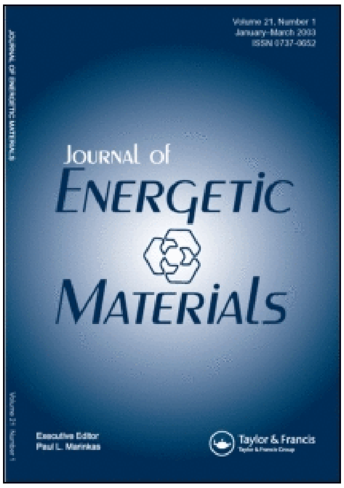

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MOLTEN SALT OXIDATION OF CHEMICAL MUNITIONS

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ABSTRACT

Molten salt oxidation is reviewed as a potential near term alternative technology for the destruction of chemical agents. Initial tests completed by the US Army in 1975 showed the promise of achieving very high removal efficiencies on actual agents. Recent testing by DOE has verified the potential for very low PIC and dioxin or furan releases. To further explore the possible application of this technology to chemical agent destruction, a molten salt reactor and associated equipment was designed to process a nominal 50 kg/h of Sarin. Mass and energy balances are presented for process conditions representing a range of molten salt potential operational modes and schemes for enhancing plant capacity. Process economics are presented.

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INTRODUCTION

Molten salt processes for the destruction of waste chemicals were proposed in the early 1970's and first patented by Yosim(1) in 1974 for halogenated pesticides and other difficult to destroy chemicals at a time when the U. S. was just coming to grips with the need to isolate and protect our environment from such materials. The process provides for introducing the waste materials and air under a liquid sparged bed of alkaline bearing molten salts at a temperature of 800 to 1100 C. A simplified description of the molten salt process is presented in Figure 1. Halogens and other salt forming species such as phosphorus, sulfur, and arsenic are immediately stripped from the wastes by the alkaline salt (usually soda ash) and retained in the salt bed. No off gas acid gas scrubber is required, and the destruction process is completed in a single stage of contacting, thus a secondary off gas combustor is not needed to be confident the exhaust is free of hazardous waste material, or products of incomplete combustion. Under normal conditions of excess air, the only off gas treatment required is filtration of the salt fume which is returned to the molten salt vessel.

MOLTEN SALT TESTS USING CHEMICAL AGENTS

The US Army conducted exploratory bench scale tests(2) of neat chemical agents (GB, VX, HD, and L), the spent salts derived from the

