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THERMAL COKING CAPSULE EXPERIMENTS ON ORGANIC REACTOR COOLANT

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STATEMENT OF PROBLEM			
Determine the pyrolysis rate of irradiated Santowax OM coolant in a static nonradioactive environment.			
ABSTRACT			
The thermal decomposition of irradiated U-3 coolant (Santowax OM) was tested in sealed capsules at 550° and 750°F for periods of 3, 6, and 11 months. Some tests were also made with unirradiated Santowax OM and capsules provided with relief valves. Little effect was observed at 550°F, but at 750°F extensive decomposition occurred leading to nearly complete solidification in six months. Rate constants were estimated for terphenyl disappearance and HB formation.

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I. INTRODUCTION

Pyrolytic decomposition of stagnant organic coolant could adversely affect reactor operation. To evaluate coolant decomposition without radiation or stirring, samples of irradiated coolant were tested for up to 11 months at 550° and 750°F. Samples were contained in stainless steel capsules. Capsules were removed from test at the end of 3, 6, and 11 months. Samples were analyzed for changes in coolant and cover gas composition, and the capsule walls were examined for film deposition. Unirradiated coolant was also tested for 3 months at 550° and 750°F.

II. EXPERIMENTAL METHODS

A. CONTAINMENT CAPSULES

Stainless steel capsules were fabricated as shown in Figure 1. Prior to loading coolant, the capsule interiors were etched in a solution of 10 parts concentrated nitric acid, 5 parts hydrofluoric acid, and 25 parts distilled water, by volume. After thorough rinsing in distilled water, the capsules were air-dried at 300°F. Capsules and caps were stamped with identifying numbers, and weighed to ± 0.001 grams. Two capsules were fitted with Circle Seal stainless steel pressure relief valves set to relieve at an internal pressure of 300 psig.

B. COOLANT SAMPLES

The coolant used for these tests was irradiated Santowax OM from the U-3 Loop at CRNL. As the test progressed and data analyzed from the three- and six-month tests, results indicated that it would be of interest to determine the pyrolysis rates of unirradiated Santowax OM under similar conditions. For this reason additional capsules were prepared and loaded with as-received Santowax OM from lot number AE-3-B7. The chemical compositions of as-loaded coolants are presented in Table I.

CAPSULE SCHEMATIC

PART No. 2
SWAGelok 0.5 in.
CAP. 316 SS
10 EACH

PART No.1 CAPSULE
12 EACH

PART No.3
PRESSURE
RELIEF
2 EACH

Figure 1

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TABLE I
CHEMICAL COMPOSITION OF COOLANTS USED IN THERMAL COKING EXPERIMENT

	Santowax OM, Irradiated U-3 Loop Coolant	Santowax OM, Unirradiated As-Received
Biphenyl (%)	1.6	2.2
Terphenyl		
Ortho (%)	48.6	57.7
Meta (%)	20.3	30.5
Para (%)	2.1	4.2
Low Boilers (%)	None detectible	0.3
Other Low Boilers (%)	0.1	.04
Intermediate Boilers (%)	7.0	3.7
High Boilers (%)	22.5	1.32
Ash (ppm)	< 8	No data
MST (A/mg x 10 ⁻⁵)	98	4
Total O ₂ (ppm)	510	200-400
Carbonyl Oxygen (ppm)	13	62
C/H Atomic Ratio	1.30	No data
Water (ppm)	680	200-300

C. SAMPLE LOADING

Loading of coolant samples into capsules took place in an inert atmosphere box. The atmosphere was controlled to contain less than 10 ppm oxygen and water. Approximate gas analysis at the time of loading was 80% argon, 10% nitrogen, 5% hydrogen and 5% helium. Capsules were filled with coolant to one-half their volume, approximately 10 grams of sample per capsule. After atmosphere samples were taken for mass spectrographic analysis, the caps were installed on the capsules and the units sealed. Following this, the capsules were transferred out of the box, the final coolant weight was determined, and each unit was helium leak checked. The two capsules fitted with pressure relief valves were pressurized to 300 psi with nitrogen, the gas filled tube crimped, and welded closed. The units were then bubble checked for leakage.

