HEAT TRANSFER SALT FOR
HIGH TEMPERATURE STEAM GENERATION

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OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION • FOR THE U.S. ATOMIC ENERGY COMMISSION

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Reactor Chemistry Division

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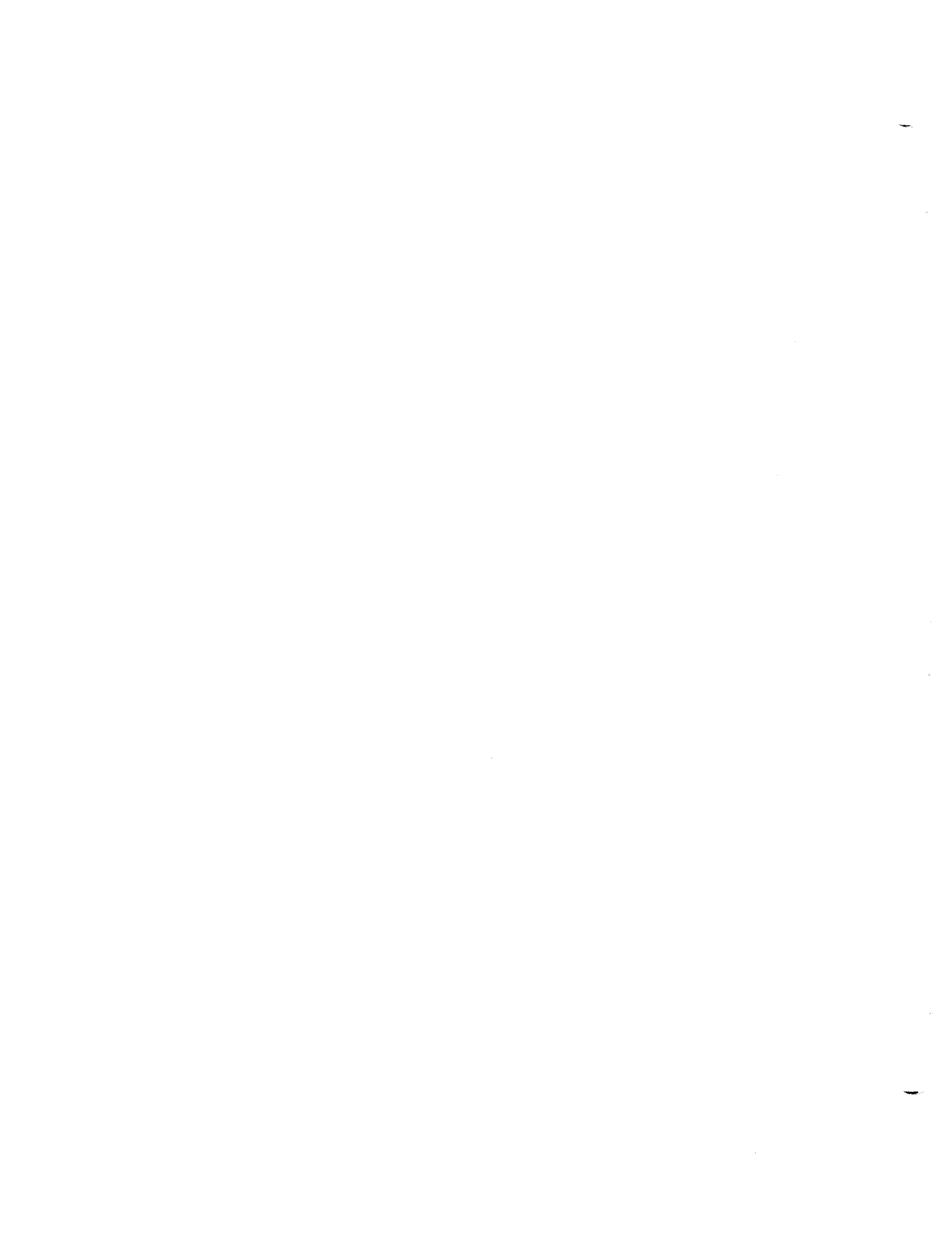
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ABSTRACT AND SUMMARY

A literature review and canvass of vendors reveals no experience and sparse, short term data bearing on the possible use of heat transfer salt (HTS - 40% $NaNO_2$, 7% $NaNO_3$, 53% KNO_3) at temperatures > 1000°F. Stability data found indicate that in a steam generator loop running with a maximum temperature of 1100°F about 0.5% of the $NaNO_2$ would decompose per day according to the overall reaction

$$5NaNO_2 \rightarrow 3NaNO_3 + Na_2O + N_2 .$$
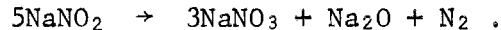
In the size loop envisioned for a Molten Salt Breeder Reactor (MSBR) the nitrogen produced would amount to 3400 ft^3/d.

Austenitic stainless steels are recommended materials of construction above 850°F and short term test data suggest high nickel alloys like Incoloy, and Inconel would also be suitable. I would infer that Hastelloy-N would be resistant also.

A tertiary HTS steam generator loop would provide an effective barrier to transfer of tritium formed in an MSBR to the steam generator. The tertiary loop is required to eliminate the possibility of a violent moderator graphite - HTS reaction. However, projected use in an MSBR at temperatures to ∿ 1100°F would require a substantial development program directed at stability and/or composition re-adjustment and verification of materials compatibility. Thus, the presence of appreciable Na_2O and/or NaOH, depending on steam leakage, at such temperatures might appreciably alter the corrosion picture.

An industrial installation for drying NaOH was operated one and one half years with a maximum temperature of 980°F without difficulties due to corrosion or stability. The salt loop was constructed of ASTM 106 Grade A steel.
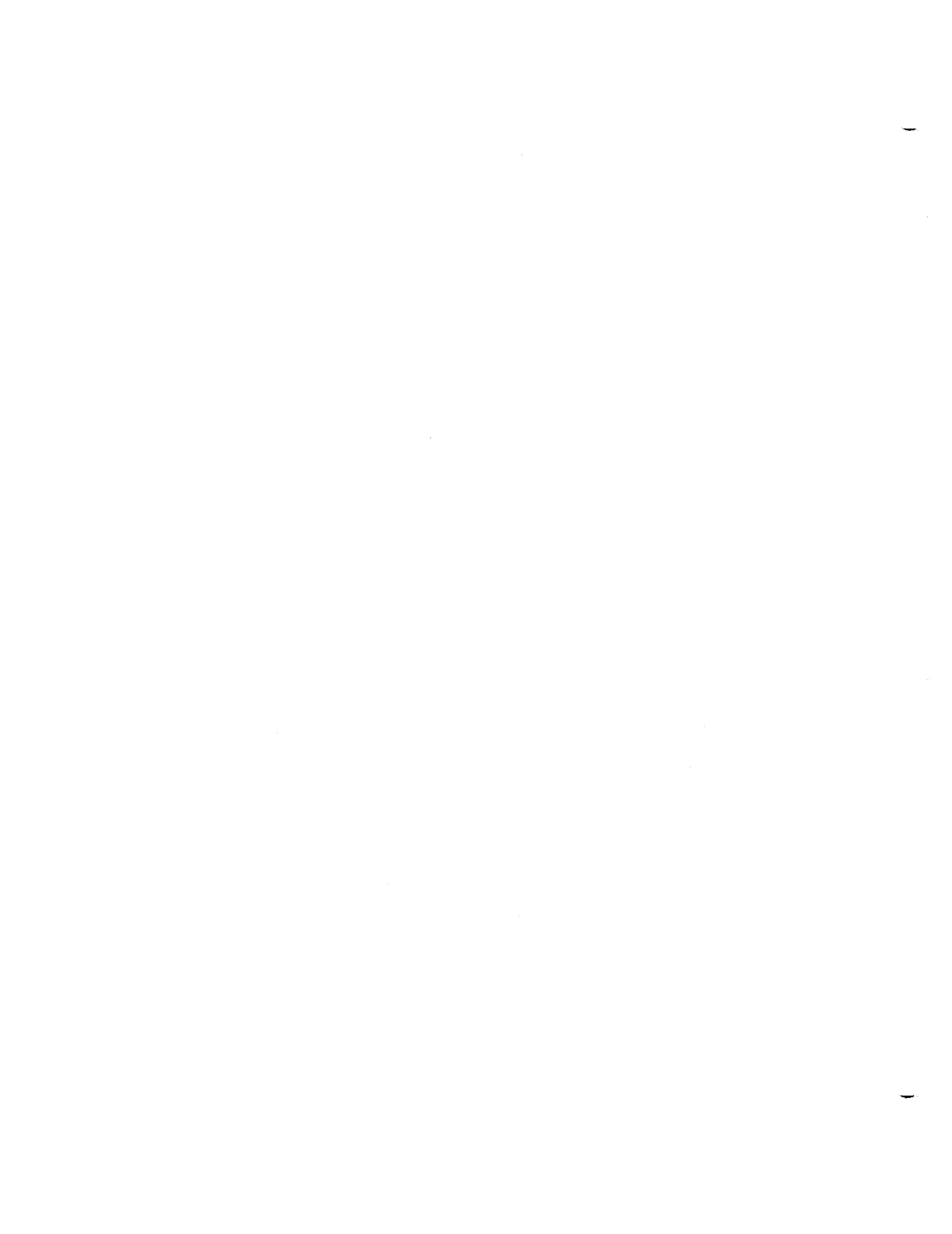
INTRODUCTION

It is estimated that a molten salt breeder reactor will produce ~ 2500 C_i ^3T per day. Transfer of appreciable percentages of this ^3T to the steam generator system must be prevented to avoid resultant contamination and containment problems. A substantial barrier to such transfer would be provided by the use of heat transfer salt - HTS (Na-K, nitrite-nitrate mixture) in the reactor steam generator circuit. The HTS would be expected to block the tritium transfer in several ways:

1. Conversion of hydrogen to water by the reactions: (Cf Appendix A).
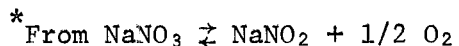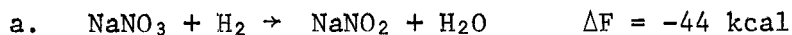
    a. $NaNO_3 + H_2 \rightarrow NaNO_2 + H_2O$ $\Delta F = -44$ kcal

    b. $H_2 + 1/2\ O_2^* \rightarrow H_2O$ $\Delta F = -46$ kcal

    *From $NaNO_3 \rightleftarrows NaNO_2 + 1/2\ O_2$

2. HTS systems are compatible with steam (used as inert cover gas in industrial applications[1]) so the water formed in 1 would not be corrosive to the steam generator tubing, thereby eliminating that possible alternate tritium transport mechanism.

