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GAMMA IRRADIATION OF NITRATE-BASED SALTS

by

**S. R. Breon, N. R. Chellow,
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ABSTRACT

Nitrate-based molten salts offer a number of advantages as coolants for fusion reactors. Although molten salts have been used extensively in the chemical and petroleum industries for many years, their radiolytic stability is virtually unknown. An experiment was devised to determine the radiolytic stability of two commercially available candidate salts--Hitec and Draw Temp 430. The salts were exposed to 0.8×10^9 R of gamma radiation in the Co⁶⁰ facility at the Argonne National Laboratory and simultaneously heated to temperatures in excess of 530°C. A helium gas stream circulated over the salts was analyzed for decomposition products. It was found that there was no observable thermal or radiolytic decomposition of either salt. Although the exposure was equivalent to only about 1 minute in a controlled thermonuclear reactor, the results were very encouraging and suggest that further experimentation on molten nitrate-based salts is warranted.

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1.0 INTRODUCTION

Molten salts possess many characteristics that make them highly desirable as coolants for fusion reactors.^{1,2} These characteristics include good thermal conductivity and excellent heat-transfer properties. In addition, molten salts have high electrical resistivity which minimizes the effects of magnetohydrodynamic (MHD) forces. Nitrate-based salts, in particular, are promising due to their low melting points, low cost, and compatibility with common structural materials.^{1,2} These salts have been used successfully in the chemical and petroleum industries.³⁻⁶

There are, however, some potential problems associated with the use of these salts in fusion environments. Despite their high electrical resistivities, the salts might undergo EMF-induced electrolysis while circulating through strong magnetic fields. Cantor and Grimes² addressed this problem and concluded that the effects can be minimized by careful design of flow geometries. At high temperatures (>770 K), nitrates are unstable and could spontaneously decompose (e.g., via the reaction $2\text{KNO}_3 \rightarrow \text{K}_2\text{O} + \text{N}_2 + 2\frac{1}{2}\text{O}_2$).

Nitrate-based salts are also very strong oxidizers, and in the presence of a reducing agent, they could detonate. However, in the absence of a reducing agent, reaction rates are very low. Extensive industrial use indicates that these salts can be handled safely under ordinary conditions.³⁻⁶ It is conceivable, though, that the energy supplied by the radiation in a fusion environment could increase the decomposition rates sufficiently to make nitrate-based molten salts difficult, if not hazardous, to work with. Severe radiation-induced decomposition would tend to exclude these salts from further consideration as fusion reactor coolants.

The purpose of this experiment was to examine the effect of radiation on candidate nitrate-based salts. Two salts--Hitec, manufactured by E. I. Du Pont, and Draw Temp 430, manufactured by E. F. Houghton & Company--were selected for exposure to gamma rays from a ^{60}Co source.

2.0 EXPERIMENTAL

Samples of Draw Temp 430 (hereinafter referred to as "Draw Salt") and Hitec were obtained from P. F. Tortorelli at the Oak Ridge National Laboratory (ORNL). Properties of these salts are given in Table 1. Specimens of each salt were crushed and loaded into separate sample containers (see Fig. 1). Loading was done in a helium-filled glove box to minimize exposure of the salt to air. The containers were designed so that a stream of helium sweep gas could enter into the space directly above the sample and then exit through an outlet line. In this way, any gases produced by decomposition would be carried along with the sweep gas to a mass spectrometer where they could be detected. Resistance heating tape was wrapped around the bottom 16 cm of the containers and the sample temperature was controlled by means of a variac.

Once the lower section of each container was filled approximately halfway, the salt was melted and then allowed to solidify into a single piece. After this, the containers were connected to a sweep-gas circulation and monitoring loop (see Fig. 2), and were continuously purged with helium. The gas loop was checked daily to ensure that flow was, indeed, being maintained.

All equipment, with the exception of the sample containers and their associated piping and wiring, was located outside the ^{60}Co cell. This was done to allow manual operation of the valves and enable direct instrument readings and control. The gas loop could be operated in a variety of modes:

- The entire system could be purged;
- One sample could be purged while the other sample was held in closed-loop circulation;
- One sample could be tested, while the other sample was held in closed-loop circulation;
- Both samples could be held in closed-loop circulation, while the lines were purged to the leak-valve that interconnected with the mass spectrometer;
- One or both samples could be backfilled to replace helium lost through small leaks in the system.