D. TEST EQUIPMENT

Two resistance furnaces, operating at 550°F and 750°F respectively, contained the capsules during the test. Capsules were held vertically in the furnaces in a cylindrical aluminum block, drilled to accept the capsules to a depth level with the sample in the capsule. A thermocouple well was also drilled into the aluminum block, to provide a means of temperature measurement and control. Those capsules in each test which were fitted with a pressure relief valve were permitted to extend above the furnace top and through the insulating cover. This was done to prevent thermal degradation of the Viton relief valve seals. Additional cooling of the 750°F pressure relief valve capsule was accomplished with a fan. A thermocouple was attached to each relief valve, for continuous temperature recording. Potentiometric instruments were used to measure and record temperature data. The recorders were calibrated against a precision potentiometer at the start of the test, and recalibrated during the test. Daily temperature measurements were made with potentiometers to verify the recorded data.

III. SAMPLE HANDLING AFTER TEST

The amount of gas generated during test was measured using the apparatus shown in Figure 2. The system consisted of a chamber and piping of known volume, attached to a mercury manometer. The pressure was measured before and after opening a capsule. The quantity of gas generated was calculated using the ideal gas law. Immediately after measuring the pressure of the newly opened capsule, a sample of gas was withdrawn for mass spectrographic analysis. Except for one six-month, 750°F capsule, coolant was drained (or chipped) from the capsules. Following removal of the coolant the capsules were slit open using a water-cooled abrasive wheel and the interior surfaces examined for evidence of film deposition. One six-month, 750°F capsule, was slit open prior to removal of the residual coolant slug.

Methods developed and reported by R. T. Keen, et al.,⁽¹⁾ for analysis of polyphenyl reactor coolants were used for analysis of thermal coking samples. Gas chromatographic methods were used for determinations of biphenyl, o-, m-, and p-terphenyl, benzene, toluene, ethyl benzene, and intermediate boilers. Other low boiling components (OLB) were also determined collectively with a gas chromatograph. Carbonyl oxygen content was determined using a spectrophotometer, applying the method described by Sullivan and Bowe.⁽²⁾ The membrane stain test (MST) is described by R. C. Shepard.⁽³⁾ The quantity of high boilers was arrived at by difference. "Coke" was estimated as material insoluble in tetrahydrofuran (THF). Test results are reported in Tables II and III.

IV. RESULTS

A. THREE-MONTH CAPSULES (IRRADIATED COOLANT)

Two capsules in each temperature group were removed after three months of test. Radiographs of the capsules indicated a clearly visible coolant level in all four. Capsules were helium leak checked, to determine if any of the blanket gas was leaking. No leaks were indicated. Capsules were opened, the gas pressure measured, and samples of the gas withdrawn. Following this the coolant was removed by inverting the capsules and