3. The tritiated water could be stripped from the HTS as required.

4. Protective oxides, which inhibit tritium diffusion, are formed on alloys in contact with HTS.[2]

Originally, consideration was given to the substitution of the HTS for the fluoroborate secondary coolant. However, the probability of a violent interaction[3,4] between the HTS and the graphite moderator (see section, Safety Precautions) in the event of contact during an accident eliminated this possibility. Such a violent interaction has been confirmed by an investigation carried out by Bamberger and Ross (Cf. Appendix B). Use of HTS as the secondary coolant would also necessitate extensive demonstration of compatibility with the very high radiation intensities in the primary heat exchanger.

An alternate possibility is the use of HTS in a separate, tertiary steam generator loop; $^7LiF\text{-}BeF_2$, or 92 $NaBF_4\text{-}8NaF$, would be used in the secondary loop. Increased cost would make this approach untenable were it not for other positive considerations. These include:

1. The low, 288°F, melting point of HTS makes possible significant simplification of the steam system; cheaper alloys would also be useable.

2. Steam leaks into HTS will not enhance corrosion as it would in $92NaBF_4$-$8NaF$ or other fluoride coolants. Assuming all the water leaking into the fluoroborate participates, ∿ 5 moles/h is equivalent to 1 mpy (uniform) for the entire secondary loop of an MSBR.

3. Use of ^7LiF-BeF_2 in the secondary loop would eliminate poisoning of the fuel by leakage of secondary salt into the fuel; minor reverse leakage would also be more tolerable since the secondary loop could be included in the reactor cell. A substantially cheaper alternate might be NaF-ZrF_4 which has a reasonably low cross section and which probably is removable by the presently considered fuel processing methods.

Robertson[5] has estimated the added cost of a third HTS loop, taking into account savings due to the above and other considerations. He concluded, "Addition of the (L_2B and HTS) loops would increase the power production costs by 0.2 - 0.3 mills/kwhr, making the total cost about 5.5 mills/kwhr." The added capital cost amounted to 10 to 13 million dollars (206 total) depending on whether Incoloy 800 could be substituted for Hastelloy N in the secondary loop.

While this estimated increased cost was not favorable to the third loop approach to tritium control, this survey of the "state of the art" as regards use of HTS under MSBR conditions was also conducted. At first blush it appeared that the widespread industrial experience would minimize the development required for such adaptation; unfortunately the survey showed such optimism was not well founded.

COMPOSITION AND STABILITY

Heat transfer salt was developed by duPont in the thirties[6] for use in the chemical and petroleum process industries, notably the Houdry catalytic cracking and refining units. The usual weight % composition is $40NaNO_2$, $7NaNO_3$, 53 KNO_3 which melts at 288°F; the melting point is not greatly affected by appreciable alterations as shown in Table 1.

Table 1[*] - The Freezing Points of $NaNO_2$-$NaNO_3$-KNO_3
Mixtures

% $NaNO_2$ by wt.	% $NaNO_3$ by wt.	% KNO_3 by wt.	Freezing Point °F
100	0	0	540
50	0	50	282
44	3	53	284
42	3	55	287
40	0	60	289
40	7	53	288 ("HTS")
38.5	11	50.5	293
38	6	56	292
35	7	58	311
34	13	53	305
34	3	63	328
30	20	50	305
30	10	60	312
30	0	70	333
0	100	0	586
0	0	100	633

[*]Table II, page 375, from Ref. 1.

Apparently the technology grew "like Topsy," as regards both composition and materials of construction, out of many years experience with salt baths used in heat treating and cleaning metals.

Presumably because of this and proprietary considerations, there is a paucity of quantitative data on stability and corrosion in the open and vendor literature; this in spite of widespread use in substantial quantities — one Houdry unit used $\sim 10^6$ lb. Low cost (still 15¢/lb) and tractability under "rough and ready" handling also may have contributed.

At the time of its introduction the low, 288°F, melting point was a major advantage since it was below the temperature at which steam was available in most plants. The need for steam jackets and tracers, design

for complete gravity drainage, and other freeze-melt problems, however,
gave the competitive nod to organics like "Dowtherm" for temperatures below
700°F. A patented European development available under license in the U.S.
by American Hydrotherm Corp.,[7,8] eliminates these problems by "salt dilution."
This involves adding sufficient water to the salt at startup—and starting
at 500°F on cooldown—to keep the salt in solution at ambient ($\sim$ 50°F)
temperature. The water is gradually flashed off at 200 - 500°F while
circulating through the system; this eliminates the severe thermal shock
of starting circulation at 500°F in a cold system or need for provision of
an alternate preheat capability. Ten pilot plant and thirty-two plant size
systems of this kind have been in operation since 1961 or are now under
construction. American Hydrotherm claims a 20% saving on capital costs
for the use of salt dilution instead of dry salt,[7] with no resultant
materials or handling problems. A point by point comparison by American
Hydrotherm[9,10] of "Dowtherm A" with HTS using salt dilution indicates
substantially lower costs for the latter. The dilution process also
eliminates problems which may be caused by increases in melting point due
to oxidation of nitrite to nitrate or formation of relatively insoluble
Na_2CO_3 from pickup of CO_2.[8] J. Alexander, Jr., and S. G. Hindin[11] have
published detailed studies of the freezing points of the system sodium and
potassium nitrites and nitrates and the effects of impurities on them.
Commercially used measures for countering these effects also discussed
included:

1. Use of hydrogen treatment in a bypass adsorption tower to reduce
excess nitrate to nitrite.*

2. Addition of concentrated HNO_3 or gaseous NO_2 to convert hydroxides
and carbonates to nitrates and nitrites.

3. Cold trap use to reduce carbonate levels.

Sodium hydroxide is formed by the reaction of the Na_2O thermal
decomposition product of HTS with water.

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*Paul Larson[12] indicated hydrogen treatment was no longer used because
 addition of fresh salt proved cheaper than providing for doing the
 reduction.

Various attempts to find definitive data on thermal stability at elevated temperatures were fruitless in spite of vendor and open literature recommendations for use to 1000 and 1100°F.[1,7,8,13-15] These efforts are well characterized by one lead which reported successful use of HTS at temperatures as high as 1200 - 1300°F. A couple of phone calls located my original informant's source but produced the usual disclaimer. The use at such temperatures was occasional and involved only several pounds of HTS, which gave no trouble "if changed frequently." When asked what happened to the HTS, the reply was, "It gets pretty lumpy .....fast."

Up to ∿ 850°F widespread experience indicates HTS is stable in carbon steel systems even when operated with air access to the system. Slow oxidation of nitrite to nitrate and carbonate formation may increase the freezing point slowly, but these changes apparently do not result in any difficulties. Thus, Crowley and Helms trip report[16] gives the composition

	%
$NaNO_3$	9.28
$NaNO_2$	30.3
KNO_3	48.7
K_2CO_3*	3.38
KCl	0.27
	Σ91.93

*Alexander and Hindin[11] indicate Na_2CO_3 would be more likely.

for bulk salt after an indeterminate period of operation (probably > 2 years). The system has remained in operation with this salt charge since April 1967 (∿ 24000 h @ 800°F maximum) with no requirement for composition adjustment; new salt is added only to make up for losses. Helms also reports[17] 8 years use of the same batch of "Hitec" at a maximum bulk salt temperature of 843°F without observable deterioration; air access was prevented by nitrogen blanketing in this application. Vendor literature recommends such nitrogen, or steam, blanketing[13,14] above 850°F, but contacts with vendor personnel uncovered no experience at such temperatures.

Data for the rate of disappearance of nitrite are given by W. H. v.d. Broek[18] for heat transfer salt used in finishing (drying to ∿ 100 %) NaOH.

In this application the composition

$$48\% \quad KNO_3$$
$$49\% \quad NaNO_2$$
$$3\% \quad Na_2CrO_4$$

was used. The addition of chromate is reported (ca. 1940)[19] to inhibit decomposition of the nitrite to nitrate and oxide but Rottenberg (1957)[3] states that the claims had not been established. Paul Larson[12] also indicated that duPont had not found such additions either harmful or beneficial; but some users insisted on making them. W. H. v.d. Broek[18] showed nitrite decrease rates graphically for 1 1/2 and 2 1/2 year periods of operation at maximum temperatures of 980 and 800°F respectively as shown in Fig. 1. Unfortunately only the plots for nitrite loss were given, so it is not possible (no analyses) to determine how the two possible reactions

1) $\quad 2NaNO_2 + O_2(air) \rightarrow 2NaNO_3$

2) $\quad 5NaNO_2 \xrightarrow{\Delta} 3NaNO_3 + Na_2O + N_2$
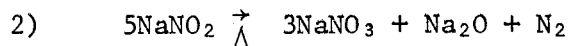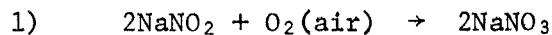
contributed to the observed changes. Also, during the high temperature operation, concern about the pump caused the system to be opened to the atmosphere frequently; this was minimized during the second run at 800°F. Still another factor may have contributed to the rapid loss of nitrite at 980°F; it is reported[20] that the decomposition of alkali nitrate – nitrite mixtures is catalyzed by iron above 520°C, and the system described by v.d. Broek was made of carbon steel. Stainless steel does not catalyze decomposition of alkali nitrate – nitrite mixtures even at 800°F.[21]

