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HEAVY WATER ORGANIC COOLED REACTOR

PHYSICAL PROPERTIES OF SOME POLYPHENYL COOLANTS

BY
H. MANDEL

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A Joint Venture for Heavy Water Organic Cooled Reactors

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CONTENTS

	Page
Abstract	5
I. Introduction.	7
II. HWOCR Polyphenyl Coolant Candidates	9
III. Thermophysical Properties	11
IV. Vapor Pressure	25
V. Flammability Characteristics.	29
VI. Heat of Fusion and Melting Point of Pure Polyphenyls.	31
VII. Conclusions.	35
VIII. Recommendations for Future Work	37
References	39
Appendix	41

TABLES

1. Composition and Liquidus Point Data for Polyphenyl Coolants	10
2. Summary of Physical Properties Data for Candidate HWOCR Coolants	13
3. Vapor Pressure of Unirradiated Santowax OM at 750°F.	27
4. Vapor Pressure of Unirradiated Santowax DOM at 750°F.	27
5. Vapor Pressure of Unirradiated Santowax HOM at 750°F.	28
6. Heat of Fusion of Biphenyl, o-, and m-Terphenyl	31
7. Comparison of Actual and Calculated Eutectic Composition of Santowax DOM	32
8. Physical Properties Data Required for Candidate HWOCR Coolants	37
9. Estimated Accuracy of Coolant Physical Properties Data	43
10. Variance and Precision of Physical Property Factor in Dittus-Boelter Type Heat Transfer Equation for Santowax OM + 10% HB Coolant (Case 1).	45
11. Variance and Precision of Physical Property Factor in Dittus-Boelter Type Heat Transfer Equation for Santowax OM + 10% HB Coolant (Case 2).	46

FIGURES

	Page
1. Viscosity of Irradiated Santowax OMP	15
2. Viscosity of Irradiated Santowax OM	15
3. Viscosity of Irradiated OM-2	16
4. Viscosity of Irradiated Santowax WR	16
5. Viscosity of Irradiated Santowax DOM	17
6. Viscosity of Irradiated Santowax HOM	17
7. Viscosity of Irradiated HB-40.	18
8. Viscosity of Irradiated Isopropylbiphenyl	18
9. Viscosity of Irradiated Biphenyl	19
10. Comparison of Viscosities of Irradiated Coolants at 550°F	19
11. Comparison of Viscosities of Irradiated Coolants at 650°F	20
12. Comparison of Viscosities of Irradiated Coolants at 750°F	20
13. Effect of Irradiation Temperature, HB Content, and Coolant Molecular Weight on Viscosity of Santowax WR.	21
14. Vapor Pressure of Unirradiated Coolants	26
15. Vapor Pressure of Irradiated Coolants.	26

ABSTRACT

The physical properties of several polyphenyl HWO CR coolant candidates are summarized and reviewed. Properties include density, viscosity, specific heat, thermal conductivity, vapor pressure, melting point and heat of fusion, and flammability characteristics. Recommendations are made for additional physical properties investigations in cases where data are lacking or additional data are considered necessary.

I. INTRODUCTION

The data compiled in this report are a summary of the properties of organic coolants required for heat transfer calculations and optimization of the Heavy Water Organic Cooled Reactor (HWOCR). The properties considered include the so-called thermophysical properties (density, viscosity, specific heat, and thermal conductivity), heat of fusion and melting point, flammability characteristics, and vapor pressure. Coolant candidates for near future reactors are restricted to the polyphenyls because of the wealth of experimental and operating data for these materials available from both domestic (OMRE, PNPF) and foreign (AECL, Euratom) organic coolant programs. Furthermore, the cost and availability of polyphenyl coolants are known or can be projected with a degree of reliability consistent with design schedules. Considerable new work is needed on such alternate materials as petroleum and coal tar derivatives before they can be considered as candidate coolants for HWOCR.

The data summarized are presented in tables and figures for convenience of comparison at specific temperatures and high boiler (HB)^{*} concentrations of interest. Recommendations are also made for properties data which are lacking and necessary or which require confirmation, for coolants of major interest.

*The high boiler fraction is defined as those compounds less volatile than the original major coolant components (the least volatile of the terphenyls is p-terphenyl). In general, more than 95% of the decomposition products are high boilers.

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II. HWOCR POLYPHENYL COOLANT CANDIDATES

The polyphenyls being considered as coolant candidates for heavy water moderated organic cooled reactors in the United States and Canada are Monsanto products which can be classified as follows: (a) the derivatives of Santowax R, consisting of Santowax OMP, OM, WR, and HB-40, (b) the derivatives of Santowax OM, consisting of Santowax DOM and HOM, (c) the derivatives of Santowax WR, consisting of Santowax WRH and WRHD, and (d) biphenyl and isopropylbiphenyl. (Santowax R itself has the disadvantages of high melting point due to its high paraterphenyl content, and varying pyrolytic HB content of up to 20 wt %.) Table 1 gives the composition and melting points of these coolants. The proposed coolant for the Euratom ORGEL project is OM-2, which is similar in composition and properties^(1,2) to Santowax WR. No physical properties data are available for the hydrogenated derivatives of WR, listed under (c) above.