CAPSULE OPENING APPARATUS
THERMAL COKING EXPERIMENT
FIGURE 2

TABLE II
THERMAL COKING CAPSULE TEST RESULTS - 550°F

	Unirradiated OM			Irradiated U-3 Coolant						
	As-Loaded	Capsule #13 3 Months	Capsule #14 3 Months	As-Loaded	Capsule #8 3 Months	Capsule #10 3 Months	Capsule #11 6 Months	Capsule #12* 6 Months	Capsule #7 11 Months	Capsule #9 11 Months
O-terphenyl	57.70	62.50	62.04	48.57 46.11	46.13	50.74	49.13	42.22	47.60	46.99
M-terphenyl	30.49	31.22	31.27	20.28 19.70	21.51	19.43	18.80	19.47	20.38	21.40
P-terphenyl	4.20	4.15	3.32	2.06 2.23	1.68	1.44	2.18	2.24	1.95	1.76
Biphenyl	2.24	1.87	2.16	1.60 1.56	1.60	1.91	2.21	0.45	1.76	1.58
Intermed. Boiler	3.70	3.24	3.25	7.02 2.31	2.5	2.9	3.61	2.31	4.0	3.3
Benzene	< .01	N.D.	N.D.	N.D. N.D.	N.D.	-	N.D.	N.D.	.02	.02
Toluene	.30	N.D.	N.D.	N.D. N.D.	N.D.	-	.02	.01	.03	.03
Ethyl Benzene	N.D.	N.D.	N.D.	N.D. N.D.	N.D.	-	.02	N.D.	.02	.02
Other Low Boil	.04	N.D.	.04	0.14	.05	-	.07	N.D.	.08	.07
High Boilers	1.32	Total shows 2.98% >100%	Total shows 2.08% >100%	22	26.53	-	23.96	33.30	24.16	24.83
C/H Ratio	1.28	1.27	1.27	1.30 & 1.29	1.30	1.29	1.30	1.28	1.30	1.29
Carbonyl O ₂ ppm	61 & 63	65 & 66	61 & 64	12 & 15	23 & 21	25 & 27	24	27	38	81
Gas Generated w/o	-	< 0.01	< 0.01	-	< .01	< .01	-	-	0.093	0.111
Benzene Insol.	-	-	-	-	-	-	.05	.08	-	-
THF Insol.	-	-	-	-	.2 & 1.	.04 & .06	< .1	< .1	-	-
MST	4.0 & 3.6	9.7	11.2	99 & 97	462 & 338	234 & 213	475	> 1400	837	1282

*Capsule with pressure relief valve

TABLE III
Thermal Coking Capsule Test Results - 750°F

	Unirradiated OM			Irradiated U-3 Coolant						
	As-Loaded	Capsule #17 3 Months	Capsule #18 3 Months	As-Loaded	Capsule #4 3 Months	Capsule #5 3 Months	Capsule #1* 6 Months	Capsule #6 6 Months	Capsule #2 11 Months	Capsule #3 11 Months
O-terphenyl	57.70	15.04	15.20	48.57 46.11	1.9	1.5	0.61 .59	N.D.	-	-
M-terphenyl	30.49	16.43	14.19	20.28 19.70	6.9	5.7	3.02 1.16	N.D.	-	-
P-terphenyl	4.20	1.78	1.20	2.06 2.23	1.3	2.5	1.16 0.56	N.D.	-	-
Biphenyl	2.24	10.64	9.10	1.60 1.56	14.0	12.4	6.99 3.37	0.21	-	-
Interced. Boiler	3.70	1.77	2.05	7.02 2.31	1.5	0.5	40.6 N.D.	N.D.	-	-
Benzene	4.01	0.99*	0.89**	N.D. N.D.	-	10.2	- N.D.	.02	-	-
Toluene	.30	0.07	0.06	N.D. N.D.	-	0.5	- N.D.	N.D.	-	-
Ethyl Benzene	N.D.	0.08	0.03	N.D. N.D.	-	0.1	- N.D.	N.D.	-	-
Other Low Boil.	.04	.16	.10	0.14	-	0.2	- -	-	-	-
High Boilers	1.32	53.04	57.18	22	-	66.4	- 94.32	99.77	-	-
C/H Ratio	1.28	1.33	1.34	1.30 & 1.29	1.38	1.44	1.95 2.18	2.25	2.46	2.25
Carbonyl O ₂ ppm	61 & 63	4 & 3	18 & 12	12 & 15	11	11	- 40	26	46	86
Gas Generated w/o	-	-	-	-	1.2	1.2	- -	-	10.4	9.4
Benzene Insol.	-	-	-	-	10.0	10.0	- -	-	-	-
THF Insol.	-	-	-	-	6.3	5.7	- 85.1	95.2	99.79	98.48
MBT	4.0 & 3.6	300 & 235	825 & off range	99 & 97	-	-	- -	-	-	-

* Capsule with pressure relief valve

**Large peak preceding benzene during gas chromatographic analysis

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permitting the samples to flow into containers. During transfer of the coolant, it was observed that the 750°F material was noticeably more viscous than the 550°F coolant. Some external warming of the 750°F capsules was necessary for the coolant to flow freely. Closer examination of the 750°F material showed it to be elastic and tar-like, with a shiny surface. When an MST was attempted on the 750°F samples, the amount of filtered residue amounted to 10%, placing the MST value beyond test limits. The filtered material was submitted for x-ray fluorescence and diffraction; no elements or compounds were detected. Radiation readings of the filtered residue indicated 26 counts/minute above background. The empty capsules were slit along their length, exposing the inner surfaces (Figure 3). Capsules were then washed in tetrahydrofuran (THF) to remove all traces of coolant.