Some laboratory studies in 316 stainless steel containers have been carried out at 1100°F.[12] Figure 2 shows the effect of various blanket gases on the melting point of "Hitec;" "melting point" in HTS parlance refers to the temperature at which any solid phase appears. Reactions considered significant in the decomposition of nitrate – nitrite mixtures (the sodium salts are indicated because they are less stable[21]) include:
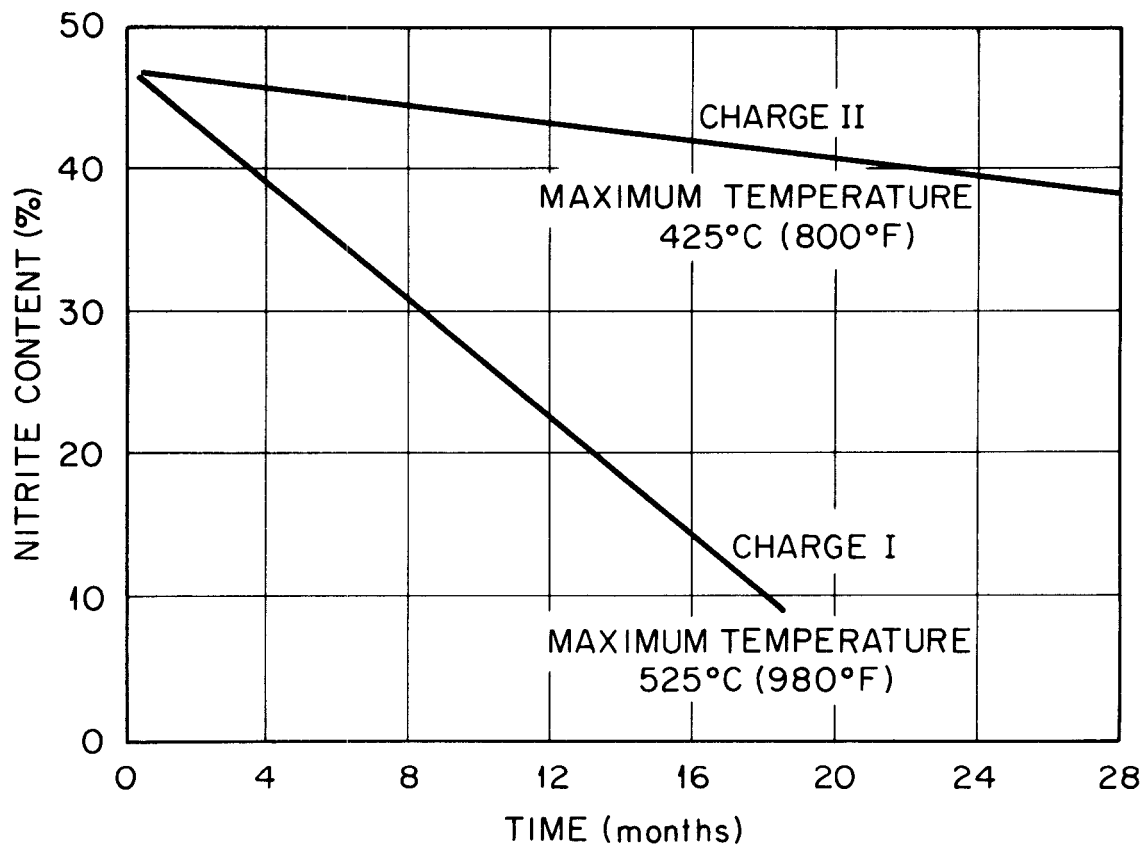
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Fig. 1. Nitrite Decomposition in Industrial Process Loop Using HTS[*]

[*]Per W. H. v.d. Broek, see Ref. 18.

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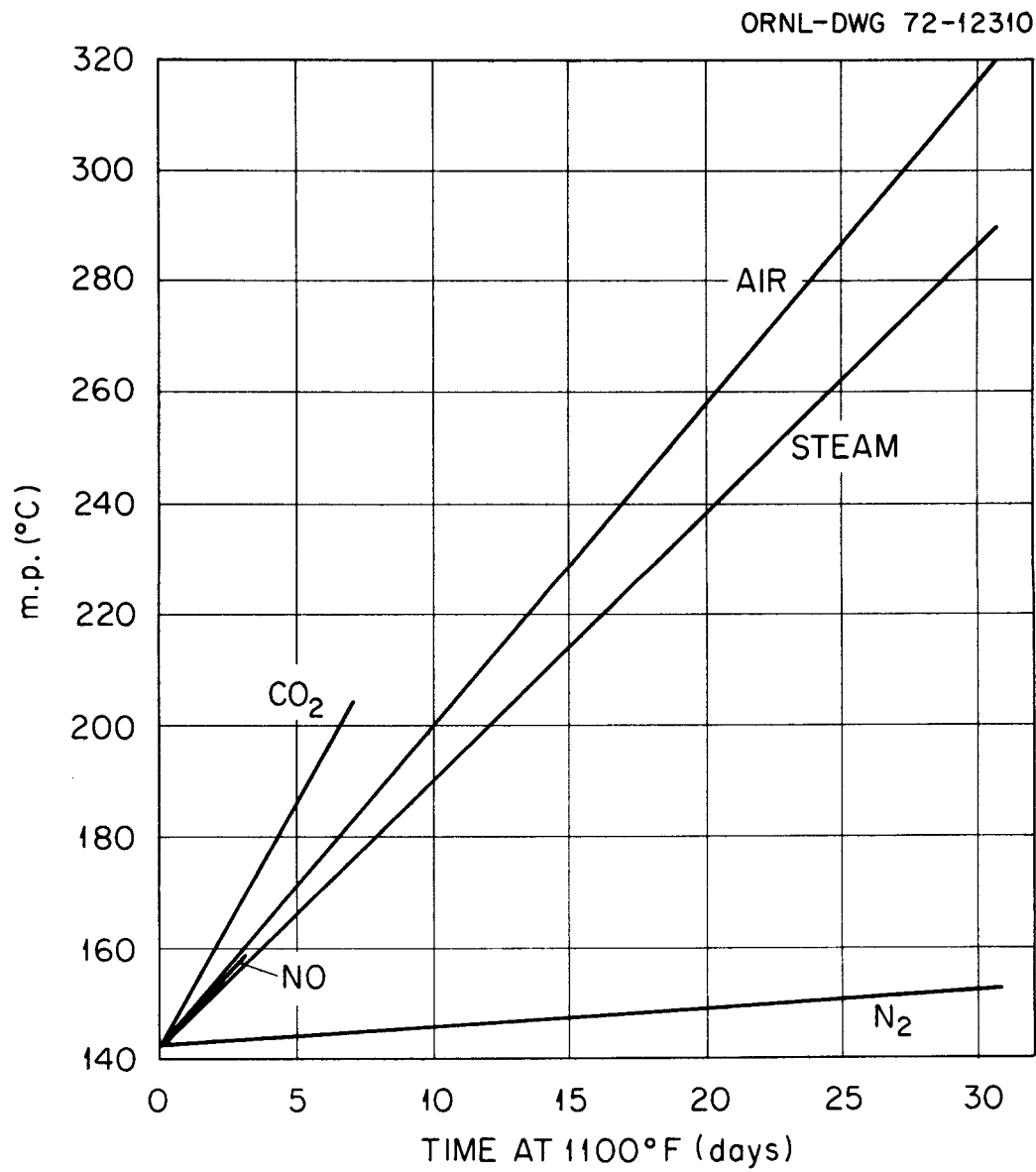
Fig. 2. Effect of Various Blanket Gases on HTS m.p.

(See Ref. 12)

$$NaNO_3 \; \rightleftarrows \; NaNO_2 + \tfrac{1}{2}O_2 \tag{1}$$

$$2NaNO_2 \; \rightarrow \; Na_2O + N_2O_3 \tag{2}$$

$$NaNO_2 + N_2O_3 \; \rightarrow \; NaNO_3 + \; 2NO \tag{3}$$

$$NaNO_2 + 2NO \; \rightarrow \; NaNO_3 + N_2O \tag{4}$$

$$NaNO_2 + N_2O \quad NaNO_3 + N_2 \tag{5}$$
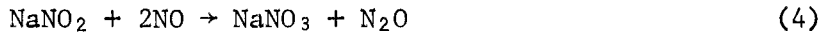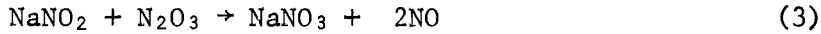
The formation of Na_2CO_3 and/or NaOH from reactions of carbon dioxide or water with the Na_2O formed in (2) account for the melting point increases with air, steam, and carbon dioxide blankets. Conversion of the nitrite to nitrate by reactions (4) and (5) probably accounts for the rapid increase in melting point with NO as the blanket gas. Reactions (2) thru (5) produce the rise under nitrogen, again through conversion of nitrite to nitrate. This is indicated by analysis of a salt mixture after six weeks under nitrogen at 1100°F which showed the composition to be that given in Table 2, with a melting point of 329°F.[12]

Table 2. Change in "Hitec" Composition After Six
Weeks at 1100°F

Compound	% Composition	
	Original	Final
$NaNO_3$	7	18
$NaNO_2$	40	28
KNO_3	53	52
Na_2O	--	2

Analysis of gas evolved gave 98.5% N_2 and 1.5% N_2O.

Gurovich and Shtokman[22] and Voskresenskaya and Berul'[23] also conducted laboratory investigations of HTS stability at temperatures > 1000°F and found appreciable nitrogen evolution.

The studies from which decomposition data are available are summarized in Table 3. After an initial period, the generally accepted overall

Table 3. Long Term Decomposition Studies of Heat Transfer Salt

Author & Ref. No.	Temp. °F	Type System	Wt. HTS Used g	Exposure Time	Original Composition				Observations	Equivalent* rate of N_2 evolution (cc/mole $NaNO_2$/d)
					$NaNO_2$	$NaNO_3$	KNO_3	Na_2CrO_4		
Larson, 12	1100	316 ss autoclave	1000	42 d	40	7	53	–	Final composition: 28% $NaNO_2$, 2% Na_2O	39
v.d. Broek, 18	980			18 mo					Final composition: 10% $NaNO_2$	11
		ASTM A106 industrial process (circulating loop)	Very large		49		48	3		
	800			30 mo					Final composition: 38% $NaNO_2$	1.3
Gurovich & Shtokman 22	934	Steel (.09C, 0.9Cr, 0.24V, 0.4Mo) autoclave	145	30 d	40	7	53	–	10 cc N_2/d after 5 d	12
Voskresenskaya & Berul' 23	1040	Ag autoclave	50	30.8 d	40	7	53	–	0.09 g N_2 evolved	8.3
	1112	Ag autoclave	50	10 d	40	7	53	–	0.07 g N_2 evolved	20
	1112	18Cr 25Ni 2Si steel autoclave	25	10.2 d	40	7	53	–	0.08 g N_2 evolved	45

*Assuming over-all reaction is $5NaNO_2 \rightarrow 3NaNO_3 + Na_2O + N_2$.

decomposition reaction is

$$5NaNO_2 \rightarrow 3NaNO_3 + Na_2O + N_2 \quad .$$
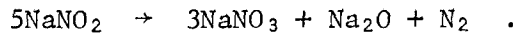
This stoichiometry was used to calculate the nitrogen evolution rate from the results reported by the experimenters; the rate was necessarily assumed to be constant, although Voskresenskaya and Berul'[23] and others indicate it decreases with time. The calculated N_2 evolution rates are those given in the final column in Table 3 and are plotted in Figure 3. Considering the disparate conditions of the several investigations the results are pleasantly consistent although more data would be helpful in more firmly removing the possibility of coincidence. The two low points due to Voskresenskaya and Berul' are for the experiments performed in silver; they and other investigators reported lower decomposition rates in noble metal containers than in carbon and alloy steels. The data due to v.d. Broek are of particular interest; recall that they were obtained from HTS in use in an industrial process loop (dewatering NaOH) and the only temperatures given were the maximums in the loop. Thus the average temperatures were appreciably lower and the data should fall below those from the isothermal studies of Larson and Gurovich and Shtokman — as they do. The consistency in all these studies suggests that the measured decomposition rates may be reasonably accurate for an engineering operation.