TABLE 1
COMPOSITION AND LIQUIDUS POINTS DATA FOR POLYPHENYL COOLANTS

Component	Santowax R Derivatives					OM Derivatives		WR Derivatives		Biphenyl	Isopropyl- biphenyl	Component Liquidus Temperature (°F)
	R	OMP	OM	WR	HB-40	DOM	HOM	WRH	WRHD			
Biphenyl (wt %)	-	-	<3	-	-	29	<0.5	-	7	>99	-	156
Isopropylbiphenyl (wt %)	-	-	-	-	-	-	-	-	-	-	>99	<32
o-Terphenyl (wt %)	>99	10 to 15	55 to 69	12	18	52	56	12	11	-	-	133
m-Terphenyl (wt %)	46	55 to 70	23 to 37	76	-	18	<3	20	19	-	-	189
p-Terphenyl (wt %)	25	20 to 30	<5	5	-	<1	<0.5	-	-	-	-	415
Hydro-terphenyls (wt %)	-	-	-	-	82	-	41	62	58	-	-	NA [†]
HB (wt %)	20	<1	<1	7	-	-	<0.5	6	5	<1	<1	-§
Coolant Liquidus Temperature (°F)	~300	~350	~185	~195	<32	~100	~95	NA [†]	~88	156	<32	-

*Trade name of the Monsanto Chemical Company

†Not available

§ Variable, may be ≥500°C

**Composition is similar to OM-2 used by Euratom (References 1, 2)

III. THERMOPHYSICAL PROPERTIES

The physical properties required for heat transfer calculations and optimization of heat transfer equipment are density, viscosity, specific heat, and thermal conductivity. The data for coolants of interest are summarized in Table 2 as a function of HB (DP* for Santowax WR) from 0 to 30 wt % over the temperature range of 400 to 800°F. The major sources of data, which in some cases have been interpolated or extrapolated from smoothed curves to cover the required temperature and HB conditions, are AI, MIT, AECL, Euratom, United Kingdom, and Monsanto publications.

Density and viscosity data for the lower melting point coolants (Santowax OM, DOM, HOM, OM-2, and HB-40) have been interpolated and replotted from the original AECL data. Since viscosity is quite sensitive to HB content, viscosity data are presented in detail for several HB contents for all coolants of interest in Figure 1 through 9. Figures 10, 11, and 12 specifically compare viscosity of these coolants as a function of HB (excluding the Santowax WR data which were reported as a function of DP) at three temperatures of interest:

reactor inlet temperature	550°F,
average in-core temperature	650°F, and
reactor outlet temperature	750°F.

At all three temperatures, it is apparent that Santowax OMP, OM, HOM, as well as OM-2 and HB-40 fall within a relatively narrow range of higher viscosity, and that Santowax DOM, isopropylbiphenyl, and biphenyl fall within a wider range of lower viscosity in the following order: DOM > IPB >> B. There are restraints on the overall accuracy of the comparison made in the cases of biphenyl and isopropylbiphenyl due to some assumed but uncertain errors in the originally published data⁽⁸⁾ shown in Figures 8 and 9 and Table 2. The biphenyl data, for example, were estimated as being about 10% low.⁽⁴⁾ Some additional experimental verification would be necessary if these two coolants were to be given serious consideration for use in an HWOCR.

Viscosity data for Santowax WR⁽⁵⁾ are shown in Figure 13 as a function of DP content, irradiation temperature and coolant molecular weight. At

*Defined as 100 - wt % (o-, m-, p-terphenyl)

Coolant	HB Content (wt %)	Density* (gm/cm ³)					Reference Number	Viscosity (centipoise)					Reference Number	Specific Heat (Btu/lb-°F)					Reference Number	Thermal Conductivity (Btu/hr-ft-°F)					Reference Number	Flash Point (°F)	Flame Point (°F)	Auto- Ignition (°F)
		400°F	500°F	600°F	700°F	800°F		400°F	500°F	600°F	700°F	800°F		400°F	500°F	600°F	700°F	800°F		400°F	500°F	600°F	700°F	800°F				
Santowax OMP	0	0.964	0.917	0.870	0.823	0.776	6, 8	0.85	0.53	0.36	0.27	0.20	6, 8	0.501	0.534	0.566	0.599	0.631	9	0.0728	0.0695	0.0659	0.0623	0.0588	6, 2, 22	375	460	>1000
	10	0.976	0.930	0.885	0.839	0.793		1.007	0.63	0.42	0.30	0.22																
	20	0.989	0.944	0.900	0.855	0.810		1.32	0.77	0.50	0.35	0.26																
	30	1.009	0.958	0.915	0.871	0.828		1.62	0.94	0.61	0.42	0.31								0.077	0.0745	0.072	0.0695	0.067				
Santowax OM	0	0.950	0.904	0.859	0.813	0.765	8	0.85	0.47	0.31	0.23	0.17	7	0.492	0.531	0.568	0.604	0.640	20	0.071	0.069	0.066	0.064		21	342	370	1072
	10	0.963	0.917	0.873	0.828	0.779		1.04	0.59	0.39	0.29	0.23																
	20	0.980	0.934	0.889	0.844	0.796		1.23	0.70	0.48	0.35	0.28																
	30	0.999	0.952	0.907	0.863	0.815		1.42	0.82	0.56	0.42	0.34																
OM-2 (comparable to Santowax WR)	0	0.952	0.907	0.860	0.813	0.756	1	0.85	0.52	0.36	0.26	0.20	1, 2	0.499	0.535	0.572	0.607	0.642	2	0.071	0.069	0.066	0.063	0.059	2	380	420	1075
	10	0.961	0.917	0.970	0.821	0.770		1.00	0.62	0.43	0.30	0.23																
	20	0.970	0.926	0.881	0.834	0.782		1.25	0.74	0.48	0.34	0.26																
	30	0.980	0.938	0.893	0.846	0.794		1.50	0.88	0.57	0.39	0.29																
DOM	0	0.922	0.872	0.820	0.766	0.708	7	0.64	0.40	0.28	0.20	0.16	7														1110	
	10	0.936	0.888	0.838	0.785	0.730		0.82	0.48	0.32	0.24	0.20																
	20	0.950	0.903	0.856	0.805	0.751		1.06	0.60	0.38	0.28	0.25																
	30	0.965	0.920	0.874	0.826	0.773		1.45	0.80	0.52	0.37	0.30																
HOM	0	0.912	0.865	0.816	0.761	0.704	7	0.75	0.47	0.32	0.24	0.18	7												350	370	880	
	10	0.927	0.882	0.836	0.784	0.729		1.00	0.60	0.40	0.28	0.22																
	20	0.942	0.899	0.856	0.806	0.754		1.25	0.74	0.49	0.35	0.26																
	30	0.957	0.917	0.876	0.830	0.779		1.54	0.88	0.58	0.40	0.30																
HB-40	0	0.866	0.828	0.788	0.737	0.682	7	0.82	0.53	0.36	0.26	0.16	7	0.45	0.49	0.53	0.57		10	0.065	0.065	0.065	0.065		10	367	396	706
	10	0.877	0.840	0.799	0.750	0.694		0.87	0.60	0.41	0.29	0.20																
	20	0.888	0.850	0.810	0.762	0.711		0.98	0.68	0.46	0.32	0.22																
	30	0.900	0.861	0.821	0.775	0.726		1.21	0.80	0.56	0.38	0.26																
Isopropyl- biphenyl	0	0.848	0.802	0.752	0.698	0.644	8	0.46	0.31	0.22	0.16	0.12	8	0.557	0.604	0.649	0.697	0.742	11	0.060	0.056	0.051	0.047		12	315	340	~850
	10	0.860	0.814	0.766	0.713	0.664		0.61	0.41	0.29	0.22	0.17																
	20	0.874	0.829	0.782	0.732	0.686		0.76	0.52	0.37	0.27	0.21																
	30	0.893	0.848	0.802	0.755	0.710		0.92	0.63	0.45	0.34	0.24																
Biphenyl	0	0.889	0.838	0.786	0.726	0.656	8	0.33	0.22	0.15	0.11	—	8	0.512	0.558	0.603	0.648	0.693	10	0.070	0.066	0.062	0.058	0.054	24	235	255	>1000
	10	0.904	0.856	0.804	0.748	0.696		0.41	0.29	0.20	0.16																	
	20	0.920	0.874	0.822	0.771	0.718		0.57	0.38	0.28	0.21																	
	30	0.936	0.892	0.843	0.794	0.744		0.84	0.52	0.37	0.28																	
Santowax WR	DP†						3	Viscosity of WR‡					3													380	420	1075
	0	0.972	0.924	0.877	0.832	0.780		0.78	0.49	0.33	0.23	0.17																
	10	0.974	0.926	0.878	0.834	0.785		0.89	0.54	0.36	0.26	0.19																
	20	0.976	0.930	0.883	0.839	0.792		1.04	0.62	0.42	0.30	0.23																
	30	0.982	0.937	0.890	0.848	0.802		1.22	0.74	0.50	0.36	0.27																