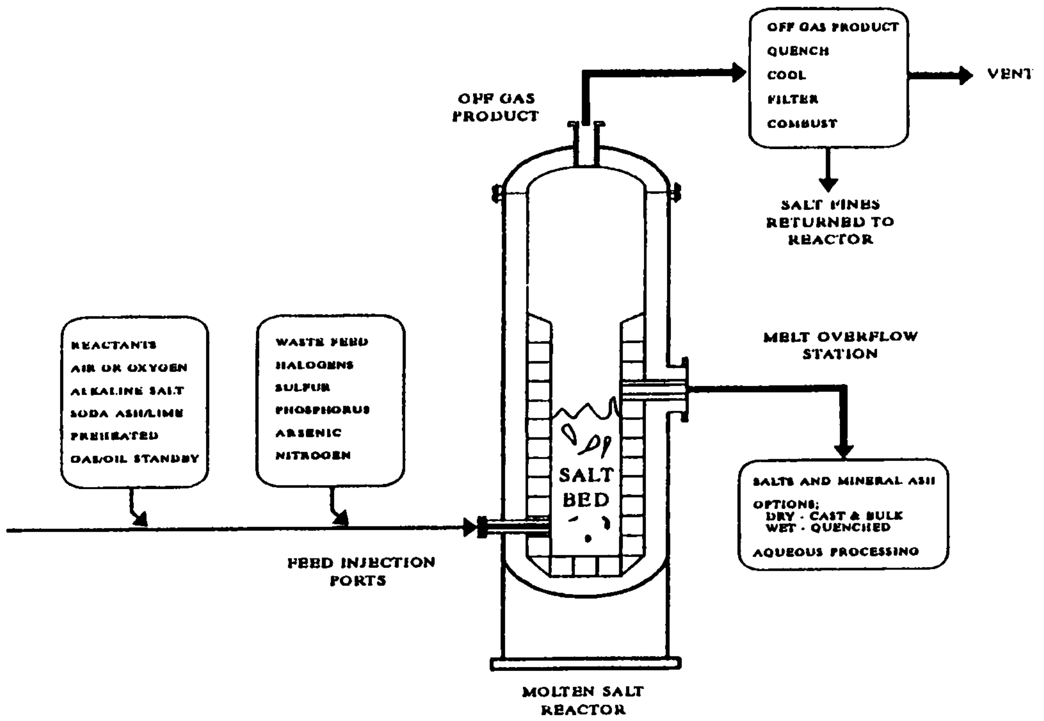

FIGURE 1. Molten Salt Concept

chemical neutralization of GB, and various toxic and war gas identification kits. None of the more than 35 test runs resulted in any measurable amount of agent being detected in the undiluted exhaust coming directly from the bench scale molten salt unit. The detection limits for the tests were the threshold limit values corresponding to the maximum permitted work area concentrations in effect at the time the tests were run, as are presented in Table 1. In theory, the exhaust from the equipment could have been directed into the work area and unmasked personnel would not have been exposed to hazardous concentrations of agent.

Agent	Stack Concentration mg/m3	Work Area Concentration mg/m3	Boundary Concentration mg/m3
Distilled Mustard (HD)	3.00E-02	3.00E-03	3.00E-04
Nitrogen Mustard (HN-1, HN-3)	3.00E-02	3.00E-03	3.00E-03
Lewisite (L)	Not Given	1.50E-03	Not Given
Sarin (GB)	3.00E-04	1.00E-04	3.00E-06
VX	3.00E-05	1.00E-05	3.00E-07
Phosgene (CG)	Not Given	8.00E-02	Not Given
Cyanogen Chloride (CS)	Not Given	5.00E-02	Not Given
Chloropicrin (PS)	Not Given	7.00E-01	Not Given
Chloroacetonphenone (CN)	Not Given	3.00E-01	Not Given
Adamsite (DM)	Not Given	5.00E-01	Not Given

TABLE 1. Detection Limits - Work Area Limits

The destruction removal efficiencies (DRE's) calculated from the run conditions and agent detection limits are shown in Table 2. These are the worst case DRE's calculated for each of the neat agent run series. The results for the GB salts and the identification kits (together with dunnage and activated charcoal) gave similar results.

Agent	Worst Case DRE
Mustard (HD)	>99.999997%
Sarin (GB)	>99.9999985%
VX	>99.999988%
Lewisite (L)	>99.9999958%

TABLE 2. Worst Case DRE's for Neat Agents

The results from these tests are even more remarkable when considering that they were run under conditions that are much less rigorous than those that would exist in a pilot or full scale unit. The gas side residence time for the bench scale apparatus was approximately 1 second (in the melt). Larger reactors with correspondingly deeper beds will have a residence time of 4 to 6 seconds.

RESULTS FROM FULL SCALE TESTS

The only full scale tests of waste destruction conducted to date using molten salt were done for the EPA Office of Research and Development(3) in the summer of 1984. The molten salt reactor used was 85 cm in inside diameter, with an overall height of about 8 m and would be considered to be full scale for many hazardous waste applications, including for chemical agents. Hexachlorobenzene (HCB) and chlordane were the test feed stocks. Hexachlorobenzene (a solid) was fed at rates up to 122 kg/h, and chlordane (a liquid) was fed at rates up to 25 kg/h. The DRE's generally exceeded 99.9999% directly out of the reactor exhaust, with the DRE's calculated from the exhaust stack (undiluted, but down stream of the fume filter) exceeding 9-9's for all of the HCB tests, and 7-9's for all of the chlordane tests. Makeup soda ash was continuously injected with the waste materials during these tests and spent molten salt was tapped out continuously and cast into standard 205 liter steel drums. Since the salt was cast into the drums, the density of the resulting waste pack was approximately twice that of the loose, granular salt; 300 kg/drum versus 140 kg/drum.