Table 1. Properties of the Nitrate-Based Salts Hitec and Draw Temp 430

Property ^{4,5}	Hitec ^R	Draw Temp ^R 430
Manufacturer	E. I. Du Pont	E. F. Houghton & Co.
Composition, wt %	53 KNO ₃ -40 NaNO ₂ -7 NaNO ₃	50 KNO ₃ -50 NaNO ₃
Melting point, °C	143	221
Electrical resistivity, ohm/cm	1.7	---
Thermal conductivity, W/m-°C	0.57	---
Coefficient of linear thermal expansion for liquid, °C ⁻¹	5.13 x 10 ⁻⁵	---
Coefficient of cubical thermal expansion for liquid, °C ⁻¹	3.62 x 10 ⁻⁴	---
Specific heat of solid, cal/g-°C	0.32	---
Specific heat of liquid, cal/g-°C	0.373	0.38
Latent heat of liquid, cal/g-°C	20	---
Density at 427°C, g/cm ³	1.77	1.84
Decomposition point, °C	593	649

Fig. 1. Sample Container.

Fig. 2. Sweep-Gas Circulation and Monitoring Loop.

The pressure transducers were used to monitor pressure surges which might occur due to sudden decomposition of the salts. For this eventuality, the pressure relief valves were set to open at about 10 psig. The system was operated at about 5 psig; no pressure surges occurred.

The leak valve could be opened or closed to allow a small portion of the sweep gas to enter the mass spectrometer. Enough gas was allowed to enter to keep the current inside the mass spectrometer at about 1.0 mA. A similar arrangement had been previously used ⁷ with success. Scans were made of the sweep gas for evidence of decomposition (decomposition of the salts should produce nitrogen gas). These scans were taken under five conditions:

1. No sample being swept by the helium gas;
2. Sample cold (temperature < melting point) and unirradiated;
3. Sample hot (temperature > melting point) and unirradiated;
4. Sample cold and irradiated;
5. Sample hot and irradiated.

Temperature and irradiation histories for the Hitec and Draw Salt are shown in Figs. 3 and 4, respectively.

There are two gamma rays produced by ⁶⁰Co. One has an energy of 1.332 MeV and the other has an energy of 1.173 MeV. The dose rate was 3×10^6 R/h which produced an integrated dose of $\sim 0.8 \times 10^9$ R. This is approximately equal to 30 seconds in a fusion reactor with a 1.0 MW/m^2 neutron wall loading.⁸

At the conclusion of the experiment, the samples were returned to ORNL for chemical analysis.

Fig. 3. Temperature and γ -irradiation History of Hitec Salt.

Fig. 4. Temperature and γ -irradiation History of Draw Salt.

3.0 RESULTS

Each time a scan of the sweep gas was made, the relevant data were recorded and assigned an identifying number called the Mass Spec Index. The index was marked on the chart paper so that relationships between relative abundance of nitrogen gas, temperature, and dose could be examined at a later time. The complete log of the data (excluding graphs from the mass spectrometer) is given in Appendix A. A representative sample of these data can be found in Table 2.

A set of typical mass spectrometer scans is shown in Figs. 5 and 6. Fig. 5 contains results for Hitec, while Fig. 6 contains results for Draw Salt. The graphs are done at different full-scale voltages and represent the four conditions indicated earlier for a sample that was being swept by the helium gas stream. The scan-related information pertaining to these graphs is summarized in Table 3.

In addition to scans made of the sweep gas, scans were made every morning of the gas present in the mass spectrometer. One such scan, Scan 139, is shown in Fig. 7. The sweep gas was eliminated by closing the leak valve every evening and allowing the ion pump to remove most of the gas from the mass-spectrometer vacuum chamber.

The scans show many interesting features. Helium is the dominant gas in all scans, and is off-scale on any range more sensitive than 2.0 V. Large quantities of hydrogen (atomic and molecular) are present, as well as argon. Smaller amounts of water, nitrogen, carbon dioxide, and a few hydrocarbons are also present. Only traces of O_2 and NO were seen. Since the above gases are present in all scans at approximately the same level, beginning with the first scans where there should have been no decomposition, these gases were assumed to be contaminants in the helium gas stream.