*Smoothed data interpolated from reference sources shown
†Defined as 100 - wt % o-, m-, and p-terphenyl
‡At an irradiation temperature of 750°F

Table 2. Summary of Physical
Properties Data for Candi-
date HWO CR Coolants*

Figure 1. Viscosity of Irradiated Santowax OMP

Figure 2. Viscosity of Irradiated Santowax OM

Figure 3. Viscosity of Irradiated OM-2

Figure 4. Viscosity of Irradiated Santowax WR

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Figure 5. Viscosity of Irradiated Santowax DOM

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Figure 6. Viscosity of Irradiated Santowax HOM

Figure 7. Viscosity of Irradiated HB-40

Figure 8. Viscosity of Irradiated Isopropylbiphenyl

Figure 9. Viscosity of Irradiated Biphenyl

Figure 10. Comparison of Viscosities of Irradiated Coolants at 550°F

Figure 11. Comparison of Viscosities of Irradiated Coolants at 650°F

Figure 12. Comparison of Viscosities of Irradiated Coolants at 750°F

Figure 13. Effect of Irradiation Temperature, HB Content, and Coolant Molecular Weight on Viscosity of Santowax WR

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7680-25604

comparable DP levels (28 to 31 wt %), the viscosity of irradiated Santowax WR decreased significantly as the irradiation temperature was increased from 700 to 800°F. The viscosity of Santowax WR containing 17% DP and irradiated at 800°F was lower than the unirradiated coolant at comparable temperatures, despite the fact that the irradiated coolant had a higher number average molecular weight. The Euratom data⁽¹⁾ as well as the Santowax OMP data⁽⁶⁾ (610 and 750°F irradiations) also indicate that viscosity depends on the "history" of the sample, i. e., irradiation temperature and dose rate. The density and viscosity data for DOM, HOM, and HB-40 shown in Table 2 were obtained using coolant from an in-pile loop irradiation in which the temperature varied from 490 to 610°F.⁽⁷⁾ Density and viscosity data for isopropylbiphenyl and biphenyl were obtained from in-pile loop experiments conducted at the MTR,⁽⁸⁾ with circulating bulk fluid temperatures of 500 to 700°F. Since the HWOCR concept envisions higher core bulk coolant and core exit temperatures than have been considered heretofore, the dependence of coolant viscosity on sample history should be established experimentally for the most promising HWOCR coolant candidates.

It should be noted that many viscosity measurements on irradiated coolant may be in error because of oxidation of the sample before measurement. Samples for viscosity measurement should be taken in a bomb rather than an open container. It would be still better to make the measurement in-line. The effect of coolant purification methods on viscosity should also be investigated further. A 7% decrease in viscosity has been observed in the U-3 loop as a result of treatment with Attapulugus clay. This decrease may be due to the removal of high molecular weight components.

Specific heat data for unirradiated Santowax OMP and OM, and for OM-2 are strikingly similar, and are not expected to show any significant changes upon irradiation to 30% HB.^(6,9) Specific heat data for Santowax DOM and HOM are unavailable, but should not vary significantly from the other OM series of coolants. Recent specific heat data for HB-40 published by Monsanto show somewhat lower values than for the OM series of coolants.⁽¹⁰⁾ Isopropylbiphenyl specific heat data reported some time ago⁽¹¹⁾ have been confirmed by more recent experimental data⁽¹²⁾ and show values which are higher than any of the other coolants considered here; biphenyl specific heat values recently reported⁽¹¹⁾ by Monsanto are also higher than the OM series of coolants.