If one accepts then that these data may be considered reasonably representative of the tertiary loop as envisioned by Robertson[5] (maximum temperature 1100°F), a decomposition rate of about 40 cc N_2/mole $NaNO_2$/d would be anticipated. For the 9×10^5 lb of HTS (40% $NaNO_2$) in the tertiary loop, this amounts to 4200 moles (3400 ft^3) N_2/d. The nitrite loss would amount to 21,200 (2200 lb) moles per day; i.e., 1.0%/d.

Extrapolation to temperatures above 1100°F is questionable since various workers[23,24] report nitrogen oxides are also evolved above 600°C. These were short term studies, however, and it may be that the evolution of oxides of nitrogen would not continue after a period, as happened in the longer term studies at lower temperatures. Voskresenskaya and Berul'[23] also reported substantial attack on the containers in some of their experiments, unlike other experimenters. When this occurred with high Cr alloys, appreciable amounts of chromate were found in the melts.
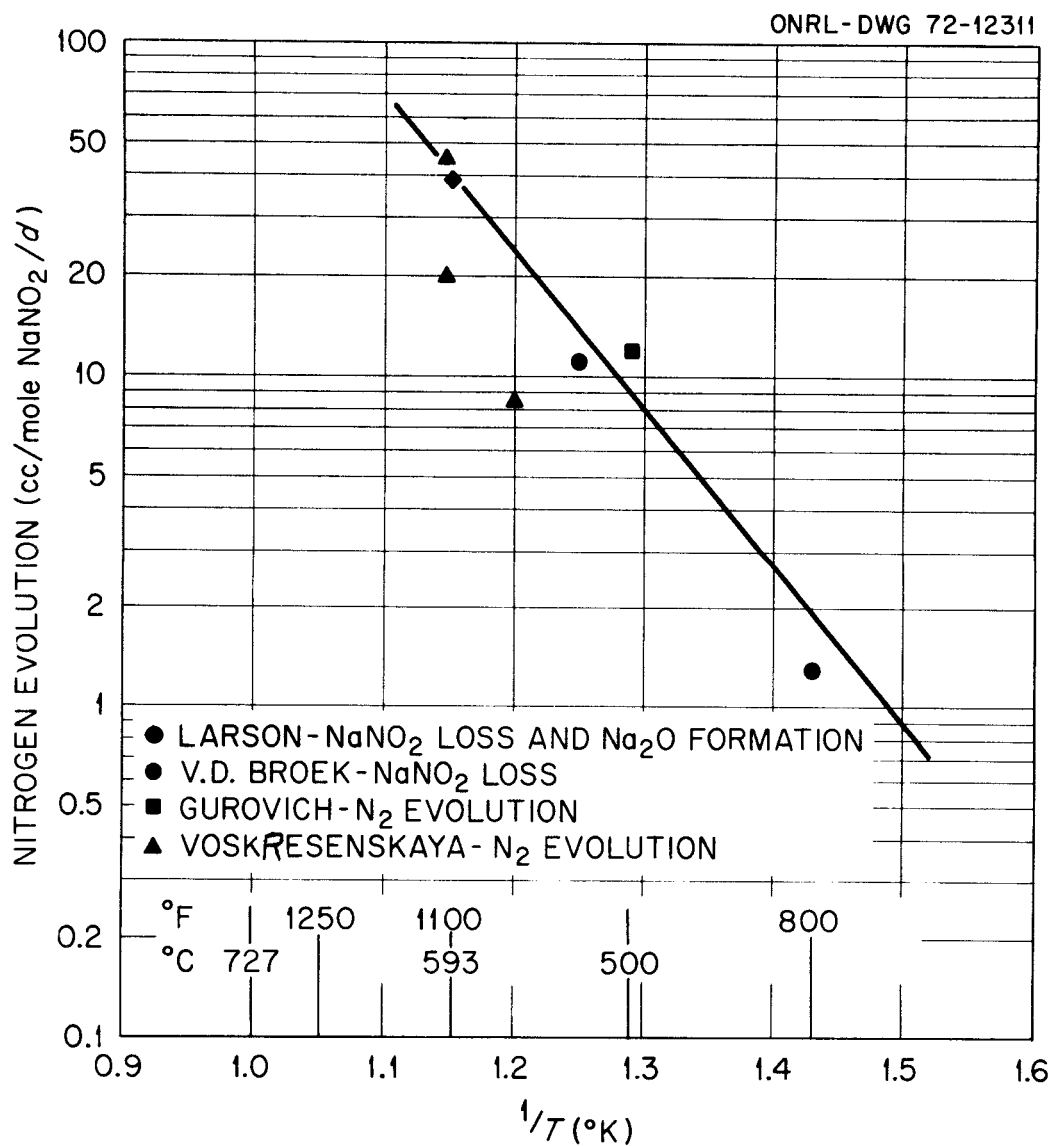
Fig. 3. Rate of Nitrogen Evolution from HTS vs Temperature

Voskresenskaya and Berul'[23] also present data showing the rate of nitrogen evolution as a function of nitrite concentration. These data, interpolated from a small graph and given in Table 4, are only indicative, but they are consistent with other observations. This suggests that a mixture consisting of 8% $NaNO_2$, 42% $NaNO_3$ and 50% KNO_3 might result in lower N_2 evolution rates. The sodium nitrate-sodium nitrite ratio was chosen on the basis of Freeman's[25] observation that the ratio (under one atmosphere of oxygen) is 5.9 at 600°C. Curiously, no observer mentions the evolution of oxygen from the usual mixtures at such temperatures. Alexander and Hindin's[11] data indicate a melting point of $\sim$ 420°F for such a mixture, still adequately low for simplification of the steam cycle.

Apparently the equilibrium

$$NaNO_3 \leftrightarrow NaNO_2 + \tfrac{1}{2}O_2$$
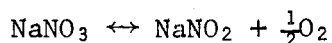
is substantially shifted to the left in HTS; Freeman's data indicate P_{O_2} would be about 0.3 atm at 600°C for the usual mixture. Presumably, however, the equilibrium would shift to the right as the percent nitrate is increased, with resultant evolution of oxygen. Perhaps a mechanism change also occurs and the oxygen appears as the oxides of nitrogen observed by some observers[24,25] at temperatures > 1100°F.

Table 4. Rate of Nitrogen Evolution vs $NaNO_2$ Concentration at 550°C*

% Composition		cc N_2/100g $NaNO_2$/hr
$NaNO_2$	KNO_3	
100	0	360
80	20	135
60	40	15
40	60	8
20	80	5

*These were short term (to 20 h) studies and are therefore only indicative.

CORROSION

As with thermal stability, corrosion data are sparse, and then only from relatively short term tests. Considerable experience has demonstrated acceptable, economic performance with no apparent necessity for quantitative, long term corrosion data. The vendors recommend against use of carbon steel above 850°F; at lower temperatures it gives excellent, long term service.[3,13,16] Rottenberg[3] gives the following description of how troubles can arise in a poorly designed maloperated, direct fired heater,

"The main danger is, as usual, overheating in the stagnant film at heating surfaces. The danger is accentuated in this case by the ability of HTS to detach particles of rust and scale from the inside of the plant and deposit it as sludge at the bottom. Even more important is the fact that with gross overheating, at local temperatures above 700°C, a new reaction sets in with the rapid breakdown of nitrate to oxygen, nitrogen, and oxide. In the presence of a mild steel surface, the oxygen combines with it in a thermite type reaction which is self-sustaining and rapidly burns through the metal. No reaction takes place with stainless steel. In plants operating at high bulk temperatures heating sections should be constructed of stainless steel to obviate this danger."

In the operation described previously, however, v.d. Broek[18] observed no corrosion of the system during 1 1/2 years of operation with a maximum temperature of 980°F. The heater section and the rest of the heat transfer loop were made of carbon steel (ASTM A106) with nickel and "Incoloy" at the interfaces in the NaOH concentrator. Conventional use of nickel and nickel alloys for materials of construction in caustic concentrators using heat transfer salt[26] and v.d. Broek's experience indicate good compatibility for these materials to $\sim$ 1000°F. Other qualitative assessments of corrosion by heat transfer salt during extended operating periods are summarized in Table 5.