In order to determine whether or not decomposition had occurred, the height of the nitrogen peak was followed. Since no change was detected by visual examination alone, a measure of "relative abundance" of N_2 was made. To do this, the ratio of the N_2 peak height to the H_2O peak height was first determined, most often from a scan with 0.050-V full scale. Then, using a 2.0-V full-scale scan taken at approximately the same time, the ratio of the H_2O peak height to the He peak height was determined. These two ratios were

Table 2. Representative Data Log

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose, 10 ⁹ R
Hitec ↑	7-02-79	10:49	1A	0.500	29	0.0
	7-02-79	15:38	16A	2.000	492	0.0
	7-02-79	15:44	17A	0.200	495	0.0
	7-03-79	14:40	41A	0.050	45	0.0044
	7-05-79	9:23	53A	0.200	30	0.1260
	7-09-79	9:25	95A	2.000	414	0.3944
	7-09-79	9:40	97A	0.050	404	0.3952
	7-10-79	11:07	112A	0.200	494	0.4670
	7-13-79	14:41	151A	0.050	523	0.6152
	7-16-79	9:27	160A	0.050	524	0.8155
Hitec ↓ Draw Salt	7-02-79	10:55	2B	2.000	29	0.0
	7-02-79	11:04	3B	0.500	29	0.0
	7-02-79	16:21	20B	2.000	243	0.0
	7-03-79	10:54	28B	0.200	284	0.0
	7-05-79	9:58	58B	0.050	30	0.1260
	7-06-79	14:45	93B	0.050	452	0.1945
	7-09-79	10:47	98B	0.200	65	0.3985
	7-10-79	10:50	110B	0.200	464	0.4670
	7-13-79	14:27	149B	0.200	569	0.6145
	7-16-79	10:04	166B	0.050	574	0.8173
Draw Salt ↓ Background	7-03-79	9:20	22	0.020	---	-----
	7-09-79	9:08	94	0.050	---	-----
	7-12-79	9:22	139	0.050	---	-----
	7-13-79	14:05	146	0.200	---	-----
Background ↑	7-16-79	8:56	157	0.050	---	-----

Fig. 5. Representative Scans of the Hitec Salt.

Fig . 6. Representative Scans of the Draw Salt.

Table 3. Conditions for the Mass Spectrometer Scans in Figures 5 and 6

Fig. No.	Mass Spec Index	Full-Scale Voltage, V	Sample Condition
5a	16A	2.000	Hot and unirradiated
5b	1A	0.500	Cold and unirradiated
5c	112A	0.200	Hot and irradiated
5d	134A	0.050	Cold and irradiated
6a	20B	2.000	Hot and unirradiated
6b	3B	0.500	Cold and unirradiated
6c	110B	0.200	Hot and irradiated
6d	58B	0.050	Cold and irradiated

Fig. 7. Mass-Spectrometer Internal Scan (Scan 139).

then multiplied to give what was defined as the relative abundance of N_2 :

$$\text{Relative abundance of } N_2 = \left[\frac{N_2 \text{ peak height on 0.050 V scale}}{H_2O \text{ peak height on 0.050 V scale}} \right] \times \left[\frac{H_2O \text{ peak height on 2.0 V scale}}{He \text{ peak height on 2.0 V scale}} \right]$$

The relative abundance was plotted as a function of temperature and dose for both types of salts (Figs. 8-11). In all four figures, the relative abundance of N_2 appears to remain at about 2×10^{-3} . There are some points, however, that run as high as 7×10^{-3} . With all but a few exceptions, the higher data points were collected at the very beginning of the experiment. The presence of a relatively large amount of N_2 at the beginning of an experiment is probably due to air entering the system while the final connections of the sample containers to the gas loop were made, and some of which remained even though the system was thereafter purged with helium. Those higher data points obtained near the end of the experiments (higher integrated γ dose and temperature) have been attributed to air leakage into the system during periods which required adjustment of feed gas to the mass spectrometer leak valve.

From Figs. 8 and 10, one can see that there was no detectable thermal decomposition of either salt. Our results for the Hitec salt are in agreement with thermal decomposition data obtained by Bohlmann (Fig. 12),³ which indicate that more than two weeks of heating at a temperature of 525°C is required to effect measurable salt decomposition.

Figures 9 and 11 indicate that there was no radiolytic decomposition of either salt after an exposure equivalent to about 80 seconds in a fusion reactor with a neutron wall loading of 1.0 MW/m^2 . Again, longer exposure or a higher dose rate may have resulted in detectable levels of decomposition.

Fig. 8. Relative Abundance of N_2 vs. Temperature for γ -irradiation of Hitec.

Fig. 9. Relative Abundance of N_2 vs. Integrated Dose of γ -irradiation of Hitec.