Thermal conductivity data for Santowax OMP and OM, and for OM-2 again show striking similarities. The increase in thermal conductivity with HB content shown by Santowax OMP is also to be expected of OM and OM-2. Recently reported data for isopropylbiphenyl⁽¹²⁾ show thermal conductivities 17 and 25% lower than those of terphenyl coolants at temperatures of 400 and 700°F, respectively. At comparable temperatures, the density of isopropylbiphenyl is 11 to 14% lower and its specific heat is 10 to 14% higher than those of Santowax OMP and OM and of OM-2.

Methods are available for calculating thermal conductivity of a coolant.^(9,12) The relationship between thermal conductivity and density for isopropylbiphenyl for example, has been found⁽¹²⁾ to be well described by the Vargaftik equation:

$$\lambda = B\rho^{4/3} , \quad \dots(1)$$

where

B = constant,

ρ = density (kg/m^3), and

λ = thermal conductivity ($\text{W}/\text{m}^\circ\text{C}$).

The value of the coefficient B from the experimental data is 1.29×10^{-5} .

Equation 1 is shown to be appropriate by comparison with calculated results. According to Vargaftik's formula:

$$B = AC_p M^{-1/3} = \text{constant (for a particular material)} \quad , \quad \dots (2)$$

where

A = a coefficient which is independent of the type of liquid but is a function of temperature,

C_p = heat capacity at constant pressure, and

M = molecular weight.

Based on experimental data for several substances over the temperature range 30 to 140°C , Vargaftik established that the product AC_p is constant and independent of temperature; using this result, the calculated value of B for monoisopropylbiphenyl using Equation 2, at a reference temperature of 30°C and $A = 4.28 \times 10^{-3}$, is 1.27×10^{-5} , in good agreement with the experimental value determined using Equation 1.

For unirradiated Santowax OMP at 400 and 700°F , the calculated value of B using Equation 1 is 1.33×10^{-5} ; for irradiated Santowax OMP at 30% HB and 400 and 700°F , B is 1.4×10^{-5} . These values suggest that thermal conductivity of polyphenyls for which data are difficult to obtain due to time or funding considerations may be estimated by Equation 1 with a probable accuracy of ~5%, using available density data and values for B of 1.3×10^{-5} for unirradiated materials and 1.4×10^{-5} for irradiated materials. Equation 1 also provides a means for estimating thermal conductivity at any temperature from one experimental thermal conductivity value, by:

$$\frac{\lambda_1}{\lambda_2} = \left(\frac{\rho_1}{\rho_2} \right)^{4/3} \quad , \quad \dots (3)$$

where

λ_1 = known thermal conductivity at temperature t_1 ,

λ_2 = unknown thermal conductivity at temperature t_2 ,

ρ_1 = known density at temperature t_1 , and

ρ_2 = known density at temperature t_2 .

The effect of the precision of experimental physical properties data on calculated heat transfer coefficients is considered in the Appendix, using a physical property factor derived from a Dittus-Boelter type equation, and a statistical analysis of variance of a function of independent variables.

IV. VAPOR PRESSURE

The vapor pressure of a coolant is important in determining the maximum allowable heat flux, limited by the operating pressure of the system. Vapor pressure data for unirradiated potential HWOCR coolants are shown in Figure 14. The coolants apparently fall into two distinct groups: the higher vapor pressure group consists of biphenyl, isopropylbiphenyl, and Santowax DOM; the lower vapor pressure coolants include Santowax HOM, OM, HB-40, OM-2, and Santowax OMP in decreasing order of vapor pressure. Vapor pressure data for five irradiated polyphenyl coolants containing 31 to 37 wt % HB are shown in Figure 15. (Data for irradiated Santowax OMP and WR, biphenyl and isopropylbiphenyl are not available.) DOM and OM-2 have respectively the highest and lowest vapor pressure, with HOM, HB-40, and OMRE coolant fairly similar and about midway between DOM and OM-2.

It is of interest that, based on AECL data,⁽⁷⁾ at the 30% HB level and $\sim 705^{\circ}\text{F}$, the vapor pressure of irradiated DOM is virtually unchanged from that of the unirradiated material at 60 psia, while that of HOM increases by about 49% to 49 psia, and that of HB-40 increases by about 68% to 42 psia. If the effect were attributed to HB content alone, it might be concluded either that the HB in the hydrogenated coolants is considerably less thermally stable, or that the hydrogenated coolants show decidedly non-ideal behavior. However, this conclusion would ignore the effects of differences in initial gas, water, and low and intermediate boiler content in these samples. Such differences are obviously important, and make comparisons, evaluations and calculations of irradiated coolant vapor pressures very difficult. It has been noted previously⁽¹³⁾ that, for simple mixtures of polyphenyls (e.g., Santowax OMP), Raoult's Law vapor pressure calculations are reasonably good. In the case of unirradiated Santowax OM, the agreement between experimental and calculated values is within 3% (see Table 3); for unirradiated Santowax DOM, the agreement is within 6% (see Table 4). For unirradiated Santowax HOM, however, using the assumptions shown in Table 5, the agreement is relatively poor at 18%.

It may be concluded from the preceding observations that calculated vapor pressure data alone are not always reliable, particularly in the case of

Figure 14. Vapor Pressure of Unirradiated Coolants

Figure 15. Vapor Pressure of Irradiated Coolants

hydrogenated or partially hydrogenated materials, including fractions obtained from a coolant hydrocracking unit (reclaimed coolant). The effects on vapor pressure, viscosity and melting point of coolant degassing by either gas purging or depressurization require investigation.