A second full scale molten salt demonstration that should be mentioned is the DOE's Molten Salt Coal Gasification Pilot Demonstration Unit(4) operated from 1978 to 1982. Although this project was not a waste destruction application, many of the unit operations and equipment are the same as would be proposed for waste molten salt facilities. The salt reactor for this program was 110 cm in inside diameter and it operated at pressures up to 20 atmospheres. It processed high sulfur coal to a clean low heating value fuel gas and had the capablity for aqueous processing of the spent salt and recycle (closed loop) of the salt sodium value as sodium carbonate.

RECENT TESTS FOR PIC's, DIOXINS AND FURANS

The DOE has an on going interest in the development of molten salt technology for potential application to its mixed radioactive/hazardous chemical wastes. As part of the DOE program, tests were done in 1994 at Oak Ridge National Laboratory(5) to determine dioxin and furan emissions from a bench scale molten salt unit very similar to the unit used previously by the Army. The U. S. Environmental Protection Agency has proposed (currently there is no requirement in the U. S.) a regulatory limit(6) of 0.1 ng/Nm^3 as 2,3,7,8-tetrachlorodibenzo-*para*-dioxin toxic equivalence (as TEQ) for hazardous waste treatment incinerators. This limit has also been adopted by most of the European Community as a standard for dioxin and furan releases. As part of these tests, DRE's and PIC's were determined as well as federal EPA Toxic Chemical Leach Procedure (TCLP) for barium, cadmium, and chrome using stabilized spent salts spiked with heavy metals.

The chlorinated feed materials used were carbon tetrachloride, 1,1,1-trichloroethane, and 2,4-dichlorophenol. The latter is considered to be a potentially strong precursor to the formation of dioxin and furans.

Total dioxins and furans were shown to be very low, less than 0.015 ng/Nm^3. No 2,3,7,8 TCDD (the most hazardous of the dioxins) was measured in any of the tests, even when the detection limit was as low as 1.2 pico grams/Nm^3. DRE's calculated based on the three chlorinated feed materials met or exceeded EPA release limits in all test runs. The only important PIC's observed in the tests were chloromethane, methylene chloride, benzene, and toluene. The levels measured were less than 4 ppb for all tests with the exception of a test that had a serious upset. In that one test, the maximum benzene level increased to 27.4 ppb, the chloromethane to 11 ppb, and the toluene to 2.6 ppb. Even with this increase, the levels were still equal to or below those seen for similar compounds in hazardous waste incinerators. The authors of the test report concluded that "these bench scale studies show sufficient evidence of reliable destruction to make further bench scale studies in this area unnecessary", and suggested that any further emissions testing be done on a pilot scale.

PRESENT STATUS OF MOLTEN SALT TECHNOLOGY

Molten salt waste treatment is currently under development or planned to be under development at the DOE's Lawrence Livermore National Laboratory, and at the Naval Surface Warfare Center, Indian Head, Maryland. The work at LLNL is focused on treating DOE's mixed hazardous, radioactive wastes, and also on high explosives. The work at Indian Head is aimed at the treatment of both explosives and propellants. Testing of propellants and high explosives in molten salt has been extensive(7), but tests have only been done using bench scale equipment. There have not been any commercial applications of the molten salt process to date.