Fig. 10. Relative Abundance of N_2 *vs.* Temperature for γ -irradiation of Draw Salt.

Fig. 11. Relative Abundance of N_2 *vs.* Integrated Dose for γ -irradiation of Draw Salt.

Fig. 12. Nitrite Decomposition in an Industrial Process Loop Using Hitec.
(From reference 3.)

4.0 CONCLUSIONS

- Neither the Hitec nor the Draw Salt showed signs of thermal decomposition. Both were stable even when held at temperatures around 550°C for more than 140 hours. In addition, after exposure to 0.8×10^9 R of gamma radiation, the salts showed no signs of radiolytic instability. This amount of radiation is approximately equal to 80 seconds (on the order of one pass through the coolant system) in a fusion reactor with a neutron wall loading of 1.0 MW/m^2 .
- From the results of these tests, the extent of decomposition of the two salts under fusion reactor operating conditions would be so small that relatively little on-line salt purity adjustment would be necessary; and this adjustment would involve essentially straightforward chemical methods.
- Further tests are indicated to determine the suitability of nitrate-based molten salts as coolants in fusion reactors. Such tests are needed to quantify the thermal stability of these salts. In addition, thermal decomposition as well as radiolytic decomposition may depend on fluence, flux, or both. The salts should be irradiated at a higher flux and to a higher fluence, with values simulating those that would be encountered in a reactor. All these factors will have to be taken into account in subsequent considerations of the use of nitrate-based salts in fusion reactors.

Acknowledgements

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APPENDIX A

Log of Data from the Hitec and Draw-Salt Irradiations

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose (10 ⁹ R)
Hitec ↑	7-2-79 ↑	10:49	1A	0.500	29	0 ↑
		11:28	4A	2.000	100	
		11:32	5A	0.500	108	
		13:11	6A	2.000	115	
		13:35	7A	2.000	160	
		13:43	8A	0.200	168	
		14:02	9A	0.200	199	
		14:26	10A	0.200	305	
		14:33	11A	2.000	312	
		14:49	12A	2.000	406	
		14:58	13A	0.200	417	
		15:06	14A	0.200	419	
		15:10	15A	0.500	420	
		15:38	16A	2.000	492	
		15:44	17A	0.200	495	
	7-2-79 ↓ 7-3-79 ↑	9:56	23A	2.000	492	0 ↓ 0.0002 0.0006 0.0008 0.0038 0.0041 0.0044 0.0060 0.0060 0.0060 0.1260 ↑
		10:06	24A	0.500	489	
		13:16	32A	2.000	36	
		13:24	33A	2.000	40	
		13:28	34A	0.200	42	
		14:28	39A	2.000	44	
		14:34	40A	0.200	45	
		14:40	41A	0.050	45	
		15:36	45A	2.000	398	
		15:43	46A	0.200	409	
	7-3-79 ↓ 7-5-79 ↑	15:48	47A	0.050	412	
		9:16	52A	2.000	30	
		9:23	53A	0.200	30	
		9:30	54A	0.100	30	

APPENDIX A (CONT'D)

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose (10 ⁹ R)
Hitec	7-5-79	9:42	55A	0.500	30	0.1260
		13:38	63A	2.000	37	
		13:43	64A	0.050	37	
		14:15	65A	2.000	190	
		14:23	66A	0.200	203	
		14:39	67A	2.000	293	
		14:45	68A	0.200	340	
		14:50	69A	0.050	351	
		15:08	70A	2.000	373	
		15:15	71A	0.200	376	
		15:27	72A	0.050	380	
		15:40	73A	0.020	371	
		15:48	74A	0.050	282	
		16:00	75A	2.000	234	
		16:07	76A	0.200	228	
	7-6-79	16:13	77A	0.050	226	0.1269
		16:32	80A	0.200	223	
		16:36	81A	0.050	223	
		9:27	83A	2.000	220	
		9:32	84A	0.200	220	
	7-9-79	9:41	85A	0.050	220	0.1793
		14:13	89A	2.000	288	
		14:22	90A	0.200	404	
		9:25	95A	2.000	414	
		9:32	96A	0.200	406	
	7-10-79	9:40	97A	0.050	404	0.3952
		13:33	99A	2.000	400	
		9:27	103A	2.000	400	
		9:32	104A	0.200	404	
		9:56	105A	0.050	403	
		11:01	111A	2.000	489	0.4670

APPENDIX A (CONT'D)

