

Intermediate Temperature Fluids for Heat Pipes and Loop Heat Pipes

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There are a number of applications that could use heat pipes or loop heat pipes (LHPs) in the intermediate temperature range of 450 to 750 K, including space nuclear power system radiators, fuel cells, geothermal power, waste heat recovery systems, and high temperature electronics cooling. Potential working fluids include organic fluids, elements, and halides. The paper reviews previous life tests conducted with 30 different intermediate temperature working fluids, and over 60 different working fluid/envelope combinations. Life tests have been run with three elemental working fluids: sulfur, sulfur-iodine mixtures, and mercury. Other fluids offer benefits over these three liquids in this temperature range. Life tests have been conducted with 19 different organic working fluids. As the temperature is increased, all of the organics start to decompose. Typically they generate non-condensable gas, and often the viscosity increases. The maximum operating temperature is a function of how much NCG can be tolerated, and the heat pipe operating lifetime. The highest long term life tests were run at 623 K (350°C), with short term tests at temperatures up to 653 K (380°C). Three sets of organic fluids stand out as good intermediate temperature fluids: (1) Diphenyl, Diphenyl Oxide, and Eutectic Diphenyl/Diphenyl Oxide, (2) Naphthalene, and (3) Toluene. While fluorinating organic compounds is believed to make them more stable, this has not yet been demonstrated during heat pipe life tests. Ongoing life tests suggest that the halides may be suitable for temperatures up to 673 K (400°C). However, property data for the halides is incomplete.

I. Introduction

There are a number of different applications that could use heat pipes or loop heat pipes (LHPs) in the intermediate temperature range of 450 to 750 K, including space nuclear power system radiators, fuel cells, geothermal power, waste heat recovery systems, and high temperature electronics cooling. The intermediate temperature region is generally defined as the temperature range between 450 and 750 K. At temperatures above 700-725 K, alkali metal (cesium) heat pipes start to become effective. Below about 725 K, the vapor density for cesium is so low that the vapor sonic velocity limits the heat transfer. Historically, water was used at temperatures up to about 425 K. More recently, it has been shown that water can be used with titanium or Monel envelopes at temperatures up to 550 K (Anderson, Dussinger, Bonner, and Sarraf, 2006, Anderson, Dussinger, and Sarraf, 2006).

At present, there is no commonly accepted working fluid over the entire intermediate temperature range. Potential working fluids include elemental working fluids such as sulfur, organic compounds, and halides. This paper reviews many of the heat pipe life tests conducted over the past 40 years, and then recommends suitable working fluid/envelope combinations. The fluids tested to date are shown in Table 1, along with melting point, normal boiling point, and critical point information (where known). Water and cesium are included in the table, since they bound the intermediate temperature range.

II. Intermediate Temperature Fluid Life Tests

A. Life Tests

Life tests are required to verify that the heat pipe envelope, wick, and working fluid are compatible for the potentially long operating life of a heat pipe. Potential problems when the system is not compatible include:

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Table 1. Intermediate Temperature Fluids.

Fluid		Melting Point, K	Normal Boiling Temp., K	Critical Temp., K	Critical Pressure, atm
Elements/Water					
Water	H ₂ O	273	373	647	218.3
Iodine	I ₂	387	458	785	116
Mercury	Hg	234.3	630	1765	1510
Sulfur	S	386	718	1314	
Sulfur/10% Iodine	S/10%I	390	—	—	—
Cesium	Cs	302	941	2045	114.7
Halides					
Tin Tetrachloride	SnCl ₄	240	388	592	37
Titanium Tetrachloride	TiCl ₄	243	409.6	638	46
Gallium Trichloride	GaCl ₃	351	474	694	
Titanium Tetrabromide	TiBr ₄	312	506	795.7	
Aluminum Tribromide	AlBr ₃	370	528	763	85.5
Antimony Tribromide	SbBr ₃	370	553	1178	55
Antimony Trichloride	SbCl ₃	346	556	794	
Organic Fluids					
Toluene	C ₆ H ₅ CH ₃	178	384	592	40.5
N-Octane	C ₈ H ₁₈	216	399	569	24.6
Dowtherm E (ortho-dichlorobenzene)	C ₆ H ₄ Cl ₂	256	453		
Phenol	C ₆ H ₆ O	314	455	694	59
Suntech Fluorocarbon "R"			463		
Decafluorobiphenyl	C ₁₂ F ₁₀		479	640	
1-Fluoronaphthalene	C ₁₀ H ₇ F		489		
Naphthalene	C ₁₀ H ₈	353	490	748	40
Formyl-piperidine	C ₆ H ₁₁ NO		496		
Quinoline	C ₉ H ₇ N	256	511	790	57
Monochloronaphthalene	C ₁₀ H ₇ Cl	270.7	523		
Octafluoronaphthalene	C ₁₀ F ₈				
Diphenyl (biphenyl, phenylbenzene)	C ₁₂ H ₁₀	343	527	780	34.5
Dowtherm A (Diphenyl/Diphenyl Oxide)	C ₁₂ H ₁₀ , C ₁₂ H ₁₀ O	285	530	770	31
Diphenyl Oxide (Phenyl Ether)	C ₁₂ H ₁₀ O	300	532	767	31
Diphenylmethane	C ₁₃ H ₁₂	299	535	770	26.7
Pyrene	C ₁₆ H ₁₀	424			
O-Terphenyl	C ₁₈ H ₁₄	331	605	857	30
M-Terphenyl	C ₁₈ H ₁₄	359	652	883	25
P-Terphenyl	C ₁₈ H ₁₄	488	662	908	30
OM (Binary mixture of ortho- and meta-terphenyl)	C ₁₈ H ₁₄				
OMD (Ternary eutectic mixture of diphenyl, ortho- and meta-terphenyl)	C ₁₈ H ₁₄				
perfluoro-1,3,5-triphenylbenzene	C ₂₄ F ₁₈				

1. Fluid decomposition
2. Corrosion, blocking the wick or developing leaks in the heat pipe envelope
3. Non-condensable gas generation, caused by either of the problems above
4. Material transport – dissolving components of the wall/wick in the condenser, and re-depositing the material in the evaporator.

To conduct a life test, an envelope material, a wick material, and a working fluid are chosen. A simple heat pipe is then fabricated using the chosen material, and tested at the desired operating temperature. Temperatures are monitored to detect the formation of non-condensable gas. Testing generally continues until the heat pipe either fails, or the duration of the life test is complete. In either case, the heat pipe is then sectioned and examined for possible incompatibilities.

Figure 1 shows a schematic of a typical life test heat pipe set up in a heater block. The life tests are gravity aided, and cooled by natural convection. The life test pipes are instrumented with three thermocouples. One thermocouple is located just above the heater block, while the other two are located in the heat pipe condenser. During operation, the temperature difference between the evaporator and condenser are monitored to detect non-condensable gas (NCG). Any NCG is swept by the working fluid to the end of the condenser, where it forms a cold end.

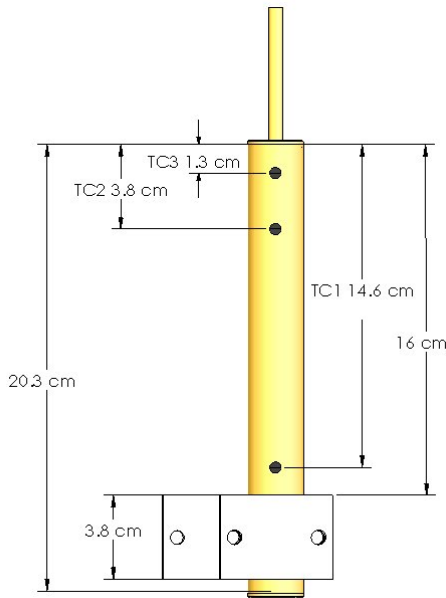


Figure 1. Typical Life Test Heat Pipe and Heater Block.

III. Elements

B. Sulfur, Sulfur/Iodine, and Iodine

Pure sulfur has design problems in the intermediate temperature range because of its high liquid viscosity, although it may be useful at higher temperatures. Sulfur has a unique temperature dependent polymerization property at 470 K, which increases its liquid viscosity peak to approximately 100 Pa-s. This is about three orders of magnitude higher than the maximum level for effective heat pipe operation. Viscosity is important in gravity aided thermosyphons, since it controls how easily the fluid can drain back to the evaporator. The addition of a small percentage of iodine reduces the viscosity to a level that may be acceptable for reasonable heat pipe operation (Polasek and Stulc, 1976, Timrot et al., 1981).