B. THREE-MONTH CAPSULES (UNIRRADIATED OM COOLANT)

Radiographs of the four capsules showed a clearly discernible liquid level. No evidence of gas leakage was found by helium leak checking. Capsules were opened, pressure measured, and gas samples taken. The coolant was removed from the capsules at room temperature. The 550°F material was a viscous liquid, slightly brownish in appearance. The 750°F material was dark brown, completely liquid, somewhat similar in consistency to that of U-3 coolant. Figure 4 shows the capsules after being slit open and cleaned with THF.

C. SIX-MONTH CAPSULES (IRRADIATED COOLANT)

The two capsules fitted with pressure relief valves, along with two other capsules were removed from test at the end of six months.

Radiographs showed a clear coolant level only in the 550°F capsules. The high temperature capsules showed evidence of voids throughout the coolant, and no definite gas-liquid interface. The 750°F capsules were reheated successively to 130°C, 200°C, 250°C, and 300°C, with no change being observed in the radiographs. All capsules were opened. The gas train was broken during test, so the volume of gas generated was not measured. No problems were encountered in removing the coolant from the low temperature capsules. Coolant in the high temperature capsules

#11, 550°F, 6 MONTHS

TOP

#12, 550°F 6 MONTHS, WITH PRESSURE RELIEF VALVE

#8, 550°F, 3 MONTHS

TOP

#10, 550°F, 3 MONTHS

TOP

#4, 750°F, 3 MONTHS

TOP

#5, 750°F, 3 MONTHS

TOP

#1, 750°F, 6 MONTHS, WITH PRESSURE RELIEF VALVE

#6, 750°F, 6 MONTHS

TOP

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Figure 3. Slit 3-Month and 6-Month
U-3 Capsules

7680-40199

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OM LOAD

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#13 - 550°F 3 MOS. TOP

#14 - 550°F 3 MOS. TOP

#17 - 750°F 3 MOS. TOP

6-23-67 #18 - 750°F 3 MOS. 7710-4063CN TOP

Figure 4. Slit 3-Month OM Capsules

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had solidified, as indicated by visual examination and probing with a needle. Numerous chunks and a length of carbonaceous material (Figure 5) were removed from the capsule without a relief valve, with a minimum of probing. Figure 6 shows this length of material after being broken up for closer examination. Figure 7 shows a doughnut shaped section found in the length of solidified coolant. Samples removed from the inner and outer portions of the doughnut had the same C/H ratio (2.25).

Attempts to remove solidified coolant from the other 750°F capsule (No. 1, with pressure relief valve) by probing were unsuccessful. Probing was kept to a minimum, as it was desired to maintain the interior of the capsule free from marks and scratches. The capsule was slit open with the solidified coolant inside, as shown in Figure 8. The upper three-fourths of the sample was hard carbonaceous chunks, with the lower one-fourth containing some semiliquid material along with chunks and pieces. The two types of material were separated and analyzed as sample No. 1 and No. 1-A, the 1-A being assigned to the semiliquid material. As seen from Table III, the composition of 1-A differs from that of 1.

The pressure relief valves which had been removed from the capsules were tested to determine if the relief pressure had changed. The valve fitted to the 550°F capsule released at 450 psig, and the 750°F valve at 425 psig, initially. On repeated tests the valves relieved at 300 psig. Disassembly of the valves showed some organic coating of the inner parts.