MOLTEN SALT OXIDATION OF SARIN

As can be seen from the above review of, it would appear that molten salt would be a good candidate for demonstration as a near term, implementable, alternative technology to incineration, especially for the large containers of bulk agent, for spent chemical neutralization solutions, and for such materials as agent loaded charcoal and dunnage. A brief analysis was therefore performed to determine the size, operating conditions, and projected costs of a 50 kg/h Sarin molten salt unit that would be of sufficient size to be evaluated at a demonstration facility, and possibly even be prototypical of full sized units that could be located at chemical munitions storage/treatment sites.

The overall basic chemistry for the reaction of Sarin with sodium carbonate and oxygen is given by equation (1). The destruction of the waste

$$C_4H_{10}O_2PF + 2\ Na_2CO_3 + 6.5\ O_2 = NaF + Na_3PO_4 + 6\ CO_2 + 5\ H_2O \quad eq(1)$$

is proposed to be conducted either with excess oxygen or with a substoichiometric amount of oxygen, in which case the unit acts as a gasifier and produces a hydrogen and carbon monoxide rich off gas. The molten salt unit is capable of operating in a gasification mode and still have excellent performance in terms of waste destruction, PIC's, etc. It will be necessary to burn off the CO and H_2 gas in a thermal oxidizer or other primary pollutant control device downstream of the off gas filter. In an commercial industrial installation, the heating value of the fuel gas would be normally be utilized for energy recovery purposes. Oxygen enriched air can be used under gasification conditions to improve throughput, as can pressurized operation. Even modest over pressures (1 to 3 atmospheres) can provide significient capacity increases and can be shown to be economically desirable.

The molten salt reactor was sized as a 85 cm inside diameter, refractory lined vessel similar to the first pilot molten salt reactor. Heat losses from the salt bed are estimated at 100 kW and include radiation and convection from the carbon steel casing and radiation from the top of the bed into the upper gas disengagement space. The reactor shell is to be rated for maximum conditions of 10 atmospheres pressure, and 350 degrees C. Injection nozzles are to be located 90 degrees apart circumferentially at the bottom of the reactor. A melt withdrawal tap and drum loading station are also to be provided. A simplified schematic of the facility is presented in

Figure 2. Physically, the plant equipment would be located in an area approximately 12 m by 12 m with a maximum elevation of about 15 m.

There are a total of seven different Cases studied; see Table 3.

Plant Conditions	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6	Case 7
Stoichiometry (for CO2 & H2O)	1.200	0.826	1.059	0.954	0.726	0.689	0.671
Reactor Over Pressure, Atm	0	0	0	0	0	1	3
Oxygen, vol%	21	21	21	21	60	60	60
Adiabatic Reaction Temperature, C	1503	1543	1401	1385	1244	1129	1074
Excess Heat, kW	100	100	100	100	100	100	100
Reactants - Input at Plant Conditions							
Sarin, kg/h	45	70	53.1	60.1	149.8	304.9	615.0
Soda Ash, kg/h	68.6	105.5	160.7	181.9	453.19	922.7	1861.3
Air, kg/h	377	330	370	358	100	184	354
Nm3/h	293	256	287	278	78	143	274
Oxygen, kg/h	0	0	0	0	108	199	381
Nm3/h	0	0	0	0	75	139	267
Products - Output at Plant Conditions							
Spent Salt, kg/h	67	103	158	179	447	910	1835
Drums/day - 205 liter	5.87	9.04	13.97	15.81	39.38	80.19	161.75
composition - wt%	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Na2CO3	0.0	0.0	50.7	50.7	50.7	50.7	50.7
Na3PO4	79.6	79.6	39.2	39.2	39.2	39.2	39.2
NaF	20.4	20.4	10.0	10.0	10.0	10.0	10.0
Product Gases, Nm3/h	325	325	325	325	325	649	1299
composition - vol %							
CO2	13.4	13.1	15.7	16.0	19.7	17.1	15.9
CO	0.0	7.5	0.0	1.8	24.6	27.9	29.5
H2O	11.2	13.1	13.1	13.9	22.0	19.9	18.9
H2	0.0	4.1	0.0	0.9	14.9	17.7	19.0
N2	70.2	61.4	68.9	66.6	18.6	17.2	16.5
O2	4.4	0.0	1.5	0.0	0.0	0.0	0.0
Ar	0.8	0.7	0.8	0.8	0.2	0.2	0.2
HHV (wet) Btu/acf	0	38	0	9	128	147	157
HHV (dry) Btu/acf	0	43	0	10	163	184	193
Total Fuel Gas - Btu/h	0	495.898	0	115.246	1.873.754	4.216.215	8.864.876