Ernst (2006) tested pure sulfur heat pipes in the 1970s. A short duration sulfur heat pipe life test of several hundred hours at 873 K (600°C) showed no gross incompatibility with 3003 aluminum. Since the operating temperature of 875 K to 1100 K is too high for aluminum, carbon steel envelopes and end caps were “Alonized” in attempt to aluminum coat the surfaces that are exposed to sulfur. Heat pipes made from the Alonized material failed

after several hundred hours because of contamination in the weld zones. Unfortunately this program came to an abrupt halt as the funding dried up.

Lundberg, Merrigan, Prenger, and Dunwoody (1980) noted that “The use of sulfur as a heat pipe working fluid is also limited by its extreme corrosiveness towards most metallic container materials.” Since molten sulfur does not attack pure fused quartz, they fabricated two wickless heat pipes with fused quartz envelopes. One heat pipe had sulfur, the other heat pipe had sulfur with 0.5% Iodine. These heat pipes were operated for a short period of time, while visually examining the behavior of the sulfur during start-up, operation, and shutdown. They state that they were planning a heat pipe life test with sulfur in a 316 SS envelope, but the results do not seem to be publicly available.

Table 2. Sulfur/10% Iodine heat pipe life test data (Anderson, Rosenfeld, Angirasa, and Mi, 2004).

Material	Operating Temperature, K	Operating Power, W	Duration, Hours	Condition
Aluminum 5052	623 (350°C)	30	1,028	Grain boundary penetration
Ti-6Al-4V	623 (350°C)	30	1,000	150 K ΔT
Titanium CP-2	523 (250°C)	20	24	Failed
304 SS	623 (350°C)	30	1,008	No sign of failure
Niobium-1% Zr	623 (350°C)	40	950	Failed

A previous series of life tests with sulfur/iodine are reported in Anderson, Rosenfeld, Angirasa, and Mi (2004); see Table 2. Testing was generally at 623 K, and lasted for roughly 1,000 hours. As shown in Table 2, 304 stainless steel was compatible, while Aluminum 5052, Ti-6Al-4V, CP-2 Titanium, and Niobium-1% Zr were not. We are not aware of any life tests with iodine.

C. Mercury

There are several problems with mercury as a wetting fluid including:

- Toxicity
- Difficulty in achieving good wetting of the wick and wall material without extensive corrosion
- High density, which translates into increased mass.

Los Alamos National Laboratory has conducted a life test with mercury on a 12-inch-long, 0.75-inch-OD SS heat pipe. (LASL 1968c, LASL 1969, Deverall, 1970, Reid, Merrigan, and Sena, 1991) The envelope was 347 SS with three wraps of 100-mesh 304 SS screen for the wick. The system operated at 603 K (330°C) for 10,000 hours. In these tests the maximum heat flux was 1.06 kW over a 2.25-inch evaporator region. Magnesium was used as an oxygen getter to clean the surface, promoting wetting. Titanium was used as an inhibitor to reduce stainless steel corrosion.

The heat pipe in the Los Alamos test had a relatively coarse wick. It appears to be very difficult to achieve wetting in finer pore wicks. Heat pipe tests with mercury in a sintered stainless steel wick failed because the mercury did not wet the stainless steel; see Anderson, Rosenfeld, Angirasa, and Mi (2004) for details.

D. Summary, Sulfur, Sulfur-Iodine, and Mercury Life Tests

Sulfur, Sulfur-Iodine, and Mercury life tests are summarized in Table 3. Mercury is compatible with 347 SS based on a long term life tests. Sulfur is compatible with pure aluminum based on a short term life test, as is Sulfur-10% Iodine with 304 SS.

Table 3. Summary of Sulfur, Sulfur-Iodine, and Mercury Life Tests.

	Compatible	Incompatible
Sulfur/Aluminum	Short Term 873 K (600°C)/~200 hrs./3003 Al (Ernst.)	
Sulfur – 10% I/SS	Short Term 623 (350°C)/1,008 hrs./304 SS (Anderson)	
Sulfur – 10% I /Al		623 (350°C)/1,028 hrs./5052 Al (Anderson)
Sulfur – 10% I /Ti		623 (350°C)/1,000 hrs./Ti-Cl-4V (Anderson) 523 (250°C)/24 hrs./CP-Ti (Anderson)
Sulfur – 10% I /Nb		623 (350°C)/950 hrs./Nb-1% Zr (Anderson)
Mercury/SS	603 (330°C)/10,000 hrs./347 SS (Los Alamos)	

IV. Organic Fluids

Life tests have been conducted with 19 different organic working fluids. Potential problems with the organic working fluids include the possibility of polymerization and/or dissociation. Polymerized fluids generally undergo an increase in liquid viscosity which, in a heat pipe, will decrease the circulation of the working fluid and therefore its heat transport capacity. Disassociation normally generates non-condensable gases (NCG), which over time will build up in the heat pipe condenser. The presence of NCG reduces the effective length of the heat pipe condenser and therefore the area available for heat radiation. This will either cause the temperature to rise at a given power level, or the power level to be decreased at a given temperature.

Typically, organic fluids develop problems more quickly as the temperature is increased. The maximum operating temperature for an organic fluid depends both on the operating temperature, and how long the heat pipe needs to operate. Figure 1 shows NCG gas generation in two pairs of 304 stainless steel (SS) heat pipes with Dowtherm A working fluid. Heat pipes operating at 723 K (450°C) gassed up in ~180 hours, while pipes operating at 673 K (400°C) took roughly 1,500 hours for NCG gas generation to start affecting their behavior.

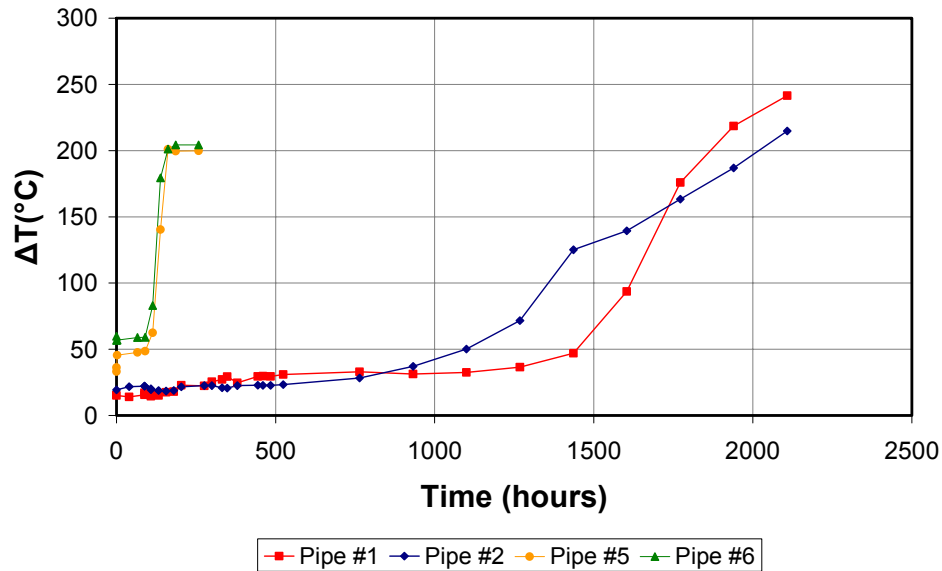
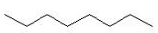
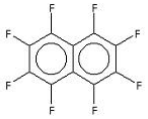
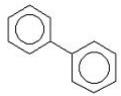
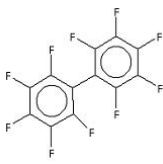
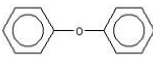
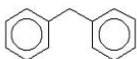


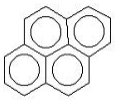
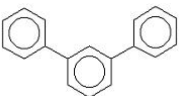
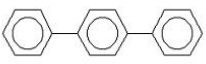
Figure 2. Working fluid decomposition and non-condensable gas generation is a strong function of temperature. ΔT between the evaporator and condenser as a function of time, Dowtherm A/304 SS life tests (Anderson et al., 2007). Pipes 5 and 6 were operating at 723 K (450°C), while pipes 1 and 2 were operating at 673 K (400°C).

Table 4 shows the organic fluids (some with fluorine) tested to date. Most of the organic fluids suitable for intermediate temperature results are ring compounds. The reason for this was discussed by Saaski and Owzarski

(1977) who pointed out that these types of compounds should be more stable than the long chain hydrocarbons. Saaski and Owzarski also pointed out replacing some (or all) of the hydrogen atoms with fluorine may make the compound more stable.

Table 4. Organic Fluids Tested in the Intermediate Temperature Range.