TABLE 3
VAPOR PRESSURE OF UNIRRADIATED SANTOWAX OM AT 750°F

Component	Content [*] (wt %)	Mole Fraction	Component [†] Vapor Pressure (psia)	Calculated Component Partial Pressure (psia)	Actual Total Pressure [*] (psia)
Biphenyl	3.3	0.048	151.9	7.3	—
Ortho-terphenyl	65.0	0.640	40.2	25.7	—
Meta-terphenyl	29.6	0.292	21.2	6.2	—
Para-terphenyl	2.1	0.020	16.6	0.3	—
Totals	100.0	1.000	—	39.5	41.5

*Corrected to 100 wt %; from AECL-1915, Reference No. 7

†From NAA-SR-5128, Reference No. 14

TABLE 4
VAPOR PRESSURE OF UNIRRADIATED SANTOWAX DOM AT 750°F

Component	Content [*] (wt %)	Mole Fraction	Component [†] Vapor Pressure (psia)	Calculated Component Partial Pressure (psia)	Actual Total Pressure [*] (psia)
Biphenyl	29.2	0.381	151.9	57.9	—
Ortho-terphenyl	51.9	0.453	40.2	18.2	—
Meta-terphenyl	18.5	0.162	21.2	3.4	—
Para-terphenyl	0.4	0.003	16.6	0.1	—
Totals	100.0	0.999	—	79.6	84

*From AECL-1915, Reference No. 7

†From NAA-SR-5128, Reference No. 14

TABLE 5
VAPOR PRESSURE OF UNIRRADIATED SANTOWAX HOM AT 750°F

Component	Content [*] (wt %)	Mole Fraction	Component [†] Vapor Pressure (psia)	Calculated Component Partial Pressure (psia)	Actual Total Pressure [*] (psia)
Biphenyl	0.2	0.002	151.9	0.3	—
Ortho-terphenyl	56.4	0.582	40.2	23.3	—
Meta-terphenyl	2.2	0.024	21.2	0.5	—
Para-terphenyl	0.1	—	—	—	—
Hydro-terphenyl [§]	41.0	0.392	37.0	14.5	—
Totals	99.9	1.000	—	38.6	47

*From AECL-1915, Reference No. 7

†From NAA-SR-5128, Reference No. 14

§Assumed to have molecular wt of 248, and same volatility range as HB-40.
Reference No. 7

V. FLAMMABILITY CHARACTERISTICS

Table 2 lists the available data on flash point, flame point and auto-ignition temperature. The flash and flame points of all the coolants except biphenyl are in the relatively narrow range of 315 to 460°F. Auto-ignition temperatures are significantly lower for hydrogenated or partially hydrogenated coolants (HB-40, HOM) and for isopropylbiphenyl. Relative fire hazard in a reactor system using large volumes of organic coolant at temperatures approaching 750°F or higher is an important consideration, particularly since spontaneous combustion of irradiated coolant (HB-40 and Santowax OM) can occur in thermal insulation soaked with these coolants,⁽¹³⁾ at temperatures of 275 and 390°C. An oily rag can initiate combustion of HB-40 vapors.⁽¹⁵⁾ Additional data are required for the irradiated coolants with lower auto-ignition temperatures. The effect of degassing of the irradiated and unirradiated coolant on auto-ignition temperature should be measured.

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VI. HEAT OF FUSION AND MELTING POINT OF PURE POLYPHENYLS

The heats of fusion of the pure polyphenyls are important parameters in calculating the eutectics of various polyphenyl mixtures. Heat of fusion data have recently become available⁽¹⁶⁾ for three of the pure materials, and are shown in Table 6.

TABLE 6
HEAT OF FUSION OF BIPHENYL,
o-, AND m-TERPHENYL

Compound	Heat of Fusion (cal/mole)
Biphenyl	4450
o-Terphenyl	4560
m-Terphenyl	6050

The heat of fusion of para-terphenyl as estimated by French and Epstein⁽¹⁷⁾ is 8050 cal/mole. If the assumption is made that the mixtures behave as ideal solutions and that the difference between the heat capacity of the pure solid and that of the liquid component is zero (which is true in many cases),⁽¹⁷⁾ the eutectic temperature of a mixture can be obtained by solving the equation⁽¹⁷⁾

$$\exp(\Delta H_1/R)(1/T_1^\circ - 1/T^*) + \exp(\Delta H_2/R)(1/T_2^\circ - 1/T^*) + \dots + \exp(\Delta H_i/R)(1/T_i^\circ - 1/T^*) = 1, \quad \dots(4)$$

and the eutectic composition may be obtained from

$$X_i^* = \exp(\Delta H_i/R)(1/T_i^\circ - 1/T^*) \quad \dots(5)$$

where

X_i^* = mole fraction of the i th component in the eutectic mixture

ΔH_i = heat of fusion (cal/mole) of the i th component,

T_i° = absolute melting point ($^\circ\text{K}$) of the i th component,

R = gas constant ($1.987 \text{ cal degree}^{-1} \text{ mole}^{-1}$),

T^* = eutectic temperature ($^{\circ}\text{K}$) of the mixture.

Equations 4 and 5, the heat of fusion data shown in Table 6, and the melting point data for the pure components may be used, for example, to calculate the eutectic temperature and composition of an ortho- and meta-terphenyl mixture. The result indicates a eutectic temperature of $\sim 45^{\circ}\text{C}$ at an ortho:meta ratio of $\sim 2.6:1$. This is in fairly good agreement with the Hanford data⁽¹⁸⁾ which show a eutectic at $\sim 40^{\circ}\text{C}$ and an ortho:meta ratio of 3:1, and with more recent AECL data⁽¹⁹⁾ which indicate an apparent eutectic at $\sim 45^{\circ}\text{C}$. In the latter case, no true eutectic was found, possibly due to the presence of traces of para-terphenyl, which gave a minimum melting mixture in the region of 80 to 85% ortho-terphenyl.⁽¹⁹⁾

The eutectic temperature of DOM (containing $<1.0 \text{ wt } \%$ para-terphenyl), similarly calculated, is $\sim 25^{\circ}\text{C}$. The melting characteristics of Santowax DOM were reported by AECL;⁽⁷⁾ the mixture was fully molten at 38°C and partly crystalline at 25°C . The reported composition of the DOM mixture used by AECL is very close to the calculated eutectic composition, as shown in Table 7.