TABLE 3. Seven Case Studies

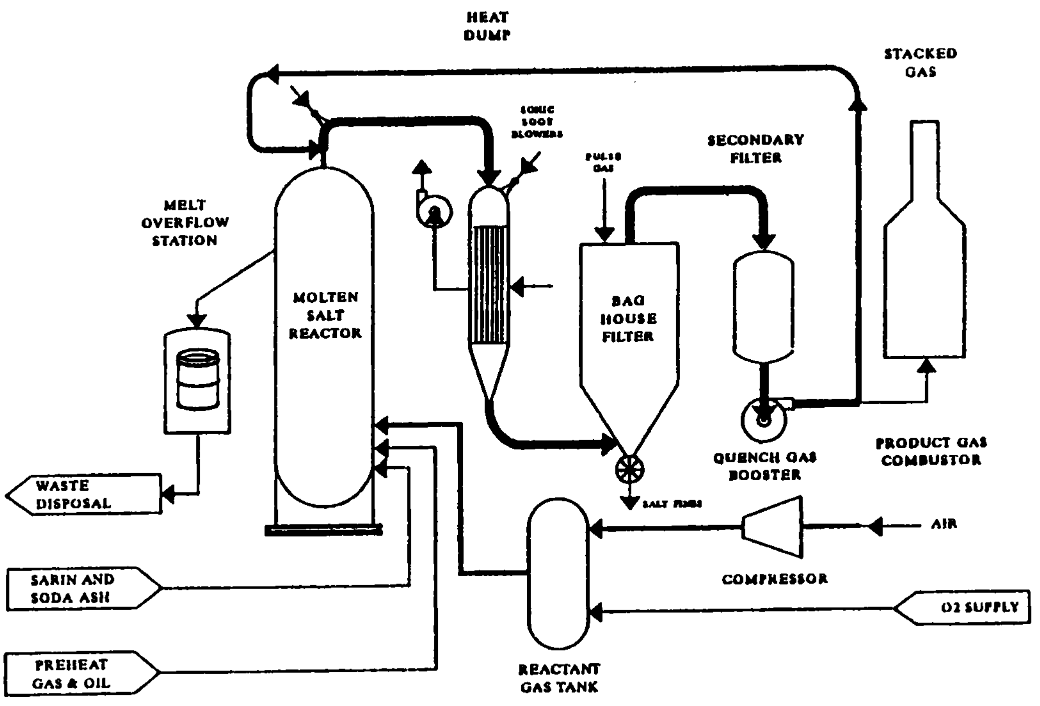

FIGURE 2. Simple Plant Schematic Diagram

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The cases represent different plant operating assumptions to illustrate the effects of excess soda ash, oxidizing and reducing modes, oxygen enrichment, and pressure operation on plant capacity (kg/h of Sarin processed) and on plant operating costs ($/kg Sarin processed).

The capacity of the molten salt unit is limited by the maximum superficial velocity permitted in the salt bed without causing excessive salt entrainment into the off gas piping. The capacity is also limited by the excess heat that must be rejected from the salt bed. The difference in the heat of reaction and the heat needed to bring the reactants to the bed operating conditions is defined as the excess heat and this must be equal to the bed heat losses for the plant to balance at the conditions of throughput, composition, and bed temperature specified. Figure 3 presents the Excess Heat plotted for the seven case studies and shows where the plant operation points equal the bed heat losses.