Toluene	$C_6H_5CH_3$	
N-Octane	C_8H_{18}	
Dowtherm E (ortho-dichlorobenzene)	$C_6H_4Cl_2$	
Phenol	C_6H_6O	
1-Fluoronaphthalene	$C_{10}H_7F$	
Naphthalene	$C_{10}H_8$	
Formyl-piperidine	$C_6H_{11}NO$	
Quinoline	C_9H_7N	
Monochloronaphthalene	$C_{10}H_7Cl$	
Octafluoronaphthalene	$C_{10}F_8$	
Diphenyl (biphenyl, phenylbenzene)	$C_{12}H_{10}$	
Decafluorobiphenyl	$C_{12}F_{10}$	
Diphenyl Oxide (Phenyl Ether)	$C_{12}H_{10}O$	
Diphenylmethane	$C_{13}H_{12}$	

Pyrene	$C_{16}H_{10}$	
O-Terphenyl	$C_{18}H_{14}$	
M-Terphenyl	$C_{18}H_{14}$	
P-Terphenyl	$C_{18}H_{14}$	
Perfluoro-1,3,5-triphenylbenzene	$C_{24}F_{18}$	

E. Life Test Series with Organic Fluids

Table 5. Life tests by Kenney and Feldman (1978).

Material	Working Fluid	Evap. Temp	Hours	Comments
304 SS	Diphenyl	748 K (475°C)	72	Failed – NCG
304 SS	Diphenyl	748 K (475°C)	72	Failed – NCG
304 SS	Diphenyl	738 K (465°C)	100	Failed – NCG
304 SS	Diphenyl	695 K (422°C)	366	Failed – NCG
304 SS	Diphenyl	673 K (400°C)	1200	O.K.
304 SS	Diphenyl	548 K (275°C)	6174	O.K.
Carbon Steel	Diphenyl	598 K (325°C)	4040	Failed – NCG
Carbon Steel	Diphenyl	498 K (225°C)	6174	O.K. 4648 hours at 320°C, then reduced to 225°C
Black Iron	Diphenyl	523 K (250°C)	7158	O.K.
Black Iron	Diphenyl	523 K (250°C)	7158	O.K.
304 SS	Dowtherm A	673 K (400°C)	1200	O.K.
304 SS	Dowtherm A	664 K (391°C)	1200	O.K.
304 SS	Dowtherm A	541 K (268°C)	24533	O.K.
304 SS	Dowtherm A	533 K (260°C)	24533	O.K.
304 SS	Dowtherm A	526 K (253°C)	24533	O.K.
Carbon Steel	Dowtherm A	523 K (250°C)	8382	O.K.
Carbon Steel	Dowtherm A	513 K (240°C)	8382	O.K.
304 SS	Diphenylmethane	748 K (475°C)	<24	Failed – NCG
304 SS	Diphenylmethane	703 K (430°C)	<24	Failed – NCG
304 SS	Formyl-piperidine	553 K (280°C)	<15	Failed
304 SS	Pyrene	573 K (300°C)	<24	Failed
304 SS	P-Terphenyl	723 K (450°C)	<96	Failed

Results for life tests with a number of different organic fluids tests are discussed in this subsection. Individual life tests are discussed below. Kenney and Feldman (1978) conducted a series of life tests with organic working fluids and 304 SS, carbon steel, and black iron heat pipes; see Table 5. All of the life tests conducted at temperatures above 673 K (400°C) failed due to non-condensable gas generation. Short term tests (~1,000 hours)

Table 6. Life Test Data for Intermediate Temperature Fluids [Saaski and Hartl, 1980].

Working Fluid	Boiling Point, K	Envelope	Time, hours	Operating Temp., K	$\Delta T_{\text{Evap. Adiab.}}$, K	$\Delta T_{\text{Adiab/Cond.}}$, K	Comments
Carbon Disulphide	320	Al 6061	28,540	335 K (62°C)	0.8	1.1	Small increase in ΔT 's
Carbon Disulphide	320	A-178 Steel	28,540	331 K (58°C)	0.4	0.1	Stable Operation
Toluene	383	Al 6061	23,130	410 K (137°C)	3.1	2.6	Stable Operation
Toluene	383	A-178 Steel	700	392 K (119°C)	1.6	4.1	Small Heat Pipe Leak - Failed
SnCl ₄	387	Al 6061	----	432 K (159°C)			Incompatible
SnCl ₄	387	A-178 Steel	27,750	429 K (156°C)	4	9.7	Stable Operation
TiCl ₄	409	Al 6061	2,500	438 K (165°C)	6	7.2	Sudden Burnout/Wick Corrosion
TiCl ₄	409	A-178 Steel	28,540	432 K (159°C)	4.1	1.7	Stable Operation
Suntech Fluorocarbon "R"	463	Al 6061	13,380	482 K (209°C)	11	15.6	Gradual Increase in NCG
Octafluoronaphthalene		Al 6061	13,400	488 K (215°C)	4.3	3.4	Stable Operation
Octafluoronaphthalene		A-178 Steel	13,400	482 K (209°C)	4.5	90	Stable Gas Leg
1-Fluoronaphthalene	489	Al 6061	13,380	493 K (220°C)	5.8	36	Gradual Increase in NCG
1-Fluoronaphthalene		A-178 Steel	4,790	489 K (216°C)	5	57	Stable Operation
1-Fluoronaphthalene		A-178 Steel	26,370	530 K (257°C)	7.4	4.3	Stable Operation
Naphthalene	491	Al 6061	27,750	488 K (215°C)	12.5	4.3	Stable Operation
Naphthalene	491	A-178 Steel	6,430	490 K (217°C)	3	3.4	Burn-out on re-start
Monochloronaphthalene	523	A-178 Steel	642	539 to 579 K (266 to 306°C)			Incompatible
Diphenyl	528	Al 6061	8,000	514 K (241°C)	24.1	--	Wick movement, evaporator failed
Diphenyl	528	A-178 Steel	24,400	526 K (253°C)	4.4	102	Stabilized Gas Leg
SbCl ₃	556	Al 6061	----	500 K (227°C)			Incompatible
SbCl ₃	556	A-178 Steel	5,000	476 K (203°C)	6.3 to 169	62	Incompatible - High $\Delta T_{\text{Evap. Adiab.}}$
O-Terphenyl	605	Al 6061	672	570 to 590 K (297 to 317°C)	7.8	67.4	Rapid Increase in NCG
O-Terphenyl	605	A-178 Steel	27,750	545 K (272°C)	10	51.5	Stabilized Gas Leg

Table 7. Organic Fluid Life Tests (Groll, 1989).

Test Duration	Working Fluids	Results		
		Compatible	Fairly Compatible	Incompatible
4.5 to 5 Years	N-Octane	St35 [†] (503 K 230°C)	321 [‡] (473, 523 K)(200, 250 °C)	
4.5 to 5 Years	Dowtherm A [§]		St35 (543 K 270°C)	St35 (573 K 300°C)
4.5 to 5 Years	Dowtherm A		321 (573 K 300°C)	321 (623 K 350°C)
4.5 to 5 Years	Dowtherm E ^{**}	St35 (493 K 220°C)		
3 Years	Toluene	St35 (523 K 250°C)		
3 Years	Toluene	13CrMo44 ^{††} (523 K 250°C)		
3 Years	Toluene	316L ^{‡‡} (553 K 280°C)		
3 Years	Naphthalene	St35 (543 K 270°C)	13CrMo44 (543 K 270°C)	
2 Years	Water	Ti 99.4 (473 K 200°C)		
2 Years	Water	CuNi10Fe ^{§§} (473 K 200°C)		
1 Year	Dowtherm A	Ti 99.4 ^{***} (543 K 270°C)		CuNi10Fe (523 K 250°C)
1 Year	Diphenyl	13CrMo44 (523 K 250°C)		13CrMo44 (673 K 400°C)
1 Year	Diphenyl ^{†††}	316L (543 K 270°C)		316L (673 K 400°C)
1 Year	OM ^{†††}			13CrMo44 (593, 673 K)(320, 400°C)
1 Year	OM			316L (623, 673 K))(350, 400°C)
1 Year	OMD ^{§§§}			13CrMo44 (623, 673 K)(350, 400°C)
1 Year	OMD			316L (620, 670 K)(350, 400°C)
1 Year	Toluene	Ti 99.4 (523 K 250°C)		
1 Year	Toluene	CuNi10Fe (553 K 280°C)		
1 Year	Naphthalene	Ti 99.4 (593 K 320°C)	CuNi10Fe (593 K 320°C)	
1 Year	Naphthalene	316L (593 K 320°C)		

[†] ST35 is a mild steel

[‡] 321 is a stainless steel resistant to acids, Groll refers to as X10CrNiTi189

[§] Dowtherm A is a eutectic mixture of diphenyl (C₁₂H₁₀) and diphenyl oxide (C₁₂H₁₀O). Also sold as Diphyl and Therminol.