Miscellaneous corrosion data are shown in Table 6, taken from the "Hitec" pamphlet.[13] While no experimental details are provided, it is probable that the results are from quite short term (a few hundred hours) tests. The two period tests at 1058°F indicate the corrosion rates of the highly alloyed materials decrease with time. Note, however, that Inconel was an exception in spite of the excellent results at 1000°F and for nickel at 1100°F. Similar results are shown by data furnished by J. Friedman[27] which are presented in Table 7. The relatively short

Table 5. Qualitative Assessments of Corrosion by Heat Transfer Salt

Alloy	Time yr.	Temp. °F	Blanket Gas	System	Comment	Ref.
	3	800	Air	Circulating	No noticeable corrosion	27
ASTM-A-285 Grade C Carbon Steel	3	785-800	Limited Air	Circulating	No noticeable corrosion	27
	2.5	875	Steam	Circulating	No noticeable corrosion	27
347 SS	1.3	1050	Air	Heat Treat Bath	Negligible corrosion	27
347 SS	2	1000	Steam	Circulating	No noticeable corrosion	27
Seamless Stainless Tubing	0.8	930-1020	?	Circulating	No visible attack	13

TABLE 6

CORROSION OF METALS BY "HITEC"

Metals	612°F.	785°F.	850°F.	1000°F.	1058°F. 1st Period	1058°F. 2nd Period	1100°F.
Steel—open hearth (ASME # S-17)	—	—	0.0003	0.001 to 0.002	—	—	0.01 to 0.05
Alloy steels							
15-16% chromium iron	—	—	—	0.0000	—	—	—
"Alcrosil" # 3	—	—	0.0002	0.001	—	—	0.002 to 0.006
"Alcrosil" # 5	—	—	0.0002	0.0005	—	—	0.001 to 0.002
Stainless steels							
Type 304	—	—	—	0.0007	—	—	—
Type 304L	—	—	—	0.0006	—	—	—
Type 309 (annealed)	0.00002	0.00001	0.0000	—	0.00110	0.00064	—
Type 309 Cb	—	—	—	—	0.00156	0.00094	—
Type 310	—	—	—	—	0.00117	0.00077	—
Type 316	—	—	—	0.0000	—	—	—
Type 321	—	—	—	—	0.00111	0.00056	—
Type 347	—	—	—	0.0004	0.00109	0.00068	—
Type 446	—	—	—	—	0.00146	0.00072	—
Inconel	—	—	—	0.0000	0.00153	0.00151	—
Carpenter 20	—	—	—	—	0.00097	0.00059	—
"Hastelloy" B	0.00011	0.000003	—	—	—	—	—
Monel	—	—	—	0.0001	—	—	—
Bronze	0.00006	0.00008	0.0001	—	—	—	—
Phosphorized Admiralty	0.00006	0.00005	0.0001	—	—	—	—
Copper	—	—	—	0.03	—	—	—
Nickel	—	—	—	—	—	—	0.00025

Corrosion Rate, Inches Penetration Per Month

18

Table 7. Resistance of Steels to Molten High Temperature Eutectic Salt at 900 and 1100°F

Quality and Cast No.	Testing Time in Hours	900°F - 482°C		1100°F - 593°C	
		Loss in Wt.[a] MGMS/CM^2	Extrapolated[b] Penetration at 1 year	Loss in Wt.[a] MGMS/CM^2	Extrapolated[b] Penetration at 1 year
MILD STEEL	24	2.0		21.4	
	100	3.6	7.1	39.6 31.7	18.4
	500	21.0		-	
ESSHETE CML	24	2.9		9.8	
	100	3.3	2.4	14.0 9.2	6.4
	500	9.2		-	
ESSHETE CRM.2	24	2.0		9.2	
	100	3.1	2.74	13.9 10.4	5.9
	500	11.0		29.3 18.5	
ESSHETE CRM.5	24	1.5		4.1	
	100	2.4	2.3	8.0 6.3	3.6
	500	9.3		17.2 14.7	
RED FOX (Type 321 SS)	24	0.3		2.7	
	100	0.3	0.5	4.0	2.3
	500	1.9		10.1 7.4	
SILVER FOX (Type 316 SS)	24	NIL		2.2	
	100	NIL	0.03	3.5	0.60
	500	0.1		4.8 3.8	

[a]The loss in weight was determined after descaling in sodium hydride.

[b]Assumes adherence to parabolic rate law.

term data presented are consistent with parabolic rate law behavior — mils penetration = k $\sqrt{t}$; the extrapolated penetrations at one year are calculated from the data using this expression. The alloy compositions tested are given in Table 8.

The "Hitec" pamphlet[13] (page 10) indicates copper is useable at moderate temperatures (to 600°F), but corrodes rapidly at higher temperatures — 30 mils/mo at 1000°F, see Table 6. Rottenberg[3] indicates, "Copper, aluminum, magnesium, and their alloys are all attacked more or less rapidly and should not be allowed to come into contact with HTS." (See additional comment on aluminum and magnesium under Safety Precautions.)

The presence of sulfate is said to be deleterious to the low alloy steels like CML and CRM2 (see Table 8), and Rottenberg[3] states, "the corrosive action of HTS is greatly affected by the presence of oxide and hydroxide." Insolubles, like carbonate, may lead to erosion or localized overheating problems.[16] The oxide and hydroxide effects would require investigation since temperatures of interest in MSBR's presumably would produce appreciable oxide which would be converted to hydroxide by any steam leakage.

Table 8. Analysis and Heat Treatment of Materials

Quality and Cast No.	Analysis Per Cent							Original Heat Treatment
	C	Mn	Si	Ni	Cr	Mo	Ti	
Mild Steel	0.116	0.44	0.24					700°C 1 Hr. AC
ESSHETE CML	0.074	0.53	0.54		0.92	0.52		920°C 1 Hr. FC
ESSHETE CRM.2	0.114	0.48	0.24		2.26	1.01		920°C 1 Hr. FC
ESSHETE CRM.5	0.069	0.48	0.30		5.03	0.56		920°C 1 Hr. FC
RED FOX (Type 321 SS)	0.047	1.73	0.60	8.56	18.44	-	0.42	1050°C 1 Hr. AC
SILVER FOX (Type 316 SS)	0.05	1.66	0.37	11.26	17.28	2.79		1100°C 1 Hr. AC
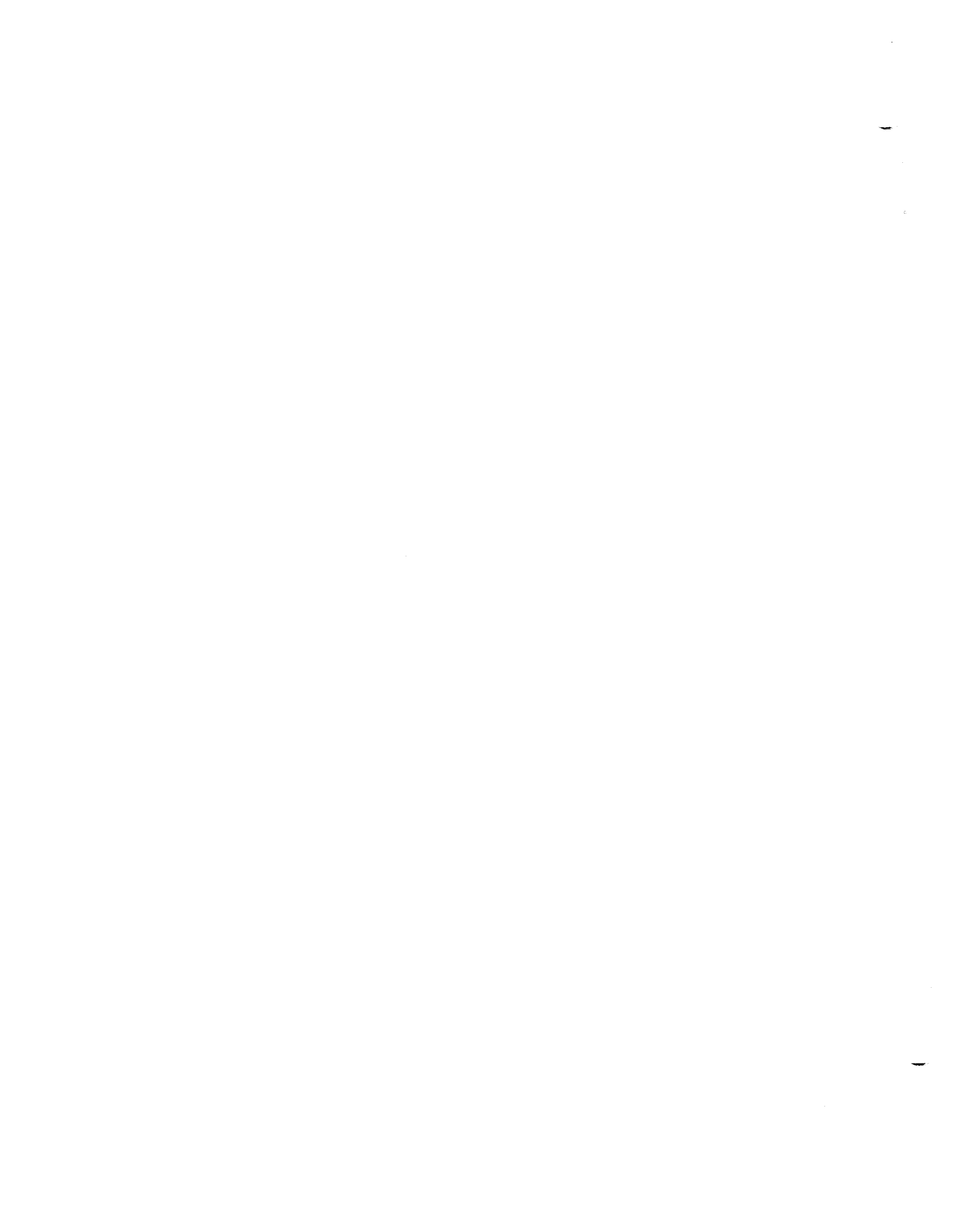
PHYSICAL PROPERTIES

Table 9, compiled by Robertson,[5] compares several salts useable in
MSBR's. Hitec compares favorably with the other two coolant salts.

The remainder of this section, and the subsequent one on Safety
Precautions is taken from the "Hitec" pamphlet.[13] Figure and Table
numbers are those used in the pamphlet.

"Freshly prepared "Hitec" is a white, granular solid; in the molten
state, it has a pale yellow color. It is a eutectic mixture of inorganic
salts having the following composition:

Potassium Nitrate	53%
Sodium Nitrite	40%
Sodium Nitrate	7%

Freezing Point

"The freezing point of fresh "Hitec" is 288°F, and it can therefore
be melted readily by plant steam at a pressure as low as 50 psig. The
presence of a small amount of moisture sharply reduces the freezing point,
and in order to determine the freezing point accurately, it is recommended
that the salt be melted and dried by heating at about 480°F. Driving off
water vapor in this manner may cause effervescence or frothing, depending
on the amount of moisture present, and it is recommended that suitable
precautions be taken to protect personnel from spattering by the hot,
molten salt.

Viscosity

"The viscosity of molten "Hitec" was measured over the temperature
range from 300 to 820°F, and these data were extrapolated to 1000°F with
an accuracy believed sufficient for design purposes. The experimental
points and extrapolated curve are shown in the graph of viscosity versus
temperature, Figure 6.