No firing of auxilary fuel is necessary when feeding chemical agents. Molten salt reactors can accomodate very low heating value reactant feed compositions and when pressurized, can closely approach the throughput of an adiabatic gasifier, the maximum capacity design point for a thermal treatment reactor. Design limitations on the refractory walls limit thermal gradients, block thickness, and maximum thermal heat flux, all of which constrain the throughput of waste material.

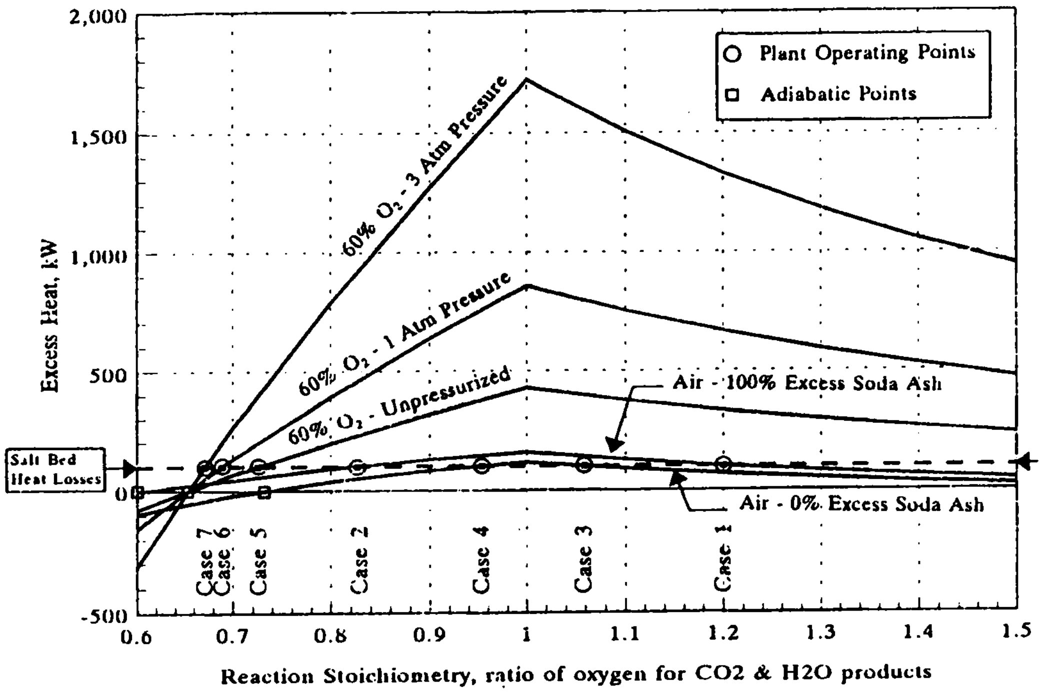

FIGURE 3. Plant Operation - Heat Balances

The first operating mode examined was the case where the amount of soda ash equaled the stoichiometric amount required by the reaction in equation (1). This would provide a case where the excess reagents used and the wastes formed would be at the minimum possible. Using an exact amount of soda ash, the plant can be operated at two points; one in the traditional oxidizing region, and a second in the reducing region. These correspond to Cases 1 and 2 in Table 3 and Figure 3.

In actual practice, more than the stoichiometric amount of soda ash will be required to be assured of excess alkalinity in the bed. All of the remaining cases were examined assuming 100% excess soda ash. Cases 3 and 4 are the same as the first two cases with the exception of the extra soda ash. The extra heat required to bring the extra soda ash to bed conditions results in a slight increase in the Sarin thoughput under oxidizing conditions (Cases 1 and 3), and a slight reduction in Sarin thoughput under reducing conditions (Cases 2 and 4. The amount of spent salt to be desposed of as waste increases from 1.5 kg salt/kg Sarin to 3.0 kg salt/kg Sarin. As will be seen later, this has only a very minor impact on the operating costs and most of the impact is caused by the differences in capacity, not the added waste salt.