^{**} Dowtherm E is a heat transfer fluid, ortho-dichlorobenzene (C₆H₄Cl₂), which is also sold as Diphyl-O

^{††} 13CrMo44 is a 1% Cr-1/2% Molybdenum Steel

^{‡‡} 316L stainless steel, Groll refers to as X2CrNiMo1812

^{§§} CuNi10Fe is a Copper Nickel Alloy, resistant to corrosion in seawater

^{***} Ti 99.4 is similar to CP-Ti, Grade 1

^{†††} Diphenyl, also known as biphenyl and phenylbenzene, (C₁₂H₁₀)

^{†††} OM, Binary mixture of ortho- and meta-terphenyl

^{§§§} OMD, Ternary eutectic mixture of diphenyl, ortho- and meta-terphenyl

with Diphenyl/304 SS and Dowtherm A/304 SS were fine. All of the long term tests that did not produce gas were operating at temperatures of 541 K (268°C) and below.

Saaski et al. (1977a, 1977b, 1980) ran a series of long duration life tests on organic fluids; see Table 6. They also looked at halides, as discussed below. Fluids were tested in aluminum 6061 and mild steel envelopes, for periods of up to 3 years. 6061 aluminum was chosen because it is commonly used in grooved aluminum heat pipes for spacecraft applications. The aluminum heat pipes had a single wrap of 100-mesh Al-1100 screen. The mild steel envelopes had a single wrap of 200-mesh 304 stainless steel screen. As shown in Table 6, most of the fluids were tested slightly above their normal boiling point. The difference between the evaporator/adiabatic, and adiabatic/condenser thermocouples was monitored. A large difference in the evaporator/adiabatic thermocouples generally indicated problems with clogging of the evaporator wick. A large difference between the adiabatic/condenser thermocouples indicated non-condensable gas generation.

Groll et al. (Groll, Brost, Heine, and Spendel, 1982, Groll, Brost, and Roesler, 1987, Groll, 1989, Heine, Groll, and Brost, 1984) tested organic fluids (and water) for periods of up to 5 years. The results are summarized in Table 7. Envelope materials included a mild steel, several stainless steels, CP-Ti, and a copper-10%nickel alloy. The difference between the evaporator and the condenser thermocouples were monitored, and used to determine compatibility. A difference of less than 10 K was Compatible, of less than 15 K was “Fairly Compatible”, and of greater than 15 K was Incompatible.

Gryzll and co-workers (Gryzll, 1991; Gryzll, Back, Ramos, and Samad, 1994; Gryzll, Back, Ramos, and Samad, 1995) conducted life tests on a series of organic fluids. In addition, they measured the density, viscosity, and surface tension of some of the potential working fluids (Gryzll, Ramos, and Back, 1996). Gryzll (1991) conducted short term (~50 hour) corrosion tests with water and diphenyl and the following coupons: 316 SS, 6061-T6 Al, Monel 400, Nickel 200, and titanium. He concluded that all of the materials were probably suitable for heat pipes with water or diphenyl. He reviewed earlier work that compared the thermal stability of diphenyl with diphenyl oxide (the other component in Dowtherm A), and concluded the diphenyl was more stable.

Gryzll, Back, Ramos, and Samad (1994, 1995) conducted a series of life tests that are summarized in Table 8. Six fluids were initially selected: naphthalene, diphenyl, o-terphenyl, quinoline, decafluorobiphenyl, and perfluoro-1,3,5-triphenylbenzene. The two fluorocarbons were selected because their earlier work had suggested that replacing hydrogen with fluorine in these compounds tends to increase their stability. Perfluoro-1,3-5-triphenylbenzene was found to undergo severe thermal decomposition at temperatures approaching 300°C during preliminary testing in glass ampoules, and was dropped from further testing.

Four heat pipes with 316 stainless steel envelopes were fabricated for each working fluid. Each heat pipe was operated as a gravity aided thermosyphon, with a 316 stainless steel wick only in the evaporator. Two heat pipes were tested at 623 K (350°C), and two cycled every 24 hours between 598 K and 653 K (325°C and 380°C). The evaporator–condenser ΔT was monitored. At the end of the life tests, the fluids were analyzed for evidence of decomposition.

Table 8. Life Tests with 316 SS envelopes and wicks (Gryzll, Back, Ramos, and Samad, 1994). Four pipes per fluid: 2 life tested at 623 K (350°C), and two cycled every 24 hours between 598 K and 653 K (325°C and 380°C).

Fluid	Duration, hours	Comment
Diphenyl	5,520	No evidence of decomposition, constant ΔT
o-Terphenyl	5,520	Two Samples changed from white to slight amber, constant ΔT
Naphthalene	5,520	Two Samples discolored, constant ΔT
Decafluorobiphenyl	5,520	All samples changed color, slight increase in ΔT for 1 pipe
Quinoline	~2,000	Changed from clear to dark brown liquid with solid precipitates, increase in ΔT for all pipes

F. Diphenyl, Diphenyl Oxide and Eutectic Diphenyl/Diphenyl Oxide

The most commonly tested organic fluids have been diphenyl, diphenyl oxide, and a eutectic mixture of diphenyl/diphenyl oxide (Trade Names Dowtherm A, Therminol, and Diphyl). Eutectic diphenyl/diphenyl oxide is nearly an azeotrope (Basilius and Prager, 1975), so the liquid and vapor have almost the same composition. This

avoids the problems encountered with other mixtures such as NaK, where fractional distillation can occur (Anderson, 1993). Life test results for these three fluids are summarized in Table 9.

Based on heat pipe and furnace tests, Kenney and Feldman (1978) found that diphenyl rapidly decomposes at temperatures of 400°C and higher. As shown in Table 5, Kenney and Feldman (1978) found that 304 SS/Dowtherm A was compatible for short tests (1200 hours) conducted at 673 K (400°C). Long term tests at temperatures around 523 K (250°C) found that Dowtherm A was compatible with 304 SS and carbon steel, and Diphenyl was compatible with 304 SS, carbon steel, and black iron. Grzyll, Back, Ramos, and Samad (1994, 1995) found that diphenyl was stable for 5,150 hour life tests with 316 SS, with steady state tests at 623 K (350°C) and thermal cycling from 598 K to 653 K (325°C to 380°C).

Researchers at Los Alamos Scientific Laboratory (1968a, 1968b, 1970) conducted a life test with Diphenyl Oxide on a 1.25 in. O.D. 347 SS heat pipe with 3 wraps of 100 mesh 304 stainless steel as the wick. The heat pipe was operated at 573 K (300°C) for 3200 hours. At the end of the test, there was a 4 K temperature difference across the pipe. They were not sure if this was caused by gas generation, or by hydrogen permeating through the wall from water vapor dissociating on the outside of the heat pipe.

Eutectic Diphenyl/Diphenyl Oxide (Trade Names Dowtherm A, Therminol, and Diphyl) has been examined as a heat pipe working fluid by a number of researchers. Groll et al. (Groll, Brost, Heine, and Spindel, 1982, Groll, Brost, and Roesler, 1987, Groll, 1989, Heine, Groll, and Brost, 1984) found that Dowtherm A was compatible with 321 SS at 573 K (300°C), and incompatible at 623 K (350°C), where the fluid reacted with the envelope material. Similarly, diphenyl was compatible with stainless steels at 520-540 K, but not at 670 K. At 690 K, the fluid reacted in a few hours with the wall material, forming noncondensable gases, and corroding the wall material.