D. ELEVEN-MONTH CAPSULES (IRRADIATED COOLANT)

Radiographs of the four capsules removed after eleven months in test indicated a clear sample level in only the 550°F capsules. The 750°F capsules showed voids, and in general were similar in appearance to the six-month capsules. Helium leak checking indicated no discernible leaks. Capsules were opened, the pressure measured, and gas samples withdrawn. Removal of the low temperature samples was accomplished without difficulty. The capsules were warmed slightly to permit complete removal of the coolant. The coolant in the high temperature capsules had

REMOVED FROM #6 , 750 °F , 6 MONTHS

Figure 5. Length of Solidified Coolant
Removed from Capsule No. 6.

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Figure . Sectioned Lengths of Material
Shown in Figure 5. _____

Figure 7. "Doughnut" Sample from
Material Shown in Figure 5.

CAPSULE #1
750°F, 6 mos.

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Figure 8. Slit Capsule No. 1.

solidified, as indicated by the radiographs. Probing at the solidified organic showed it to be loose and easily dislodged from the capsule walls. Both 750°F capsules were emptied in this manner. The emptied capsules were slit open to expose the inner surfaces and washed in THF. The appearance of the walls was similar to the six month capsule.

E. ANALYSIS OF GAS SAMPLES

Mass spectrographic analyses of gas samples gave inconclusive quantitative results. The analyses did indicate that methane and hydrogen were formed in high concentration with some ethane and benzene also being formed.

V. DISCUSSION OF RESULTS *

A. 550°F TESTS

Essentially no change in composition of irradiated coolant occurred after eleven months at 550°F. The only parameter that did change measurably was the MST result, which increased from 100 to 1000 ($A/mg \times 10^{-5}$) over a period of eleven months.

B. 750°F TESTS

Considerable decomposition of both irradiated and unirradiated coolant occurred even after three months in test at 750°F, a decrease in total terphenyl from 70% to 10% for irradiated coolant, and a decrease in total terphenyl from 100% to 30% for unirradiated coolant. After six months in test, essentially all the irradiated coolant had been converted to HB, coke, or gas. Assuming the applicability of first order kinetics, terphenyl decomposition rates have been calculated for the three and six month 750°F runs (Table IV).

*The authors would like to acknowledge Dr. R. A. Holroyd's contribution to this section.

TABLE IV
TERPHENYL DISAPPEARANCE RATES, 750°F

	First Order Rate Constant (hr ⁻¹ x 10 ⁴)			
	Unirradiated OM	Irradiated OM		Other Work
	This Work	This Work		
	3 Month	3 Month	6 Month	
Total terphenyl	4.7	9.0	8.3	3.6*
o-terphenyl	6.1	15.2	10.5	1.7**
m-terphenyl	3.1	5.3	7.0	

* Reference 4. Short-term tests with OM.

**Reference 5. Short-term tests with pure o-terphenyl.

The results indicate that the rate constants for the disappearance of terphenyls in irradiated OM are approximately twice those for unirradiated terphenyls. The rate constant for the disappearance of OM as determined in this work is approximately the same as determined elsewhere. The rate constant for the disappearance of o-terphenyl as determined in this work is 3-1/2 times greater than for pure o-terphenyl. No explanation for this result is offered.

A similar comparison has been made for HB formation rates (Table V). Because of a lack of understanding of the mechanisms of HB formation, comparison with other workers' data is probably not pertinent. The results do show that for at least the first three months, the HB formation rates for both the unirradiated and irradiated coolants used in these tests are the same.

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TABLE V

HB FORMATION RATES, 750°F

Starting Material	Average HB Concentration-%	Rate Constant - $\text{hr}^{-1} \times 10^4$	
		1st Order	-1 Order
Unirradiated OM	25 (3 month test)	3.6	1.6
Irradiated OM	44 (3 month test)	3.8	1.1
Irradiated OM	49 (6 month test)	2.6	0.6
Unirradiated OM (Reference 4)	3 (short-term)	1.2	1.2
Irradiated OM (Reference 6)	7 (in-pile)	4.8	4.15
	22 (in-pile)	6.8	4.16
	36 (in-pile)	10.2	4.16

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