TABLE 7
COMPARISON OF ACTUAL AND CALCULATED
EUTECTIC COMPOSITION OF
SANTOWAX DOM

Component	Mole Fraction	
	Actual	Calculated
Biphenyl	0.381	0.379
o-Terphenyl	0.453	0.440
m-Terphenyl	0.162	0.174
p-Terphenyl	0.003	0.006

While the reasons for the difference between the observed (38°C) and calculated (25°C) eutectic temperatures are not apparent at this time, one obvious possible source of error in the calculated value is inaccuracy in the heat of fusion data for the pure components. Coolant melting point data obtained in the

past using visual observation techniques — e. g., Fisher-Johns melting point apparatus — have not been very satisfactory, particularly for the more opaque irradiated materials. Experimental measurements of the heats of fusion of the pure components (including the para-terphenyl for which data are lacking) are scheduled for the near future using differential scanning calorimetry. This technique allows for direct and accurate measurement of the energy involved in a thermal transition and the temperature at which it occurs. The same apparatus will also be used to obtain melting point data for irradiated and unirradiated coolants of interest to the HWO CR program.

VII. CONCLUSIONS

Physical properties based on measurements and extrapolations of data currently available have been presented for a number of potential HWO CR coolants. Considering first those properties which affect heat transfer of the reactor coolant — density, viscosity, specific heat, and thermal conductivity — it was shown that the derivatives of Santowax R (OMP, OM, and OM-2) are very similar. Specific heat and thermal conductivity data are lacking for Santowax WR, DOM, and HOM, but they probably are not significantly different from Santowax OM. HB-40 apparently has a somewhat lower specific heat, and isopropylbiphenyl has a higher specific heat than Santowax OM and its derivatives. Biphenyl has a somewhat higher specific heat than the OM series, and has a comparable thermal conductivity. When all the coolants are compared on the basis of viscosity changes as a function of HB content at 550, 650, and 750°F, it is apparent that Santowax DOM, isopropylbiphenyl and biphenyl are in the lower viscosity bracket, with biphenyl being by far the lowest. The same coolants which show lower viscosity characteristics also show higher vapor pressure at temperatures of interest. The lack of vapor pressure data for irradiated isopropylbiphenyl and biphenyl makes absolute comparisons difficult.

Considering melting points, isopropylbiphenyl and HB-40 have the obvious advantage of being liquids at room temperature. Both of these coolant candidates (as well as HOM) have the disadvantages of lower auto-ignition temperatures of 700 to 850°F (compared to >1000°F for WR, OMP, OM, OM-2, DOM and biphenyl) and, very likely, greater pyrolytic and radiolytic degradation rates. While relative decomposition rates have not been considered here, this obviously is a very important parameter and one which can easily override the savings achieved in eliminating heat tracing of coolant lines.

VIII. RECOMMENDATIONS FOR FUTURE WORK

There are some obvious omissions in available physical properties data; measurements are required in these areas to furnish a more firm basis on which to design the HWOCR demonstration plant. The required properties data are summarized in Table 8. Current Canadian and European work may fill some of the gaps. The effect of coolant history, e. g. , irradiation temperature and dose rate, should also be evaluated for the more promising HWOCR coolant candidates. Hopefully, it will be possible to obtain test samples at various irradiation conditions, and to perform measurements at test loop sites (MIT and AECL).

TABLE 8
PHYSICAL PROPERTIES DATA REQUIRED
FOR CANDIDATE HWOCR COOLANTS

	HB-40	OM	WR	Reclaimed Coolant
Specific Heat	A	—	B	X
Viscosity*	A	A	B	X
Auto-ignition temperature*	A	A	B	X
Melting Point*	A	A	B	X
Vapor Pressure*	A	A	B	X

*As a function of HB content, irradiation temperature, and degassing.

A: Current Canadian work is expected to supply needed information.

B: European work on OM-2 may be applicable to supplement MIT work on Santowax WR.

X: More work required.

Specific heat data for hydrocracked coolants are unavailable and will require measurements; data for HB-40 and Santowax WR may become available from other sources. The uncertainties in melting point data for coolant containing appreciable amounts of HB are sufficiently great to warrant additional measurements with laboratory equipment, providing quantitative and reproducible results. Experimental vapor pressure data will be required for the coolant ultimately selected for use in the HWOCR. The experimental parameters are HB content, irradiation temperature and dose rate, dissolved gas content, and effect of degassing on vapor pressure, melting point, and viscosity. Flammability characteristics, especially auto-ignition temperature of lower melting irradiated coolant candidates at various HB levels, should be determined with and without degassing.

To reduce makeup costs, it is expected that HB will eventually be reclaimed by hydrocracking to give useful coolant. A pilot plant to study this process is to be constructed at the Piqua Nuclear Power Facility. Reclaimed coolant will differ considerably in composition from the original coolant. Specifically, it may be expected to contain considerable biphenyl and a variety of alkylated compounds.⁽¹³⁾ Few physical property measurements have been made on reclaimed coolant. Vapor pressure and viscosity measurements will be especially important. To supplement the Piqua pilot plant, bench scale hydrocracking experiments have been proposed. Physical property measurements on samples from the bench scale experiments and those from Piqua should give a good picture of the range of values to be expected in HWOCR.