Table 9. Summary of Diphenyl, Diphenyl Oxide and Eutectic Diphenyl/Diphenyl Oxide Life Tests

	Compatible	Incompatible
Diphenyl/Aluminum	514 K (241°C)/8,000 hrs./6061 Al (Saaski et al.)	
Diphenyl/Mild Steel	523 K (250°C)/~9,000 hrs. (Groll et al.) 498 K (225°C)/6,174 hrs (Kenney/Feldman)	673 K (400°C)/~9,000 hrs. (Groll et al.) 598 K (325°C)/4,040 hrs. (Kenney/Feldman) 526 K (253°C)/24,400 hrs. (Saaski et al.)
Diphenyl/Black Iron	523 K (250°C)/7,158 hrs (Kenney/Feldman)	
Diphenyl/Stainless Steel	Cycle 598 K to 653 K (325°C to 380°C)/5,520 hrs./316 SS (Grzyll) 623 K (350°C)/ 5,520 hrs./316 SS (Grzyll) 548 K (275°C)/6,174 hrs/304 SS (Kenney/Feldman) 543 K (270°C)/~9,000 hrs./316L SS (Groll et al.) Short Term 673 K (400°C)/1200 hrs./304 SS (Kenney/ Feldman)	748 K (475°C)/72 hrs./304 SS (Kenney/Feldman) 738 K (465°C)/100 hrs./304 SS (Kenney/Feldman) 695 K (422°C)/366 hrs./304 SS (Kenney/Feldman) 673 K(400°C)/~9,000 hrs./316L SS (Groll et al.)
Diphenyl Oxide/ Stainless Steel	Short Term 573 K (300°C)/3200 hrs./347 SS & 304 SS (LASL)	
Dowtherm A/Mild Steel	543 K (270°C)/~40,000 hrs/ST 35 (Groll et al.) 523 K (250°C)/8383 hrs. (Kenney/Feldman)	573 K (300°C) /~40000 hrs./ST 35 (Groll et al.)
Dowtherm A/ Stainless Steel	573 K (300°C)/~40,000 hrs/321 SS (Groll et al.) 541 K (268°C)/24,500 hrs/304 SS (Kenney/Feldman) Short Term 673 K (400°C)/1,200 hrs (Kenney/Feldman) 618 K (345°C)/1,000 hrs./304 SS (Anderson et al.)	723 K (450°C)/180 hrs./304 SS (Anderson et al.) 673 K (400°C)/1,770 hrs./304 SS (Anderson et al.) 623 K (350°C)/~40,000 hrs/321 SS (Groll et al.) 473 K (200°C)/17,016 hrs. (Basilus/Prager) (slow gas generation)
Dowtherm A/Copper		473 K (200°C)/7,016 hrs/ (Basilus and Prager) slow gas generation
Dowtherm A/ Copper-Nickel		523 K (250°C)/~9,000 hrs./CuNi10Fe (Groll et al.)
Dowtherm A/Titanium	543 K(270°C)/~9,000 hrs. (Groll et al.)	406°C (680K)/~2,000 hrs (Anderson et al.)

Basiulis (Basiulis and Fuller 1971, Basilus and Prager 1975) conducted a large series of life tests, however, most were at temperatures below the intermediate temperature range. Two tests were conducted with Dowtherm A and a stainless steel envelope were run at 473 K (200°C) for 17,016 hours. One of the pipes had a SS mesh wick, while the other pipe has a copper mesh wick. They noted that non-condensable gas was generated during the tests.

Table 10. Eutectic Diphenyl/Diphenyl Oxide Tests (Anderson et al., 2007).

Envelope/Wick	Operating Temp.	Duration Hours	Comment
CP-Titanium	680 K (407°C)	2,000	High NCG
304 SS	723 K (450°C)	180	High NCG
304 SS	673 K (400°C)	1,770	High NCG
304 SS	623 K (350°C)	1,000	O.K.

Anderson et al. (2007) ran Dowtherm A life tests with 304 SS and titanium. Test results are shown in Table 10. All of the tests at 400°C and above generated gas. As shown in Figure 1, the gas generation rate increased as the temperature was increased. The 1,000-hour test with 304 SS at 623 K (350°C) has not yet shown signs of gas generation.

When using diphenyl, diphenyl oxide, or diphenyl/diphenyl oxide at temperatures over 673 K (400°C), non-condensable gas is generated in a relatively short time period; see Table 9. The exact period depends on the fluid and material, and decreases as the temperature increases. For example, Kenney and Feldman found that their diphenyl pipes took less than 72 hours to gas up at 748 K (475°C), and 366 hours to gas up at 695 K (422°C). Between 300 and 400°C, these fluids are generally suitable, for short duration tests near 400 C, and long duration tests near 300 C (For example, Groll et al. found that 321 SS was compatible for ~40,000 hours at 573 K (300°C), but not at 623 K (350°C).

G. Other Organic Fluids

Vasil'ev, Volokhov, Gigeovich, and. Rabetskii (1988) conducted life tests with naphthalene at 593 K (320°C) in thermosyphons for roughly 3,000 hours. Two envelopes were tested, titanium and Alloy 20 stainless steel. No degradation in performance was observed during the life tests. The Alloy 20 pipe was operated for brief periods at temperatures up to 653 K (380°C).

Life tests results for organic fluids (other than diphenyl and diphenyl oxide) are summarized in Table 11. Fluids have been ranked by the highest temperature for a compatible life test with any envelope material.

Table 11. Summary of Organic Fluid Life Tests Other Than Diphenyl and Diphenyl Oxide.

	Compatible	Incompatible
Naphthalene/Aluminum	488 K (215°C)/27,750 hrs./6061 Al (Saaski et al.)	
Naphthalene/Mild Steel	543 K (270°C)/~26,000 hrs./ST 35 & 13CrMo44. (Groll et al.)	
Naphthalene/Stainless Steel	Cycle 598 K to 653 K (325°C to 380°C)/5,520 hrs./316 SS (Grzyll) 623 K (350°C)/ 5,520 hrs./316 SS (Grzyll) 593 K (320°C)/~9,000 hrs/316L SS / (Groll et al.) 593 K (320°C)/Alloy 20/~3,000 hours (Vasil'ev et al.) Short Term 653 K (380°C)/Alloy 20 (Vasil'ev et al.)	
Naphthalene/Cu-Ni	593 K (320°C)/~9,000 hrs/CuNi10Fe (Groll et al.)	
Naphthalene/Titanium	593 K (320°C)/~9,000 hrs/CP-Ti (Groll et al.) 593 K (320°C)/~3,000 hours/Ti (Vasil'ev et al.)	
O-Terphenyl/Al		580 K (307°C)/672 hrs./6061 Al (Saaski et al.)
O-Terphenyl/Mild Steel	545 K (272°C)/27,750 hrs/Al-178 (Saaski et al.) stable NCG	
O-Terphenyl/SS	Cycle 598 K to 653 K (325°C to 380°C)/5,520 hrs./316 SS (Grzyll) 623 K (350°C)/ 5,520 hrs./316 SS (Grzyll)	
Decafluorobiphenyl/SS	Cycle 598 K to 653 K (325°C to 380°C)/5,520 hrs./316 SS (Grzyll) 623 K (350°C)/ 5,520 hrs./316 SS (Grzyll)	

	Compatible	Incompatible
Toluene/Aluminum	410 K (137°C)/23,120 hrs./6061 Al (Saaski et al.)	
Toluene/Mild Steel	523 K (250°C)/~26,000 hrs./ ST 35 & 13CrMo44 (Groll et al.)	
Toluene/Stainless Steel	523 K (250°C)/~26,000 hrs./316 SS (Groll et al.)	
Toluene/Copper Nickel	553 K (280°C)/9,000 hrs./CuNi10Fe (Groll et al.)	
Toluene/Titanium	523 K (250°C) 9,000 hrs. (Groll et al.)	
1-Fluoronaphthalene/Aluminum	493 K (220°C)/13,380 hrs./6061 Al Some NCG (Saaski et al.)	
1-Fluoronaphthalene/Mild Steel	530 K (257°C)/26,370 hrs./A178 (Saaski et al.)	
1-Fluoronaphthalene/Stainless Steel		530 K (257°C)/26,370 hrs./304 SS (Saaski et al.)
N-Octane/Mild Steel	503 K (230°C)/~40,000 hrs./ST 35 (Groll et al.)	
N-Octane/Stainless	523 K (250°C)/~40,000 hrs./321 SS (Groll et al.)	
Dowtherm E	493 K (220°C)/~40,000 hrs./ST 35 (Groll et al.)	
Octafluoronaphthalene/Aluminum	482 K (209°C)/13,400 hrs./6061 Al Some NCG (Saaski et al.)	
Octafluoronaphthalene/Mild Steel		488 K (215°C)/13,400 hrs./A178 Some NCG (Saaski et al.)
Quinoline/SS		Cycle 598 K to 653 K (325°C to 380°C)/5,520 hrs./ 316 SS (Grzyll) 623 K (350°C)/ 5,520 hrs./316 SS (Grzyll)
Monochloronaphthalene/Stainless Steel		560 K 287°C/642 hrs./A178 (Saaski et al.)
Formyl-piperidine/SS		553 K (280°C)/15 hrs/304 SS/ (Kenney/ Feldman)
P-Terphenyl/SS		723 K (450°C)/~96 hrs/304 SS (Kenney/Feldman)
ortho- and meta-terphenyl/Mild Steel		673 K (400°C)/~9,000 hrs./13CrMo44 (Groll) 593 K (320°C)/~9,000 hrs./13CrMo44 (Groll)
ortho- and meta-terphenyl/SS		673 K (400°C)/~9,000 hrs./316L SS (Groll) 623 K (350°C)/~9,000 hrs./316L SS (Groll)
diphenyl, ortho- and meta-terphenyl/Mild Steel		673 K (400°C)/~9,000 hrs./13CrMo44 (Groll) 623 K (350°C)/~9,000 hrs./13CrMo44 (Groll)
diphenyl, ortho- and meta-terphenyl/SS		673 K (400°C)/~9,000 hrs./316L SS (Groll) 623 K (350°C)/~9,000 hrs./316L SS (Groll)
Perfluoro-1,3,5-triphenylbenzene		Severe thermal decomposition at 573 K (300°C) Grzyll