Case 5 was run to illustrate the effect of using oxygen enriched air when in the reducing or gasification mode. An enrichment value of 60 vol % was used for Case 5 and the remaining Cases 6 and 7. In Case 5, the Sarin capacity of the plant increases significiently, from 60.1 kg/h in Case 4 to almost 150 kg/h.

All of cases 1 through 5 are with the molten salt reactor operating unpressurized. The molten salt process in the gasification mode lends itself readily to pressure operation, however, and Cases 6 and 7 were done assuming a plant operating at an over pressure of 1.0 atmospheres and 3.0 atmospheres. In each case, the Sarin throughput approximately doubes, eg. from 150 kg/h in Case 5 (no over pressure), to 300 kg/h in Case 6 (1.0 atmospheres), to 600 kg/hr in Case 7. The increased capacity to process agent with basically the same plant hardware obviously is desirable in order to reduce costs.

CAPITAL COSTS

An estimate of the plant equipment and facility costs (capital costs) was made and is presented in Table 4. The costs for the molten salt reactor was based on the unit fabricated costs of the refractories, and the steel shell together with past cost data for similar procurement packages. Costs are provided for three different sets of plant requirements; 1) air blown, oxidizing, no pressure, 2) no pressure, reducing operation with the need for a off gas thermal oxidizer, and 3) pressurized, reducing operation, and oxygen blown. Cases 1 and 3 fall into the first category, Cases 2, 4, and 5 fall into the second category, and Cases 6 and 7 fall in the last, more costly category.

Estimated Capital Costs	Cases 1, 3	Cases 2, 4, 5	Cases 6, 7
Process Equipment			
Molten Salt Reactor/Melt Station	$1,500,000	$1,500,000	$1,750,000
Material Handling/Storage	$350,000	$350,000	$450,000
Product Gas Cooler/Baghouse	$250,000	$250,000	$350,000
Product Gas Oxidizer	$0	$150,000	$350,000
Reactor Piping	$262,500	$285,000	$367,500
Electrical	$350,000	$380,000	$490,000
Instrumentation	$437,500	$475,000	$612,500
Subtotal	$3,150,000	$3,390,000	$4,370,000
Facility			
Civil/Structural/Arch	$1,890,000	$2,034,000	$2,622,000
Mechanical Equipment	$1,260,000	$1,356,000	$1,748,000
Facility Piping	$315,000	$339,000	$437,000
Facility Electrical	$378,000	$406,800	$524,400
Facility Instrumentation	$315,000	$339,000	$437,000
Subtotal	$4,158,000	$4,474,800	$5,768,400
Total Invested Capital	**$7,308,000**	**$7,864,800**	**$10,138,400**

TABLE 4. Molten Salt Plant Capital Costs

The total facility costs ranged from $7.3 million to $10 million and include only normal industrial facility requirements. Special requirements for handling and containing toxic agents, disassembly or draining of containers, or special decontamination stations are not in the cost estimates and would have to be provided for separately. There is a 25% contingency included in the cost estimates. The costs should be considered as engineering cost study estimates with an approximate +/- 30% accuracy. No costs were included for local or federal permits, environmental or hazards analysis, or other very real costs that would be part of an actual program to build and operate such a facility with actual agents.

OPERATING COSTS

Annual direct plant operating costs are estimated for each of the seven cases and are presented in Table 5. For Case 3, the only case where the plant would be operated under oxidizing conditions, using air, and without pressure (for comparison with more conventional processes), the operating cost per kg Sarin was \$8.14 or \$8,140 per metric ton. Cases 4, 5, 6, and 7 are increasingly more aggressive in increasing throughput, and their costs are \$7.58, \$4.79, \$3.81, and \$3.26 per kg Sarin, respectively. Another benefit that would accrue from the increase in plant capacity is the reduction in time the plant would have to be operated at a particular site to dispose of a given inventory of agent. Continuing operations would of necessity mean continuing exposure to potentially hazardous conditions, and continuing costs of the attendant program overhead expenses.