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APPENDIX

The variance of a general function of variables involves the square of the partial derivative of the function with respect to each variable.²⁵ If X is calculated from some function of $x_1, x_2, x_3, \dots$, that is,

$$X = f(x_1, x_2, x_3, \dots) \quad \dots (6)$$

then

$$\sigma^2(X) = \left(\frac{\partial X}{\partial x_1}\right)^2 \sigma^2(x_1) + \left(\frac{\partial X}{\partial x_2}\right)^2 \sigma^2(x_2) + \left(\frac{\partial X}{\partial x_3}\right)^2 \sigma^2(x_3) + \dots \quad \dots (7)$$

where $\sigma^2(X)$ is the variance.

Equation 7 provides a method for calculating the total variance of a function (provided the x 's vary independently), and hence the total precision. It is also a method for determining which component of the function makes the largest contribution to the variance of the total, and therefore points the way to where the maximum improvement can be effected, and where it is fruitless to attempt an improvement in the precision. The above may be applied to an analysis of the effect of variance of coolant physical properties data on heat transfer coefficients in equations of the Dittus-Boelter type:

$$\frac{hD}{k} = A \left(\frac{Dv\rho}{\mu} \right)^B \left(\frac{C_p \mu}{k} \right)^C \quad \dots (8)$$

where

$A, B,$ and C are constants, and

h = heat transfer coefficient ($\text{Btu}\cdot\text{hr}^{-1}\cdot\text{ft}^{-2}\cdot^\circ\text{F}^{-1}$)

D = tube diameter (ft)

k = thermal conductivity of the coolant ($\text{Btu}\cdot\text{ft}^{-1}\cdot\text{hr}^{-1}\cdot^\circ\text{F}^{-1}$)

v = coolant velocity ($\text{ft}\cdot\text{hr}^{-1}$)

ρ = coolant density ($\text{lb}\cdot\text{ft}^{-3}$)

μ = coolant viscosity ($\text{lb}\cdot\text{ft}^{-1}\cdot\text{hr}^{-1}$)

C_p = specific heat at constant pressure ($\text{Btu}\cdot\text{lb}^{-1}\cdot^\circ\text{F}^{-1}$)

Since we wish to consider only the effect of variance of the physical properties data on the calculated heat transfer coefficient, Equation 8 may be rearranged so that physical properties are separated from design parameters, such that if constants B and C have values of 0.84 and 0.40, respectively (these are reasonable values but not necessarily the only recommended values), Equation 8 then becomes:

$$h = A \left(\frac{v^{0.84}}{D^{0.16}} \right) \left(\frac{k^{0.60} \rho^{0.84} C_p^{0.40}}{\mu^{0.44}} \right) \quad \dots (9)$$

The last factor in Equation 9, called the physical properties factor (F_p), and determined by physical properties of a particular coolant, has been used previously to calculate relative F_p values for various polyphenyls as coolants.²⁶ The physical property factor (F_p) and the available data on the estimated accuracies of these properties may be used to evaluate the effect of the variance of the individual physical properties on the heat transfer coefficient since it is directly proportional to F_p , so that if:

$$X = F_p = k^{0.60} \rho^{0.84} C_p^{0.40} \mu^{-0.44} \quad \dots (10)$$

and

$$k = x_1$$

$$\rho = x_2$$

$$C_p = x_3$$

$$\mu = x_4$$

then

$$\sigma^2(X) = (\bar{X})^2 \left[\left(\frac{\sigma^2(x_1)(0.60)^2}{\bar{x}_1^2} \right) + \left(\frac{\sigma^2(x_2)(0.84)^2}{\bar{x}_2^2} \right) + \left(\frac{\sigma^2(x_3)(0.40)^2}{\bar{x}_3^2} \right) + \left(\frac{\sigma^2(x_4)(0.44)^2}{\bar{x}_4^2} \right) \right] \quad \dots (11)$$

where

$$\bar{X} = X(\bar{x}_1, \bar{x}_2, \bar{x}_3, \bar{x}_4)$$

and

$\bar{x}_1, \bar{x}_2, \bar{x}_3, \bar{x}_4$ are the mean values of the independent variables.

If the reported physical properties data are assumed to be the mean values, then the only other input data required are the standard deviations for each physical property measurement. A survey of the relative accuracy of coolant physical properties data from a number of sources taken from the U.S.^{6,8,20,21}, U.K.,⁹ and Euratom^{1,2} indicates that these accuracies or errors are not uniformly defined on a statistical basis. For the purposes of simplification, and as a first approximation, the estimated accuracy (where accuracy is limited by the precision of the method) for each physical property value for a candidate HWO CR coolant (Santowax OM + 10% HB) was taken as shown in Table 9.

TABLE 9
ESTIMATED ACCURACY OF COOLANT PHYSICAL
PROPERTIES DATA

Property		Estimated Accuracy (%)	Standard Deviation
Thermal Conductivity	$k(\bar{x}_1)$	± 4	$0.04 \bar{x}_1$
Density	$\rho(\bar{x}_2)$	± 0.5	$0.005 \bar{x}_2$
Specific Heat	$C_p(\bar{x}_3)$	± 4	$0.04 \bar{x}_3$
Viscosity	$\mu(\bar{x}_4)$	± 4	$0.04 \bar{x}_4$

Note that the standard deviation of each variable is the product of the estimated accuracy times the reported or mean value (Table 2). The estimated accuracies of the first three properties are in line with AI estimates.^{8,20,21} The accuracy of μ or viscosity is assumed here to be of the same order of magnitude as k and C_p ; however, it has been reported as ± 0.03 centipoise,⁸ which has a significant effect on the calculated variance as a function of temperature, as will be shown

later. The physical properties data for Santowax OM + 10% HB were taken from Table 2. Values for thermal conductivity at 10% HB were extrapolated from the Santowax OMP data assuming a similar order of change with HB content. The results are shown in Table 10. It is to be noted that the precision in F_p (hence, its effect on the calculated heat transfer coefficient) from 500 to 800°F is constant at $\pm 6.7\%$. It should be noted also that approximately 50% of the variance comes from the x_1 factor, or k , the thermal conductivity, with the remainder almost equally divided between C_p and μ . It is obvious that little can be gained by improving the precision of the density measurements.