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose (10 ⁹ R)	
<div><div></div><div>Hitec</div><div></div><div>Hitec</div><div>Draw Salt</div><div></div></div>		11:07	112A	0.200	494	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	
		13:39	118A	2.000	524		
		13:44	119A	0.200	526		
		13:50	120A	0.050	526		
	7-10-79	15:02	122A	0.050	522		
	7-11-79	10:40	124A	2.000	529		
		10:47	125A	0.200	524		
		10:59	126A	0.050	526		0.4670
		13:37	132A	2.000	524		0.4680
		13:43	133A	0.200	528		0.4683
		13:48	134A	0.050	29		0.4685
	7-11-79	15:26	135A	2.000	530		0.4734
		15:32	136A	0.200	532		0.4737
	7-12-79	9:31	140A	2.000	538		0.5277
	7-12-79	9:38	141A	0.200	528		0.5280
	7-13-79	14:36	150A	0.200	522		0.6149
		14:41	151A	0.050	523		0.6152
		14:50	152A	2.000	527		0.6156
		14:58	153A	0.200	530		0.6160
	7-13-79	15:03	154A	0.050	530		0.6163
	7-16-79	9:14	158A	2.000	524		0.8148
		9:20	159A	0.200	524		0.8151
		9:27	160A	0.050	524		0.8155
	7-16-79	9:32	161A	0.020	524		0.8157
	7-2-79	10:42	167A	2.000	29		0.0
7-2-79	10:55	2B	2.000	29	0.0		
	11:04	3B	0.500	29			
	16:01	18B	0.200	41			
	16:08	19B	2.000	41			
	16:21	20B	2.000	243			
7-2-79	16:29	21B	0.200	261			

2

[illegible]

APPENDIX A (CONT'D)

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose (10 ³ R)
Draw Salt	7-6-79	14:45	93B	0.050	452	0.1945
	7-9-79	10:47	98B	0.200	65	0.3985
	7-9-79	15:03	100B	2.000	421	0.4086
	7-9-79	15:08	101B	0.200	446	0.4088
	7-10-79	10:04	106B	2.000	450	0.4657
		10:10	107B	0.200	450	0.4660
		10:17	108B	0.050	449	0.4663
		10:42	109B	2.000	456	0.4670
		10:50	110B	0.200	464	
		11:17	113B	2.000	501	
		11:24	114B	0.200	520	
		13:15	115B	2.000	550	
		13:21	116B	0.200	559	
		13:29	117B	0.050	562	
	7-10-79	14:53	121B	0.050	570	
	7-11-79	11:06	127B	2.000	566	
		11:18	128B	0.200	565	
		11:24	129B	0.050	565	0.4670
		13:22	130B	2.000	566	0.4672
		13:28	131B	0.200	567	0.4675
		15:38	137B	2.000	572	0.4740
	7-11-79	15:47	138B	0.200	571	0.4745
	7-12-79	9:45	142B	2.000	572	0.5284
	7-12-79	9:51	143B	0.200	572	0.5287
	7-12-79	9:58	144B	0.050	570	0.5290
	7-13-79	14:27	149B	0.200	569	0.6145
	7-13-79	15:10	155B	2.000	574	0.6166
	7-13-79	15:16	156B	0.050	574	0.6169
	7-16-79	9:38	162B	2.000	570	0.8160
		9:43	163B	0.200	572	0.8163
		9:51	164B	0.200	572	0.8167

APPENDIX A (CONT'D)

Sample	Date	Time	Mass Spec Index	Full Scale Voltage, V	Temperature °C	Integrated Dose (10 ⁹ R)
↓	↓	9:57	165B	0.050	574	0.8170
Draw Salt	7-16-79	10:04	166B	0.050	574	0.8173
Background	7-2-79	10:00	168	2.000	---	-----
↑	7-3-79	9:20	22	0.020	---	-----
	7-5-79	9:05	51	0.050	---	-----
	7-6-79	9:12	82	↑	---	-----
	7-9-79	9:08	94	↑	---	-----
	7-1 -79	9:13	102	↑	---	-----
	7-1 -79	10:26	123	↓	---	-----
	7-12-79	9:22	139	0.050	---	-----
	7-13-79	13:58	145	2.000	---	-----
	↑	14:05	146	0.200	---	-----
	↓	14:11	147	0.050	---	-----
	7-13-79	14:18	148	0.050	---	-----
Background	7-16-79	8:56	157	0.050	---	-----

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