The difference in cost between Case 1 and Case 3 where the amount of soda ash used was doubled, is only \$0.24 / kg out of a total cost of about \$8.00 / kg. Where the amount of soda ash used becomes a very significant cost, is in the cases where the Sarin throughput is higher. In Cases 5, 6, and 7, the costs for soda ash, drums, and waste disposal ranges from 45 to 67 % of the total operating costs. In these cases one would want to optimize the amount of soda ash and not set it at an arbitrary ratio.

Note that there is no capital cost component in the operating costs as

Cost Component	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6	Case 7
Annual Basis - 330 day, hrs	7900	7900	7900	7900	7900	7900	7900
Electrical, kW @ $0.07/kWh	$41,254	$41,254	$41,254	$41,254	$41,254	$42,296	$47,242
Oxygen @ $300/ton cryogenic	$0	$0	$0	$0	$255,960	$471,630	$902,970
Fuel Oil @ $1.00/gal	$1,000	$1,000	$1,000	$1,000	$1,000	$1,000	$1,000
Soda Ash, @ $275/ton	$149,034	$215,729	$349,121	$395,178	$984,555	$2,004,566	$4,043,674
Drums, @ $55/ea 17H	$106,541	$154,275	$253,556	$286,952	$714,747	$1,455,449	$2,935,763
Waste Disposal, @ $250/ton	$132,325	$189,600	$312,050	$353,525	$882,825	$1,797,250	$3,624,125
Maintenance @ 7% TIC	$511,560	$550,536	$511,560	$550,536	$550,536	$709,688	$709,688
Operators, 5 shifts @ $100k/man-y	$1,500,000	$1,500,000	$1,500,000	$1,500,000	$1,500,000	$1,500,000	$1,500,000
Subtotal	$2,441,713	$2,652,394	$2,968,540	$3,128,444	$4,930,877	$7,981,878	$13,764,462
Operating Contingency @ 15%	$366,257	$397,859	$445,281	$469,267	$739,632	$1,197,282	$2,064,669
Total Estimated Annual Operating	$2,807,970	$3,050,253	$3,413,821	$3,597,711	$5,670,509	$9,179,160	$15,829,131
Annual Sarin Processed, kg	355,500	521,400	419,490	474,790	1,183,420	2,408,710	4,858,500
Operations Cost, $/kg	$7.90	$5.85	$8.14	$7.58	$4.79	$3.81	$3.26
Relative Costs	2.42	1.80	2.50	2.33	1.47	1.17	1.00

TABLE 5. Estimated Annual Operating Costs

estimated. Unlike a commercial project, the entire cost of the capital improvements (and their ultimate disposal) will have to be carried by the tonnage of agent, dunnage, and spent neutralization reagents that are fed to the plant. This will be a relatively fixed amount of material for each potential site, but will vary from site to site. The costs presented must be used very carefully if compared with costs developed for other processes to be sure that all of the component costs are done on a comparable basis.

CONCLUSIONS

Molten salt as an alternative technology to incineration, is positioned to be piloted on a significant scale with actual agents, and holds the promise for operation with a minimal impact on the environment and at reasonable costs. It is believed that its simple, stable, and forgiving operating characteristics will be perceived by the public as a safer alternative to gas phase incineration of chemical wastes. Its compact, small plant size lends itself to being located at the point of storage or generation of such wastes, further avoiding the public's fear of large, centralized facilities. Once the waste material is injected at the bottom of the large, molten salt bed, it can not escape without passing through the bed and being destroyed.

Costs for the construction and operation of a molten salt facility were developed and show a wide range of options as to how such a plant might be operated economically. Operation in the gasification mode with its attendant benefits using enriched oxygen and pressure were shown to permit a plant that would normally operate at a throughput of 50 kg/h be operated at an increased throughput of 600 kg/h. The relative cost of operating at these two throughputs was a factor of 2.5 times less for the 600 kg/h capacity.

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