Since all of their life tests to date have been compatible, two fluids stand out in Table 11: toluene and naphthalene. Toluene was compatible with a copper-nickel alloy at 553 K (280°C), as well as with aluminum, mild steel, stainless steel, and titanium at lower temperatures. This is probably close to the maximum useful range of toluene, since the critical point of toluene is 592 K (319°C). Water is generally a better working fluid, since it can also be used in this temperature range, and has a Merit number that is roughly 50 times higher than toluene. However, toluene has three advantages over water, which may make it a suitable choice for certain conditions. The advantages include:

- Compatibility with a larger number of envelope/wick materials
- Melting temperature of 178 K (-95°C) versus 273 K (0°C)
- Lower saturation pressure (e.g., 23.4 atm. at 550 K versus 60.4 atm. for water)

Naphthalene is compatible with stainless steel, copper-nickel, and titanium, based on long term life tests at 593 K (320°C) and above. It has also been shown to be compatible at lower temperatures with aluminum and mild steel. It was compatible with stainless for short term tests at 380°C.

While fluorinated compounds are believed to be more stable than the same compound with out fluorine, this has not been verified in life tests date. Gryzll, Back, Ramos, and Samad, (1994) found that Decafluorobiphenyl ($C_{12}F_{10}$) was less stable than Diphenyl ($C_{12}H_{10}$) under the same test conditions. Perfluoro-1,3,5-triphenylbenzene underwent severe thermal decomposition. Naphthalene was compatible with mild steel at 623 K (350°C) for 5,520 hours, while Monochloronaphthalene was found to be unsuitable after 642 hours at 560 K (287°C), and Octafluoronaphthalene had NCG gas generation at 488 K (215°C). Other stable, fluorinated life tests have been conducted at temperatures of 530 K (257°C) and below.

V. Halides

A halide is a compound of the type MX, where M may be another element or organic compound, and X may be fluorine, chlorine, bromine, iodine, or astatine. Starting with Saaski and Owarski (1977), a number of researchers have suggested that halides are potential heat pipe fluids. They are attractive because they are more stable at high temperatures than organic working fluids, and because their Merit number peaks in the intermediate temperature range. Information on halide properties can be found in Anderson, Rosenfeld, Angirasa, and Mi (2004) and Devarakonda and Anderson (2005).

Saaski and his co-workers (Saaski and Owarski, 1977; Saaski and Tower, 1977, Saaski and Hartl, 1980) life-tested the halides $SbCl_3$, $SnCl_4$, and $TiCl_4$ with aluminum 6061 and mild steel envelopes, for periods of up to 3 years. The test set-up was discussed above in the section on organic working fluids. The halides were life tested slightly above their normal boiling point. The results are shown in Table 6. All 3 halides were incompatible with aluminum. Gross corrosion of the evaporator and evaporator wick was observed with $SbCl_3$ and $SnCl_4$ in aluminum. $SnCl_4$ and $TiCl_4$ were compatible with mild steel (and stainless steel), with the life tests running roughly 3 years. The $SbCl_3$ reacted with the stainless steel wick and generated significant quantities of gas.

Locci and coworkers (Locci, Devarakonda, Copeland, and Olminsky, 2005, Tarau, Sarraf, Locci, and Anderson, 2007) conducted a series of halide life tests with $AlBr_3$, $SbBr_3$, and $TiCl_4$. CP-2 titanium was tested, along with two aluminum alloys, Al-6061 and Al-5052. All tests were conducted at 500 K. The life test results are summarized in Table 12. $AlBr_3$ was not compatible with any of the materials tested. It attacked the alloying materials in the grain boundary with the aluminum alloys, and formed TiAl products with the CP titanium. Tarau, Sarraf, Locci and Anderson (2007) did have a successful test with $TiCl_4$ and CP2-titanium.

Table 12. NASA Glenn Research Center Life Tests with Halides and Water (Locci, Devarakonda, Copeland, and Olminsky, 2005, Tarau, Sarraf, Locci, and Anderson, 2007). All Tests Conducted at 500 K (227°C).

Envelope	Al-6061 (0.8-1.2 Mg, 0.4-0.8 Si)	Al-5052 (2.2-2.8 Mg, 0.25 max Si)	CP2-Ti
Fluid			
$AlBr_3$	1,100 hrs. Intergranular Corrosion $\Delta T = 100$ K	4,290 hrs. Failed $\Delta T = 70$ K	1,100 hrs. Secondary Products – TiAl $\Delta T = 90$ K
$SbBr_3$	5,000 hrs. Wall Thickness Change $\Delta T = 90$ K	–	–
$TiCl_4$	Not Suitable (Saaski)	Probably Not Suitable	4,019 hrs. - Stable $\Delta T = -25$ K
Water	Not Suitable	Not Suitable	8,000 Hours – Ongoing

Saaski and Owzarsky (1977) proposed an electrochemical method to predict the compatibility of halide working fluids with envelope materials. Tarau, Sarraf, Locci and Anderson (2007) found that this procedure had good agreement with the halide life tests discussed above. This is discussed further in a companion paper (Anderson et al., 2007).

In the same paper, Anderson et al. (2007) used the electrochemical method to select halides and (hopefully) compatible materials. Aluminum alloys were not considered, because aluminum would react with almost all of the halides. CP-titanium was selected, along with three superalloys: Hastelloy B-3 (Ni-Mo), Hastelloy C-2000 (Ni-Cr-Mo), and Hastelloy C-22 (Ni-Cr-Mo-W). The procurement of the 3 superalloys was initially based on the great general corrosion behavior to acids or excellent stress corrosion cracking and pitting resistance reported on the alloys. The three alloys can be used to investigate the influence of ternary additions, e.g. the effect of Mo, Cr, or W

to the heat pipe environment. Weldability was another critical factor that was considered, and in general the interest of using superalloys is the much higher specific strength to compete against the lower density Ti- or Al-alloys.

The titanium pipes had a 50 x 50 mesh titanium screen wick, and the C-22 pipes had an 80 x 80 mesh C-22 wick. The other two pipes were bare. Note that all of the superalloy pipes had C22 endcaps and fill tubes (due to availability).

Operating temperatures and results for the halide life tests by Anderson et al. are shown in Table 13. The operating temperatures were chosen for the maximum safe operating pressure for each envelope/fluid combination. All of the currently operating life tests have roughly 3,000 hours. As shown in Table 13, all of the GaCl₃/superalloy pipes failed in the C-22 pinch-off within 1 week. The SnCl₄/superalloy pipes have a high ΔT. To date, both TiCl₄ and AlBr₃ appear to be compatible with the three superalloy envelopes. Note that the AlBr₃ tests are conducted at 673 K (400°C), so that this fluid operates up to the temperature where cesium starts to be effective.

In addition, TiBr₄ appears to be compatible with titanium at temperatures up to 653K (380°C). Since TiBr₄ has a lower vapor pressure than TiCl₄ at a given temperature (normal boiling point of 506 K versus 410 K), life tests should be conducted with TiBr₄ and superalloy envelopes at higher temperatures.

Table 13. Halide life tests by Anderson, Bonner, Dussinger, Hartenstine, Sarraf, and Locci, 2007).