In the second case, the estimated accuracy of the viscosity, μ , was taken as ± 0.03 centipoise (the temperature measurement was reported as being the largest source of error).⁸ If it is assumed that ± 0.03 centipoise is the standard deviation of the viscosity measurement (and that the accuracies of the other variables are the same as shown in Table 9), then the results are as shown in Table 11. Note now that the precision varies from ± 7.2 to $\pm 12.8\%$ as the temperature increases from 500 to 800°F. Note also that above 600°F, the viscosity, μ , becomes the most significant variable.

In view of these latter results and what has been said before of the possible, and still somewhat uncertain effects of irradiation temperature, dose rate, molecular weight of the HB, oxidation and hydrocracked coolant fraction on coolant viscosity, it would appear to be more realistic at this time to use values for total variance in F_p which are on the order of $\pm 10\%$ in the temperature region of interest to HWO CR, i. e. , 700 to 750°F.

TABLE 10

VARIANCE AND PRECISION OF PHYSICAL PROPERTY FACTOR (F_p) IN DITTUS-BOELTER
TYPE HEAT TRANSFER EQUATION FOR SANTOWAX OM + 10% HB COOLANT
(Case 1)*

Property	Partial Variance	500°F	% of Total	600°F	% of Total	700°F	% of Total	800°F	% of Total	$\bar{X}$ or F_p
k	$\frac{n_1^2 \sigma^2(x_1)}{(\bar{x}_1)^2}$	5.76×10^{-4}	49.8	5.76×10^{-4}	49.8	5.76×10^{-4}	49.8	5.76×10^{-4}	49.8	4.05
ρ	$\frac{n_2^2 \sigma^2(x_2)}{\bar{x}_2^2}$	0.18×10^{-4}	1.5	0.18×10^{-4}	1.5	0.18×10^{-4}	1.5	0.18×10^{-4}	1.5	4.70
C_p	$\frac{n_3^2 \sigma^2(x_3)}{\bar{x}_3^2}$	2.56×10^{-4}	22.0	2.56×10^{-4}	22.0	2.56×10^{-4}	22.0	2.56×10^{-4}	22.0	5.15
μ	$\frac{n_4^2 \sigma^2(x_4)}{\bar{x}_4^2}$	3.10×10^{-4}	26.7	3.10×10^{-4}	26.7	3.10×10^{-4}	26.7	3.10×10^{-4}	26.7	5.44
Totals		11.60×10^{-4}	100.0	11.6×10^{-4}	100.0	11.6×10^{-4}	100.0	11.6×10^{-4}	100.0	—
$\sigma^2(X)$		0.0190	—	0.0256	—	0.0307	—	0.0344	—	—
σ		0.138	—	0.16	—	0.175	—	0.186	—	—
Precision at 95% confidence level ($\pm 1.96\sigma$)		± 0.270	—	± 0.314	—	± 0.346	—	± 0.365	—	—
Precision % $\left(\frac{\pm 1.96\sigma}{F_p}\right) \times 100$		± 6.7	—	± 6.7	—	± 6.7	—	± 6.7	—	—

*Assuming estimated accuracy (%) as follows:

Thermal conductivity (k) = ± 4
 Density (ρ) = ± 0.5
 Specific heat (C_p) = ± 4
 Viscosity (μ) = ± 4

TABLE 11

VARIANCE AND PRECISION OF PHYSICAL PROPERTY FACTOR (F_p) IN DITTUS-BOELTER
TYPE HEAT TRANSFER EQUATION FOR SANTOWAX OM + 10% HB COOLANT
(Case 2)*

Property	Partial Variance	500°F	% of Total	600°F	% of Total	700°F	% of Total	800°F	% of Total	$\bar{X}$ or F_p
k	$\frac{n_1^2 \sigma^2(x_1)}{\bar{x}_1^2}$	5.76×10^{-4}	42.6	5.76×10^{-4}	28.9	5.76×10^{-4}	19.7	5.76×10^{-4}	13.9	4.05
ρ	$\frac{n_2^2 \sigma^2(x_2)}{\bar{x}_2^2}$	0.18×10^{-4}	1.3	0.18×10^{-4}	0.9	0.18×10^{-4}	0.6	0.18×10^{-4}	0.4	4.70
C_p	$\frac{n_3^2 \sigma^2(x_3)}{\bar{x}_3^2}$	2.56×10^{-4}	18.9	2.56×10^{-4}	12.9	2.56×10^{-4}	8.7	2.56×10^{-4}	6.2	5.15
μ	$\frac{n_4^2 \sigma^2(x_4)}{\bar{x}_4^2}$	5.03×10^{-4}	37.2	11.4×10^{-4}	57.3	20.7×10^{-4}	71.0	32.9×10^{-4}	79.5	5.44
Totals		13.53×10^{-4}	100.0	19.9×10^{-4}	100.0	29.20×10^{-4}	100.0	41.4×10^{-4}	100.0	—
$\sigma^2(X)$		0.0222	—	0.0440	—	0.0773	—	0.1225	—	—
σ		0.149	—	0.210	—	0.278	—	0.354	—	—
Precision at 95% confidence level ($\pm 1.96\sigma$)		± 0.292	—	± 0.412	—	± 0.545	—	± 0.694	—	—
Precision % $\left(\frac{\pm 1.96\sigma}{F_p}\right) \times 100$		± 7.22	—	± 8.77	—	± 10.6	—	± 12.8	—	—

*Assuming estimated accuracy as follows:

Thermal conductivity (k) = $\pm 4\%$
 Density (ρ) = $\pm 0.5\%$
 Specific heat (C_p) = $\pm 4\%$
 Viscosity (μ) = ± 0.03 centipoise

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