Electrical Resistivity

"The electrical resistivity of molten "Hitec" is 1.7 ohms per cu.cm.
This value is approximately midway between that of water, 25×10^{6} ohms
per cu.cm., and that of mercury, 95.9×10^{-6} ohms per cu.cm.

Table 9. Selected Properties of the MSBR Molten Salts[5]

	$^7LiF-BeF_2-ThF_4-UF_4$	$NaF-NaBF_4$	$^7LiF-BeF_2$	$KNO_3-NaNO_2-NaNO_3$
Composition, mole %	71.7-16-12-0.3	92-8	66-34	44.2-48.9-6.9[a]
Molecular weight, approximate	64	104	33	84
Density, lb/ft^3 at 1000°F	212	117	124	105
Viscosity, lb/ft-hr at 1000°F	41	3	29	3
Specific heat, Btu/lb-°F	0.32	0.36	0.57	0.37
Thermal conductivity, Btu/ft-hr-°F	0.67 to 0.68	0.23	0.58	0.33
Estimated cost, $/lb	57.00	0.50	12.00	0.15
Circulation required per loop[b] for 556-MW(t) heat load:				
lb/hr	23.4×10^6	18.3×10^6	13.3×10^6	14.7×10^6
gpm	14,260	19,500[c]	13,380	17,370
Liquidus temperature, °F	930	725	850	288

[a]Eutectic composition.

[b]Based on properties at average temperatures in MSBR system.

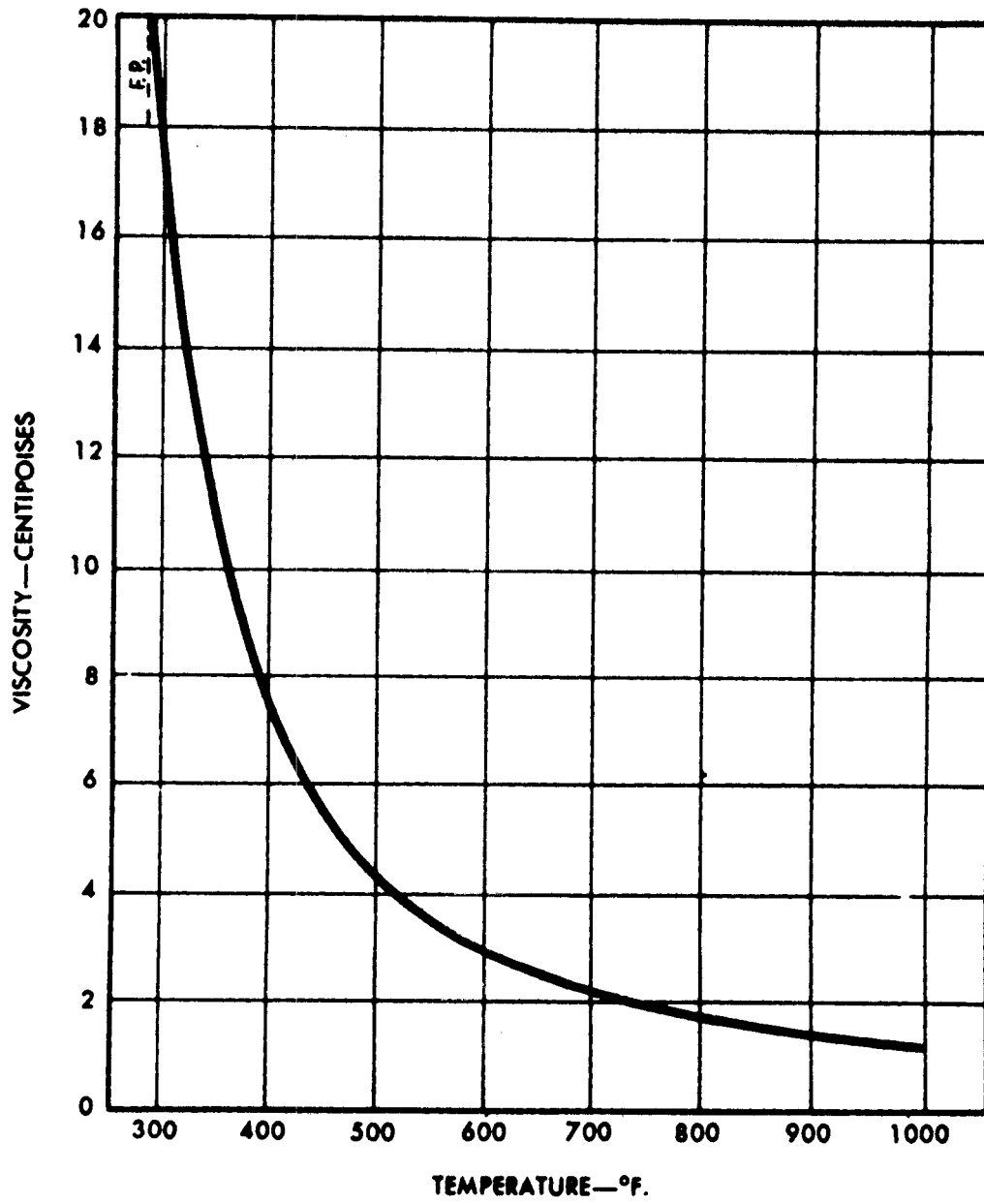[c]Based on 250°F Δt in modified MSBR.

24

Fig. 6.   Viscosity of Liquid HITEC

Density

"The density-temperature relationship for molten "Hitec" is presented
in the alignment chart of Figure 7. By drawing a straight line from the
desired point on the temperature scale through the reference point, density
can be read from the chart in grams per cu.cm., pounds per cu.ft., or
pounds per U.S. gallon.

Thermal Conductivity

"Thermal conductivity measurements on "Hitec" heat transfer salt have
been reported by W. D. Powers, ANP Quarterly Progress Report, September 30,
1957, ORNL-2387 (Classified). A value of 0.35 B.T.U./(hr.) (sq.ft.)
(°F./ft.) independent of temperature in the range from 468 to 689°F was
obtained using a parallel-plate, variable sample gap apparatus such as
that described in ASME paper 56-SA-31 by C. F. Lucks and H. W. Deem.
Battelle Memorial Institute, in a similar apparatus, extended the tem-
perature range and obtained a value of 0.33 B.T.U./(hr.) (°F./ft.)
independent of temperature in the range from 400 to 900°F. (private
communication from H. W. Hoffman, Oak Ridge National Laboratory and
Battelle Memorial Institute).

"An earlier Russian article by Vargaftik, Neimark, and Oleshchuck
which appeared in the Bulletin of the V.T.I., September 1952, No. 9 had
reported values in the temperature range from 302 to 932°F which ranged
from 0.26 to 0.17 B.T.U./(hr.) (sq.ft.) (°F./ft.).

Heat Transfer Coefficient

"Heat transfer coefficients (h) for "Hitec" flowing in turbulent
motion inside horizontal circular, iron pipes have been determined over
the temperature range of 580 to 960°F at linear velocities up to 6 feet
per second. Details of this experimental work are published in
Transactions of American Institute of Chemical Engineers 36, 371 (1940).
Data on the thermal conductivity of "Hitec" were lacking, and the experi-
mental results, therefore, were correlated in terms of the function
$\frac{hD}{\mu^{0.4}}$. Since the publication of this work, additional determinations of
the heat transfer coefficient were reported by H. W. Hoffman and S. I.
Cohen in a paper entitled Fused Salt Heat Transfer — Part III: Forced-

TEMPERATURE

°F.   °C.

DENSITY

#/FT.³   #/U.S. GAL.

DENSITY

GMS./C.C.
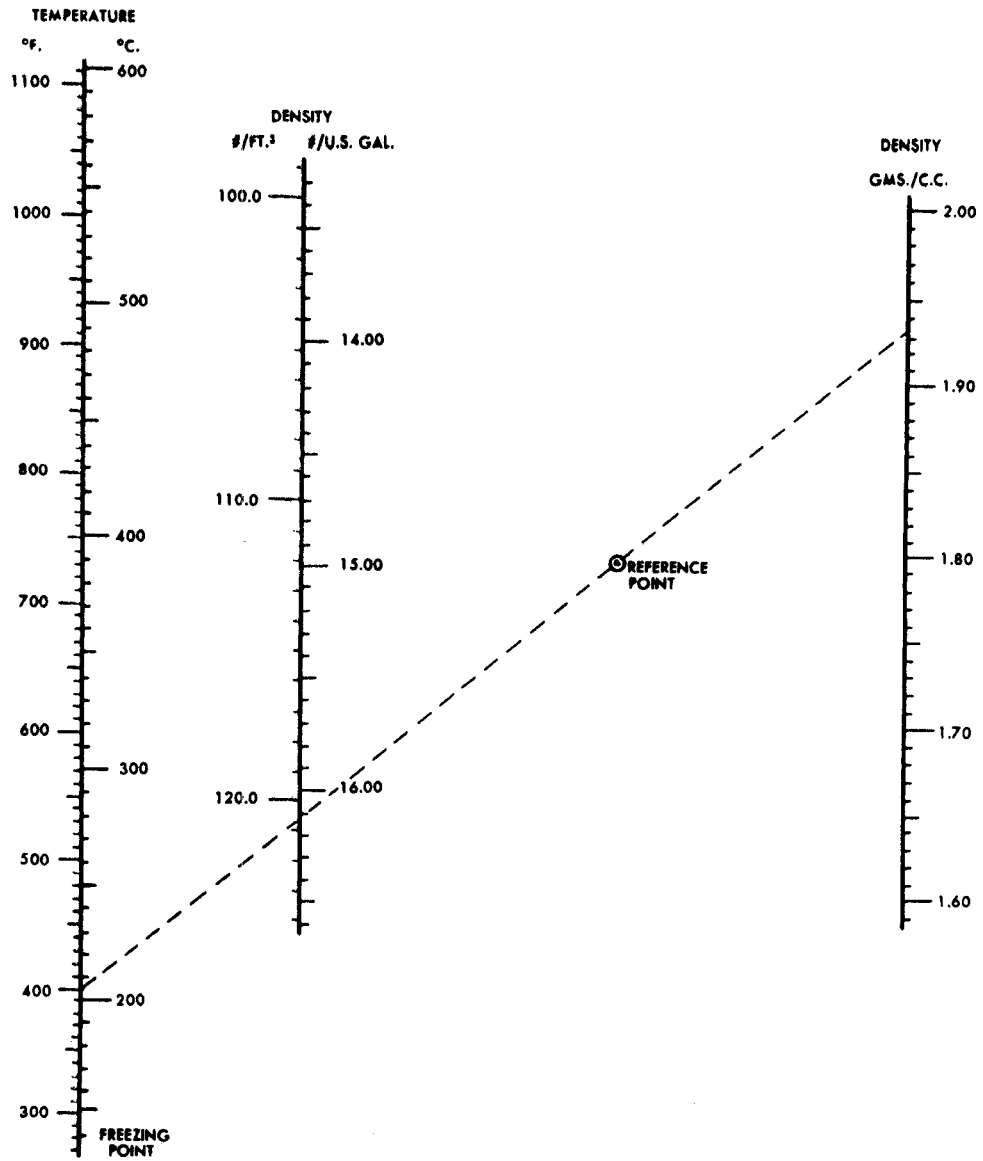
NOTE: TO FIND DENSITY, DRAW A STRAIGHT LINE FROM DESIRED POINT
ON TEMPERATURE SCALE THROUGH THE REFERENCE POINT.