Fluid		CP-Ti	Hastelloy		
			C22	C2000	B3
AlBr ₃	ΔT	–	2.5	0	3.3
	Temperature	–	673K (400°C)	673K (400°C)	673K (400°C)
GaCl ₃	ΔT	153	Failed	Failed, C22 Fill Tube	Failed, C22 Fill Tube
	Temperature	613K (340°C)	633K (360°C)	633K (360°C)	633K (360°C)
SnCl ₄	ΔT	–	99.5	106	84.8
	Temperature	–	553K (280°C)	553K (280°C)	553K (280°C)
TiCl ₄	ΔT	–	5.5	3.5	7.9 *
	Temperature	–	573K (300°C)	573K (300°C)	573K (300°C)
TiBr ₄	ΔT	1.9	–	–	–
	Temperature	653K (380°C)	–	–	–

* small amount of NCG at end of pipe

Table 14. Halide Life Test Summary

	Compatible	Incompatible
TiBr ₄ /Titanium	653K (380°C)/3,024 hours/CP-Ti (Anderson)	
TiCl ₄ /Mild Steel	432 K (159°C)/28,540 hrs./A-178 Steel (Saaski)	
TiCl ₄ /Superalloy	573K (300°C)/3,144 hrs./Hastelloy (Anderson)	
TiCl ₄ /Titanium	500 K (227°C)/4,019 hrs./CP-Ti (Locci)	
TiCl ₄ /Aluminum		438 K (165°C)/2,500 hrs./Al-6061 (Saaski)
SnCl ₄ /Aluminum		432 K (159°C)/--- hrs./Al-6061 (Saaski)
SnCl ₄ /Mild Steel	429 K (156°C)/27,7500 hrs./A-178 Steel (Saaski)	
SnCl ₄ /Superalloy		553K (280°C)/3,144 hrs./Hastelloy (Anderson)
AlBr ₃ /Superalloy	673K (400°C)/2,952 hrs./Hastelloy (Anderson)	
AlBr ₃ /Aluminum		500 K (227°C)/4,290 hrs./Al-5052 (Locci) 500 K (227°C)/1,100 hrs./Al-6061 (Locci)
AlBr ₃ /Titanium		500 K (227°C)/1,100 hrs./CP2-Ti (Locci)
GaCl ₃ /Superalloy		633K (360°C)/--- hrs./Hastelloy C-22 (Anderson)
GaCl ₃ /Titanium		613K (340°C)/2,952 hrs./CP-Ti (Anderson)
SbCl ₃ /Aluminum		500 K (227°C)/--- hrs./Al-6061 (Saaski)
SbCl ₃ /Mild Steel		476 K (203°C)/5,000 hrs./A-178 Steel (Saaski)
SbBr ₃ /Aluminum		500 K (227°C)/5,000 hrs./Al-6061 (Locci)

Halide life tests are summarized in Table 14. Based on relatively short term life tests, the halides appear to be suitable for temperatures up to 673 K (400°C), and possibly at higher temperatures. Tests are ongoing with TiBr₄/titanium at 653K (380°C), and with AlBr₃/Superalloys at 673K (400°C). Very long term life tests show that TiCl₄ and SnCl₄ are both compatible with mild steel. No tests to date with an aluminum envelope have been successful. This is due to the very high decomposition potential of aluminum when compared to other metals.

VI. Conclusions

Life tests have been run with three elemental working fluids: sulfur, sulfur-iodine mixtures, and mercury. Other fluids offer benefits over these three liquids in this temperature range. Mercury is toxic, has a high density, and problems have been observed with getting the mercury to wet the heat pipe wick. Sulfur and Sulfur/Iodine have high viscosities, and low thermal conductivities.

Mercury is compatible with 347 SS at 603 K (330°C) based on a long term life test. Sulfur is compatible with pure aluminum at 873 K (600°C) based on a short term life test. Since sulfur has a very high liquid viscosity in this temperature range, iodine is generally used to reduce the viscosity. Sulfur-10% Iodine is compatible with 304 SS at 623 (350°C) based on a 1,000 hour life test.

Life tests have been conducted with 19 different organic working fluids. As the temperature is increased, all of the organics start to decompose. Typically they generate non-condensable gas, and often the viscosity increases. At high enough temperatures, carbon deposits can be generated. The maximum operating temperature is a function of how much NCG can be tolerated, and the heat pipe operating lifetime. Three sets of organic fluids stand out as good intermediate temperature fluids:

1. Diphenyl, Diphenyl Oxide, and Eutectic Diphenyl/Diphenyl Oxide (Dowtherm A, Therminol VP, Diphyl)
2. Naphthalene
3. Toluene

When using diphenyl, diphenyl oxide, or diphenyl/diphenyl oxide at temperatures over 673 K (400°C), non-condensable gas is generated in a relatively short time period. The exact period depends on the fluid and material, and decreases as the temperature increases. Between 300 and 400°C, these fluids are generally suitable, for short duration tests near 400 C, and long duration tests near 300 C (For example, Groll et al. found that 321 SS was compatible for ~40,000 hours at 573 K (300°C), but not at 623 K (350°C).

All tests with naphthalene and toluene to date have been compatible, but at lower temperatures than the diphenyl and diphenyl oxide tests. Toluene was compatible with a copper-nickel alloy at 553 K (280°C), as well as with aluminum, mild steel, stainless steel, and titanium at lower temperatures. This is probably close to the maximum useful range of toluene, since the critical point of toluene is 592 K (319°C). While water is generally a better working fluid at 550 K and below, potential benefits of toluene include a lower melting temperature, a lower vapor pressure at a given temperature, and proven compatibility with more envelope materials.

Naphthalene is compatible with stainless steel, copper-nickel, and titanium, based on long term life tests at 593 K (320°C) and above. It has also been shown to be compatible at lower temperatures with aluminum and mild steel. It was compatible with stainless for short term tests at 380°C. Further life testing with naphthalene is recommended.

While fluorinated compounds are believed to be more stable than the same compound without fluorine, this has not been verified in life tests date. Decafluorobiphenyl (C₁₂F₁₀) was less stable than Diphenyl (C₁₂H₁₀) under the same test conditions. Naphthalene was compatible with mild steel at 623 K (350°C) for 5,520 hours, while Monochloronaphthalene was found to be unsuitable after 642 hours at 560 K (287°C), and Octafluoronaphthalene had NCG gas generation at 488 K (215°C). Other stable, fluorinated life tests have been conducted at temperatures of 530 K (257°C) and below.

Based on relatively short term life tests, the halides appear to be suitable for temperatures up to 673 K (400°C), and possibly at higher temperatures. Tests are ongoing with TiBr₄/titanium at 653K (380°C), and with AlBr₃/Superalloys at 673K (400°C). Very long term life tests show that TiCl₄ and SnCl₄ are both compatible with mild steel. No tests to date with an aluminum envelope have been successful. This is due to the very high decomposition potential of aluminum when compared to other metals. However, property data for the halides is incomplete.

