1 Mw fell within + 15% of a mean value, even though the time period covered included several fuel rearrangements in the reactor and installation of the stainless steel thimble in place of the aluminum thimble in the reactor. No measurable difference in the dose rate could be detected when the stainless steel thimble was substituted for the aluminum thimble.

The axial dose rate distribution for Santowax OMP is presented in Figure 11.

6.2 Monitoring Methods

Various monitoring methods have been developed and will be used throughout an irradiation to check for variations in the gamma and fast neutron dose rates.

TABLE 1.

ABSORBER PROPERTIES USED FOR CONVERSION OF DATA

	Chemical Composition	Z/A	N _H Atoms H/g	N _C Atoms C/g
Aluminum		0.482	-	_
Beryllium	-	0.444	-	-
Polyethylene	(CH ₂)	0.571	8.61×10^{22}	4.30×10^{22}
Polystyrene	(C6H5CHCH2)	0.539	4.63×10^{22}	4.63×10^{22}
Santowax OMP	((c ₆ H ₅) ₂ c ₆ H ₄) _n	0.530	3.66×10^{22}	4.72×10^{22}



FIGURE II DISTRIBUTION OF THE NEUTRON, GAMMA AND TOTAL DOSE RATES FOR SANTOWAX OMP AT I MW AS CALCULATED FROM CALORIMETRIC DOSE RATE MEASUREMENTS

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The methods are summarized below.

(a) Threshold Detectors for Fast Neutrons

In order to monitor the fast neutron flux in the higher energy range. The three threshold detectors listed in Table 2 will be used.

TABLE 2.

Threshold Detectors to Be Used for Monitoring Fast Neutron Dose Rate

Reaction	Effective Threshold Energy Mev	Half-Life of Product
$S^{32}(n,p)P^{32}$	2.9	14.3 days
$Mg^{24}(n,p)Na^{24}$	6,3	15.0 hrs.
$Al^{27}(n,\alpha)Na^{24}$	8.6	15.0 hrs.

Irradiations have been carried out with all of the materials listed. However, in the case of the $S^{32}(n,p)P^{32}$ reaction, reproducible results could not be obtained due to the difficulty in counting the 1.71 Mev β particles emitted. A counting method to permit use of the $S^{32}(n,p)P^{32}$ reaction is now being developed. Reproducible count rates were obtained grom the Mg²⁴(n,p)Na²⁴ and the Al²⁷(n,a)Na²⁴ reactions and a typical set of data are presented in Figure 12. The axial flux distribution and the relative difference between the 8.6 Mev threshold and the 6.3 Mev threshold are clearly shown. The count rates shown have not been corrected for counter efficiency.



FIGURE 12 FAST FLUX DISTRIBUTION (INTEGRATED)

(b) Thermal and Resonance Neutrons.

While thermal neutrons in themselves cause no radiation damage to the organic material, both the gamma and fast neutron dose rates are strongly dependent on the thermal neutron flux. Accordingly, irradiation of 0.5% Co-Al wire bare and cadmium covered will be used to determine the thermal neutron flux; since Co⁵⁹ has a resonance at 120 ev, the cadmium covered measurement will be used to monitor the epithermal neutron flux which does cause irradiation damage. Both bare and Cd-covered relative measurements have been made. Figure 13 presents the difference between the cadmium covered and bare cobalt activity as a function of position in the reactor core.

As in the case of the data presented in Fig. 12, the data of Fig. 13 have not been corrected for counter efficiency. Calibrated sources are being used to calibrate the counting equipment to give the absolute count rates so that the neutron fluxes can be calculated.

🧼 (c) Gamma Flux Measurement.

A small ionization chamber utilizing the monitor tube wall as the ground potential and a center electrode and cable fabricated from aluminum wire swaged with Al₂0₃ insulation into an aluminum sheath has been designed, constructed and is undergoing testing. Results to date have been encouraging although the data has not been as reproducible as would be desired. Testing and minor modifications are still in progress so that the device will be available for routine measurements during loop operation. Figure 14 shows an axial traverse made in the central fuel position with **the** reactor at full power.



FIGURE 13 BARE LESS Cd - COVERED COUNTING RATES FOR Co⁵⁹-AI WIRE IRRADIATED

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FIGURE 14 GAMMA DOSE RATE DISTRIBUTION ALONG THE CORE AS MEASURED BY THE ION CHAMBER

7.0 Schedule of Loop Operation

As previously mentioned, the first in-pile loop operation is expected by the middle of July. The first experiment will be with Santowax OMP as this is the material now usually specified for use in organic moderated and cooled nuclear reactors. This first experiment will start with distilled and filtered reactor grade Santowax OMP and irradiation will continue to a high boiler content of 50-60 wt. %. The wt. % HB will then be lowered to what is indicated to be the optimum concentration for reactor operation (probably 30-40% HB) and a long-term "feed and bleed" run at constant % HB will be made. At the completion of this run, a new material such as a refinery side stream may be selected, after consultation with the AEC and its contractors, and the above experimental program repeated with the new material. In all cases, the experimental program will be flexible and constant evaluation of new and old materials will be made for testing in the loop.

* 98% ortho, meta, and para terphenyls (approx. 12% ortho, 61% meta, and 25% para) and containing less than 2% diphenyl and pyrolytic tars.

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