Fig. 7. Density of Liquid HITEC

Convection Heat Transfer in Circular Tubes Containing the Salt Mixture
$NaNO_2-NaNO_3-KNO_3$, ORNL-2433 (1957). The latter work apparently utilized
a more accurate apparatus than that used in the earlier determinations.
The data was correlated using the Colburn j function, $\left(\dfrac{h}{CpG}\right)\left(\dfrac{Cp\mu}{k}\right)^{2/3}$.

Hoffman and Cohen report that the variables ranged as follows:

Reynolds modulus (N_{Re}), $\dfrac{DG}{\mu}$		4850-24,710
Prandtl modulus (N_{Pr}), $\dfrac{Cp\mu}{k}$		4.2-9.1
Average fluid temperature		552-828°F.
Heat flux		62,800-194,500 B.T.U./hr.-ft.^{2}

Under these conditions, the heat transfer coefficient varied from 800 to
2900 B.T.U./(hr.) (sq.ft.) (°F). In heat transfer applications, "Hitec"
behaves as a normal fluid and the standard correlations may be used in
designing heat exchange equipment.

Thermal Expansion

"The coefficient of linear thermal expansion for solid "Hitec",
measured by the Dilatometer method of ASTM-D696-44, is 2.85×10^{-5}
per °F. The coefficient of cubical thermal expansion for liquid "Hitec"
is 2.016×10^{-4} per °F. (private communication from H. W. Hoffman, Oak
Ridge National Laboratory).

Specific and Latent Heat

"The total heat of "Hitec" was measured calorimetrically over the
temperature range of 70 to 1100°F. The slopes of the total heat-
temperature lines, Figure 8, indicated that the specific heat of solid
"Hitec" is 0.32 and that of the melt 0.373 calories per gram per °C
(or B.T.U. per pound per °F). Latent heat of fusion appeared to be
approximately 20 calories per gram (35 B.T.U. per pound).

Efficiency

"The efficiency of "Hitec" heat transfer salt is well illustrated by
a direct comparison to hot air, or flue gas, on a performance basis.

Such a comparison is given in Table I, and the details of the calcu-
lations used to obtain these data are given in the previously cited
article appearing in Transactions of American Institute of Chemical
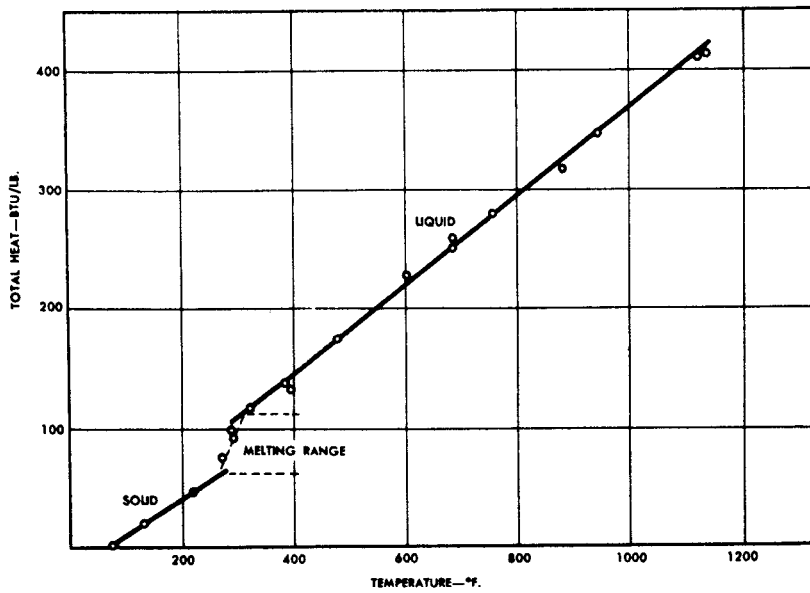Engineers 36, 371 (1940).

Fig. 8

TABLE I

COMPARISON OF "HITEC" AND AIR AS HEAT TRANSFER FLUIDS

Basis of Comparison:
Fluid Conduit: 1" I.D. Tubes
Average Fluid Temperature: 800°F.
Pressure Drop due to Friction: 3.5 lbs./(sq.in.)/100 ft. of tubing
Difference between inlet and outlet fluid temperatures: 50°F.

	"HITEC"	AIR	
		at 3 atm.press.	at 10 atm.press.
Press. drop./100 ft.	3.5 lbs./sq.in.	3.5 lbs./sq.in.	3.5 lbs./sq.in.
Lbs./hr./tube	6000	185	360
Heat carrrying capacity/tube	111,900 B.T.U./hr.	2310 B.T.U./hr.	4500 B.T.U./hr.
No. of 1" tubes required to transfer 111,900 B.T.U./hr. at 50°F. temperature difference	1	48½	25
Total lbs./hr.	6000	8970	8970
Theoretical HP required to overcome friction	0.014	24.1	7.2
Individual coefficient of heat transfer	858	28	40

SAFETY PRECAUTIONS

Principal Hazards

"The principal hazards associated with "Hitec" are those which apply
to any operation involving the use of liquids at elevated temperatures.
Adequate precautions should be taken to protect operating personnel from
burns in case of equipment failure. There is a lesser danger that "Hitec,"
by supporting the combustion of other materials, may escalate an existing
fire hazard. Similarly, there is a danger that prolonged contact of
"Hitec" with human skin may cause dermatitis. Finally, "Hitec" should
not be ingested by operating personnel in more than trace amounts because
of the sodium nitrite which it contains.

"'Hitec' itself liberates no toxic vapors, but adequate ventilation
should be supplied to remove products of combustion due to accidental or
process contamination of the "Hitec" system. Although the combustion
products of most organic compounds are principally carbon dioxide and
water, traces of highly toxic gases such as carbon monoxide, oxides of
nitrogen, etc., may also be present. It is reported that, in instances
where a process utilizing "Hitec" generates steam, operating personnel
complain of a salty taste after being in the area for a period of time,
particularly when the weather is humid. Adequate ventilation will relieve
this condition.

Combustion Hazards

"Although "Hitec" itself is nonflammable, it will support the
combustion of other materials, and salt units should not be located in
the vicinity of wooden construction. Similarly, contamination of the
work area with "Hitec" may increase any fire hazard created by the
presence of combustible materials. These problems may be minimized by
washing the work area regularly with water and by substituting non-
flammable materials for combustible materials. For example, sand rather
than sawdust should be used for clean-up, diking, etc. A number of tests
have been carried out to estimate the hazards associated with the
accidental contact of flammable materials with hot, molten "Hitec," but

since it is impossible to anticipate all conditions under which this
product may be used, it is recommended that the user carry out additional
tests before undertaking any novel applications.

"Combustible vapors generally react relatively slowly with "Hitec" and
escape before combustion is complete. Gasoline vapors, high in unsaturated
compounds, were bubbled through the molten salt for two hours at 1100°F,
and over 90% of the gasoline was recovered unchanged. The reaction between
"Hitec" and ammonia was also studied under similar conditions, and less
than 50% of the ammonia reacted with the "Hitec" over a two-hour period
at 1100°F. In both cases, changes in the salt were minor.

"Flammable liquids tend to vaporize rapidly in contact with molten
"Hitec" and burn at the surface in the same manner as they might be
ignited on any hot surface. Crude oil, for example, burned in a similar
manner on the surface of "Hitec" at 1150°F. and on the surface of molten
lead at the same temperature. Motor gasoline, cracked gasoline, gas oil,
and crude oil mixed with sulfur were released below the surface of an
open container of "Hitec" at 1100°F. In all cases, the hydrocarbons
vaporized and burned, and in the cases of crude oil and gasoline, there
was considerable spattering of hot salt due to the rapid, sub-surface
vaporization of the liquids. Again, there was little change in the salt
composition, indicating that the principal reactions were between the
vaporized hydrocarbons and the air above the salt bath.

"Combustible solids, such as wood, coke, paper, plastics, cyanides,
chlorates, ammonium salts, and active metals, such as aluminum and
magnesium, offer somewhat greater hazards. Magnesium, except as an
alloying agent in low concentration, must not come in contact with "Hitec."
Heat treatment of aluminum parts in molten "Hitec" is a common and safe
practice when suitable precautions are taken, as discussed in "Potential
Hazards in Molten Salt Baths for Heat Treatment of Metals," Research
Report No. 2, The National Board of Fire Underwriters (1946), 85 John
Street, New York 7, New York. Thus, we strongly recommend that before
attempting to use "Hitec" in this type application the potential hazards
as discussed in the reference article be reviewed.

"In these cases it is possible for the solid combustible materials to
remain in contact with the hot salt until they are completely oxidized,

and large volumes of combustion gases can be formed rapidly beneath the salt surface. This could, of course, result in rupture of equipment or violent spattering of hot salt.

"Violent combustion resulted when ground petroleum coke was held beneath the surface of "Hitec" at 1150°F., and in contact with iron, a thermite-like reaction took place. Similarly, mixtures of fine aluminum and iron filings reacted with "Hitec" at 1000°F. with the evolution of enough heat to burn through a nickel crucible.

"Finely divided charcoal flared up and burned vigorously when placed on the surface of "Hitec" at 1200°F., but grease burned quietly under the same conditions. When an oxygen-balanced mixture of "Hitec" and paraffin was heated rapidly to 1100°F., the paraffin merely volatilized and burned, leaving the salt practically unchanged. However, an 80/20 mixture of "Hitec" and nylon burned vigorously at 750°F., and subsequent analysis indicated the oxidizing salts had taken part in this reaction, as they had in the case of the coke held beneath the salt surface.