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References

- Anderson, W.G., Bonner, R.W., Dussinger, P.M., Hartenstine, J.R., Sarraf, D.B., and Locci, I.E., "Intermediate Temperature Fluids Life Tests – Experiments" Proceedings of the 2007 IECEC, AIAA, St. Louis, MO, June 25-27, 2007.
- Anderson, W.G., Dussinger, P.M., Bonner, R.W., and Sarraf, D.B., "High Temperature Titanium-Water and Monel-Water Heat Pipes," Proceedings of the 2006 IECEC, AIAA, San Diego, CA, June 26-29, 2006b.
- Anderson, W.G., Dussinger, P.M., and Sarraf, D.B., "High Temperature Water Heat Pipe Life Tests," STAIF 2006, pp. 100-107, American Institute of Physics, Melville, New York, 2006a.
- Anderson, W.G., "Evaluation of Heat Pipes in the Temperature Range of 450 to 700 K," STAIF 2005, Albuquerque, NM, February 13-17, 2005.
- Anderson, W.G., Sarraf, D.B., Dussinger, P.M., Stern, T., and Barth, J., "Development of a High Temperature Water Heat Pipe Radiator," Proceedings of the 2005 IECEC, AIAA, ISBN 1563477696, San Francisco, Ca, August 15-18, 2005.
- Anderson, W.G., Rosenfeld, J.R., Angirasa, D., and Mi, Y., "The Evaluation of Heat Pipe Working Fluids In The Temperature Range of 450 to 750 K," Proceedings, STAIF-2004, pp. 20-27, Albuquerque, NM, February 8-12, 2004.
- Anderson, W.G., "Sodium-Potassium (NaK) Heat Pipe," *Heat Pipes and Capillary Pumped Loops*, Ed. A Faghri, A. J. Juhasz, and T. Mahefky, ASME HTD, 236, pp. 47-53, 29th National Heat Transfer Conference, Atlanta, Georgia, August 1993.
- Basiulis, A., and Prager, R. C., "Compatibility and reliability of heat pipe materials," AIAA-1975-660, 10th AIAA Thermophysics Conference, Denver, Colo., May 27-29, 1975
- Basiulis, A., and Fuller, M., "Operating Characteristics and Long Term Capabilities of Organic Fluid Heat Pipes," AIAA No. 71-408, AIAA 6th Thermophysics Conference, 1971.
- Busse, C.A., Loens, J., and Campanile, A., "Hydrogen Generation in Water Heat Pipes at 250°C," Preprints of the First International Heat Pipe Conference, Stuttgart, Federal Republic of Germany, October 15-17, 1973.
- Devarakonda, A. and Anderson, W.G., "Thermo-Physical Properties of Intermediate Temperature Heat Pipe Fluids," STAIF 2005, Albuquerque, NM, February 13-17, 2005. NASA Report NASA/CR—2005-213582, available from the NASA Glenn Technical Reports Server, <http://gltrs.grc.nasa.gov/>.
- Devarakonda, A., Xiong, D., and Beach, D.E., "Intermediate Temperature Water Heat Pipe Life Tests," STAIF 2005, Albuquerque, NM, February 13-17, 2005. NASA Report NASA/TM-2005-213581, 2005, available from the NASA Glenn Technical Reports Server, <http://gltrs.grc.nasa.gov/>.
- Devarakonda, A., and Olminky, J.E., "An Evaluation of Halides and Other Substances as Potential Heat Pipe Fluids," Proceedings of the 2004 IECEC, Providence, RI, August 16-19, 2004.
- Dussinger, P.M., Anderson, W.G. and Sunada, E.T., "Design and Testing of Titanium/Cesium and Titanium/Potassium Heat Pipes," Proceedings of the 2005 IECEC, AIAA, ISBN 1563477696, San Francisco, Ca, August 15-18, 2005.
- Deverall, J.E., "Mercury as a Heat Pipe Fluid," ASME Paper 70-HT/Spt-8, American Society of Mechanical Engineers, 1970.
- Eastman, Y., personal communication, 2007
- Ernst, D.M., personal communication, 2006.
- Feldman, K.T., and Kenney, D.D., "The Compatibility of Mild Carbon Steel and Water in a Heat Pipe Application," Journal of Heat Recovery Systems (now Applied Thermal Engineering), Vol. 1(4), pp. 299-307, 1981.
- Groll, M., Brost, O., Heine, D., and Spendel, T., "Heat Transfer, Vapor-Liquid Flow Interaction and Materials Compatibility in Two-Phase Thermosyphons," CEC Contractors Meeting, Heat Exchangers – Heat Recovery, Brussels, June 10, 1982.
- Groll, M., Brost, O., and Roesler, S., "Development of High Performance Closed Two-Phase Thermosyphons as Heat Transfer Components for Heat Recovery from Hot Waste Gases," EG-Status Seminar, Brussels, October, 1987.
- Groll, M., "Heat Pipe Research and Development in Western Europe", Heat Recovery Systems and CHP (Combined Heat & Power), 9(1), pp. 19-66, 1989.
- Grzyll, L.R., Ramos, C., and Back, D.D., "Density, Viscosity, and Surface Tension of Liquid Quinoline, Naphthalene, Biphenyl, Decafluorobiphenyl, and 1,2-Diphenylbenzene from 300 to 400°C," J. Chem. Eng. Data, Vol. 41, pp. 446-450 1996.
- Grzyll, L.R., Back, D.D., Ramos, C., and Samad, N.A., "Characterization and Testing of Novel Two-Phase Working Fluids for Spacecraft Thermal Management Systems Operating Between 300°C and 400°C," Final Report to Phillips Laboratory, Kirtland Air Force Base, No. PL-TR-95-1089, 1995.
- Grzyll, L.R., Back, D.D., Ramos, C., and Samad, N.A., "Characterization and Testing of Novel Two-Phase Working Fluids for Spacecraft Thermal Management Systems Operating Between 300°C and 400°C," Proceedings of the 1st Annual Spacecraft Thermal Control Symposium, Albuquerque, NM 1994.
- Grzyll, L.R., "Heat Pipe Working Fluids for Thermal Control of the Sodium/Sulfur Battery," Proceedings of the 26th Intersociety Energy Conversion Engineering Conference, Vol. 3, pp. 390-394, American Nuclear Society, La Grange, Illinois, 1991.
- Hartenstine, J.R., personal communication, 2007.

Heine, D., Groll, M., and Brost, O., "Chemical Compatibility and Thermal Stability of Heat Pipe Working Fluids for the Temperature Range 200 °C to 400 °C," 8th ChiSA Congress, Prague, September 3-7, 1984.

Jaworskie, D., personal communication, April 5, 2007.

Kenney, D.D., and Feldman, K.T., "Heat Pipe Life Tests at Temperatures up to 400°C," Proceedings of the 13th Intersociety Energy Conversion Engineering Conference, pp. 1056-1059, San Diego, CA, Aug. 20-25, 1978.

Locci, I.E., Devarakonda, A., Copeland, E.H., and Olminsky, J.K., "Analytical and Experimental Thermo-Chemical Compatibility Study of Potential Heat Pipe Materials," Proceedings of the 2005 IECEC, San Francisco, CA, August 15-18, 2005.

Los Alamos Scientific Laboratory, "Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending April 30, 1970, Part 1," Report No. LA-4446-MS, pp. 2-5, May, 1970.

Los Alamos Scientific Laboratory, "Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending January 31, 1969, Part 1," Report No. LA-4039-MS, pg 8, February, 1969.

Los Alamos Scientific Laboratory, "Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending January 31, 1968, Part 1," Report No. LA-3881-MS, pg. 4, February, 1968a.

Los Alamos Scientific Laboratory, "Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending April 30, 1968, Part 1," Report No. LA-3941-MS, pg. 2, May, 1968b.

Los Alamos Scientific Laboratory, "Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending October 31, 1968, Part 1," Report No. LA-4109-MS, pp. 5-6, November, 1968c.

Lundberg, L.B., Merrigan, M., Prenger, F.C., and Dunwoody, W., "Sulphur Heat Pipes," Energy Technology, Los Alamos Scientific Laboratory, LA 8797-PR, October-December 1980, pp. 69-70.

Novotna, I., Nassler, J., and Zelko, M., "Compatibility of Steel-Water Heat Pipes," Proceeding of the Third International Heat Pipe Symposium, Tsukuba, Japan, pp. 89-95, September 12-14, 1988.

Polasek, F., and Stulc, P., "Heat Pipe for the Temperature Range from 200 to 600°C," Proc., Second International Heat Pipe Conference, Bologna, Italy, 2, pg. 711, 1976.

Reid, R.S., Merrigan, M.A., and Sena, J. T., "Review of Liquid Metal Heat Pipe Work at Los Alamos," 8th Symposium on Space Nuclear Power Systems, Albuquerque, NM, January 6-10, 1991.

Saaski, E.W., and Owzarski, P.C., "Two-Phase Working Fluids for the Temperature Range 50° to 350°C," Sigma Research, Inc., Final Report, Contract NAS3-20222, NASA Lewis Research Center, June 1977a.

Saaski, E.W., and Tower, L., "Two-Phase working fluids for the temperature range 100-350°C," American Institute of Aeronautics and Astronautics, 12th Thermophysics Conference, Albuquerque, NM, June 27-29, 1977b.

Saaski, E.W., and Hartl, J.H., "Two-Phase Working Fluids for the Temperature Range 50 to 350°C," Sigma Research, Inc., Phase II Final Report, Contract NAS3-21202, NASA Lewis Research Center, March, 1980.

Sanzi, J.L., "Thermal Performance of High Temperature Titanium – Water Heat Pipes by Multiple Heat Pipe Manufacturers," Space Technology and Applications International Forum (STAIF-07), American Institute of Physics, Melville, New York, 2007.

Sarraf, D.B., Bonner, R.W., and Colahan, D., "Passive Thermal Management for a Fuel Cell Reforming Process," Proceedings of the 2006 IECEC, AIAA, San Diego, CA, June 26-29, 2006.

Tarau, C., Sarraf, D.B., Locci, I.E., and Anderson, W.G., "Intermediate Temperature Fluids Life Tests – Theory," Proceedings, STAIF 2007, Albuquerque, NM, February 11-15, 2007.

Timrot, D.L., Serednitskaya, M.A., Medveditskov, A.N., and Traktueva, S.A., "Thermophysical Properties of a Sulfur-Iodine Binary System as a Promising Heat Transfer Medium for Heat Pipes," Journal of Heat Recovery Systems (now Applied Thermal Engineering), Vol. 1(4), pp. 309-314, 1981.

Vasil'ev, L.L., Volokhov, G.M., Gigevich, A.S., and Rabetskii, M.I., "Heat Pipes Based on Naphthalene," Journal of Engineering Physics and Thermophysics, Vol. 54, No. 6, pp. 623-626, 1988.