"All attempts to detonate "Hitec" or mixtures of "Hitec" with combustible products, such as paraffin, petroleum, nylon, and topped Houdry condensate, at 375°F. and 25 inches of mercury vacuum, were unsuccessful using either a No. 8 blasting cap or 0.5 lb. of blasting gelatin as the initiator. The salt was similarly insensitive at 1100°F. Attempts to detonate oxygen-balanced mixtures of "Hitec" and either finely divided charcoal or topped Houdry concentrate by heating the mixtures vigorously under confinement were also unsuccessful. It was estimated that pressures as high as 18,000 psi were attained during these tests.

"Water from spray sprinklers or low-velocity fog nozzles will not penetrate the surface of molten "Hitec" sufficiently to cause dangerous spattering, and this type of fire protection is recommended for oil-fired units and for processes involving combustible materials. Carbon dioxide and approved dry-powder type fire extinguishers can be used satisfactorily to extinguish fires in the vicinity of a salt unit, but vaporizing liquid (carbon tetrachloride), foam, and aqueous types, other than sprinklers or low-velocity fog types, should not be used. An adequate supply of clean, dry sand is useful for slagging and diking to confine the spread of escaped molten salt.

"If, for some reason, water is introduced into a unit containing "Hitec," the heat input should be kept low until all the water has been evaporated, and the system should be vented adequately in order to release the steam which is formed."

APPENDIX A

November 4, 1970

To: W. R. Grimes

From: S. Cantor

Subject: The Reaction, $H_2(g) + (Na,K)NO_3(\ell) \rightleftarrows (Na,K)NO_2(\ell) + H_2O(g)$
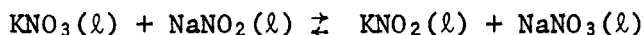
    I concur with the conclusion of Ed Bohlmann, namely, that the equilibrium, given above can prevent tritium from reaching the feed water and steam of a molten-salt reactor.

    The basic data and considerations are as follows:
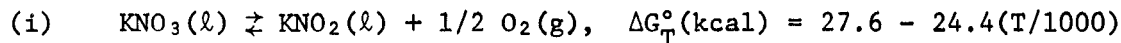
Nominal Composition of HTS

      KNO_3 - $NaNO_2$ - $NaNO_3$ 53 - 40 - 7 weight %
                                 49 - 44 - 7 mole %

HTS is a reciprocal system:

      $KNO_3(\ell) + NaNO_2(\ell) \rightleftarrows KNO_2(\ell) + NaNO_3(\ell)$
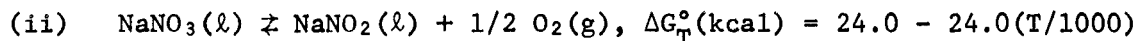
Assuming that nitrate ion is larger than nitrite ion, this equilibrium is shifted somewhat to the left (the "large-large, small-small" rule in reciprocal systems). The ratio of activities, KNO_3/KNO_2 and $NaNO_2/NaNO_3$ would, therefore, be greater than unity.
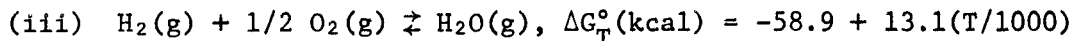
    Three equilibria to consider are:

(i) $KNO_3(\ell) \rightleftarrows KNO_2(\ell) + 1/2\ O_2(g)$, $\Delta G_T^\circ(kcal) = 27.6 - 24.4(T/1000)$

        Ref. - R. F. Bartholomew, J. Phys. Chem. <u>70</u>, 3442 (1966).

(ii) $NaNO_3(\ell) \rightleftarrows NaNO_2(\ell) + 1/2\ O_2(g)$, $\Delta G_T^\circ(kcal) = 24.0 - 24.0(T/1000)$
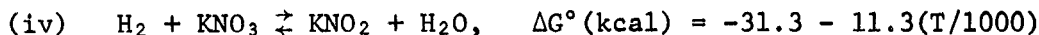
        Ref. - E. S. Freeman, J. Phys. Chem. <u>60</u>, 1487 (1956).
        Freeman's data reworked by L. E. Gastwirt and E. F. Johnson,
        MATT-98, Aug. 1961, p. 157.

(iii) $H_2(g) + 1/2\ O_2(g) \rightleftarrows H_2O(g)$, $\Delta G_T^\circ(kcal) = -58.9 + 13.1(T/1000)$

        Ref. - O. Kubaschewski, E. L. Evans, and C. B. Alcock,
        <u>Metallurgical Thermochemistry</u>, 4th Edition (1967), p. 424.

Combining (i) and (iii),

(iv) $H_2 + KNO_3 \rightleftarrows KNO_2 + H_2O$, $\Delta G^\circ(kcal) = -31.3 - 11.3(T/1000)$

at 873°K, $K = 2.06 \times 10^{10} = \dfrac{P_{H_2O}\ a_{KNO_2}}{P_{H_2}\ a_{KNO_3}}$.

Guessing that $a_{KNO_2}/a_{KNO_3} = 0.1$, then $P_{H_2} = 5 \times 10^{-12}\ P_{H_2O}$.

Assuming that P_{H_2} should be less than 10^{-13} atm, then $P_{H_2O} \leq .02$ atm is tolerable.

Similarly when we combine (ii) and (iii),

(v) $H_2 + NaNO_3 \rightleftarrows NaNO_2 + H_2O$, $\Delta G°(kcal) = -34.9 - 10.9(T/1000)$

also at 873°K, $K = 1.30 \times 10^{11} = \dfrac{p_{H_2O}\ a_{NaNO_2}}{p_{H_2}\ a_{NaNO_3}}$

Now assuming that $a_{NaNO_2}/a_{NaNO_3} = 7$ (the mole fraction ratio when HTS is initially constituted), then

$$p_{H_2} = 5.4 \times 10^{-11}\ p_{H_2O}$$

Thus at 873°K, equilibrium (v) rather than (iv) fixes the tolerable p_{H_2O}. Again, supposing that p_{H_2} should be less than 10^{-13} atm, then p_{H_2O} should be maintained at less than 2×10^{-3} atm.

Finally, please note that the equilibrium constant for reaction (v) increases with decreasing temperature. For instance, at 800°K, the equilibrium constant = 8.25×10^{11}, which for $a_{NaNO_2}/a_{NaNO_3} = 7$, means that the tolerable p_{H_2O} is a factor of 6 higher than at 873°K.

S. Cantor

SC:mp

cc: E. G. Bohlmann

APPENDIX B

Reactions of HITEC with Graphite and with 72-16-12 (1iF-BeF$_2$-ThF$_4$) Salt

C. E. Bamberger and R. G. Ross[*]

A batch of 50 g of HITEC was prepared by melting the analytical grade components (NaNO$_2$, NaNO$_3$, KNO$_3$) in a quartz tube under argon. After melting, argon was bubbled through the liquid for 15 minutes (in order to remove any significant amount of moisture); the melt was cooled under flowing argon and stored in a dessicator.

In the first experiment a 10 g portion of HITEC was melted in an open quartz tube giving a liquid layer of 3/4" depth. A piece of 4.5 g of "MSRE" graphite hanging from a platinum wire was dipped briefly (30 to 120 sec) into the melt at successive increasing temperatures and the visible signs of reaction were noted. Following is a listing of the observations. It made no difference whether the quartz tube was open to the atmosphere or was blanketed by flowing argon.

No graphite introduced

340°C: Very small bubbles formed in the bulk of the melt which on raising did not seem to disturb the surface of the liquid. The bubbles were so small that they were observed only by means of a cathetometer.

Graphite in the Melt

340°C: Large bubbles formed at the interface.

440°C: Faster evolution of large bubbles at the interface, the melt remained clear yellow.

490°C: The melt became black 10-20 sec. after the graphite was introduced. The formation of bubbles was more vigorous than before. After removing the graphite the melt cleared up in ca. 5 minutes.
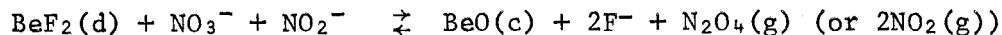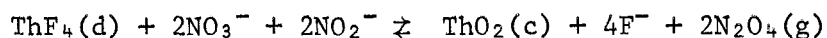

545°C: The melt became black again, then the melt started foaming
vigorously and after approximately 45 seconds the graphite piece
became suddenly incandescent. At this point a sharp rise of tem-
perature was recorded. The graphite was removed and after approxi-
mately 10 minutes the melt cleared up again. The experiment was
terminated.

The experiment was repeated and the reactions were recorded on 16 mm
movie film, the only difference being that the melt was heated rapidly to
440°C and then to 550°C. The observations were the same as above but the
time required for the incandescence of the graphite was ca. 2 minutes.
After removal and cooling the graphite used in both experiments was
washed and examined visually; it revealed a high degree of pitting and
enhancement of the cracks that were initially present.

In a third experiment, approximately 9 g each of HITEC and 72-16-12
$(LiF-BeF_2-ThF_4)$ salt were loaded into a quartz tube (the HITEC at the
bottom) and were gradually heated. At 250°C the molten HITEC bubbled
at the interface with the lumps of fluoride salt and became cloudy-white,
then completely opaque. In the range 350-410°C a large amount of yellow-
brownish gas was evolved. The lumps of salt were no longer visible. At
430°C vigorous foaming occurred which displaced liquid and suspended
material upwards. The experiment was terminated. Upon cooling, the
solids were digested in hot water and repeatedly washed prior to
analyzing the residue by x-ray diffraction. The results indicated the
presence of ThO_2 (major phase), BeO and LiF. (A few unidentified lines
were also present.)

This could be interpreted to be the result of reactions such as

$$ThF_4(d) + 2NO_3^- + 2NO_2^- \rightleftarrows ThO_2(c) + 4F^- + 2N_2O_4(g)$$

$$BeF_2(d) + NO_3^- + NO_2^- \rightleftarrows BeO(c) + 2F^- + N_2O_4(g) \text{ (or } 2NO_2(g))$$

From what is known of the oxide chemistry of U(IV) there is no doubt
that UO_2 would be the first insoluble phase to form if fuel salt is
contacted by HITEC.

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