

Ozone Destruction of Hanford Site Tank Waste Organics

S. A. Colby

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OZONE DESTRUCTION OF HANFORD SITE TANK WASTE ORGANICS

S. A. Colby

ABSTRACT

Ozone processing is one of several technologies being developed to meet the intent of the Secretary of the U.S. Department of Energy, Decision on the Programmatic Approach and Near-Term Actions for Management and Disposal of Hanford Tank Waste Decision Statement, dated December 20, 1991, which emphasizes the need to resolve tank safety issues by destroying or modifying the constituents (e.g., organics) that cause safety concerns. As a result, the major tank treatment objectives on the Hanford Site are to resolve the tank safety issues regarding organic compounds (and accompanying flammable gas generation), which all potentially can react to evolve heat and gases.

This report contains scoping test results of an alkaline ozone oxidation process to destroy organic compounds found in the Hanford Site's radioactive waste storage tanks. Ozone has been used for the past 100 years in the municipal waste industry to oxidize trace organics. Ozone is created by passing oxygen gas through a corona electric field. Industrial equipment can mass produce ozone gas in concentrations up to 10 weight percent. Because ozone gas is relatively dilute, large quantities of carrier gas are needed, especially for treating wastes that contain high organic concentrations.

A laboratory scale continuous-stirred tank reactor contacted gaseous ozone with a simulated waste at near ambient conditions of temperature and pressure. Estimated scale-up of the analytical data indicates that

70,000 kilowatt hours per day of power and 90,000 kilograms per day of oxygen carrier gas would be required to treat 20 million liters of 3:1 diluted tank waste per year (i.e., a 20-gallon-per-minute plant operating 180 days per year). Note the estimated scale-up has been developed for comparison of this candidate process to other processes and is based on preliminary laboratory testing.

Analytical data indicate that the ozone reactor provides sufficient mass transport to use greater than 90 percent of the ozone to oxidize waste simulant constituents. Additionally, the process performed similarly under varying operating conditions (e.g., temperature and hydroxide concentration) indicating that the process is robust. The likely by-products from ozone oxidation of organics are carbonate and small carbon chain refractory organics that are considered to pose minimal safety concerns. A full-scale process, however, would require large volumes of gas to contact the relatively dilute ozone with feedstock waste.

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LIST OF TERMS

COD
CSTR
EDTA
TOC

chemical oxygen demand
continuous-stirred tank reactor
ethylenediaminetetraacetic acid
total organic carbon

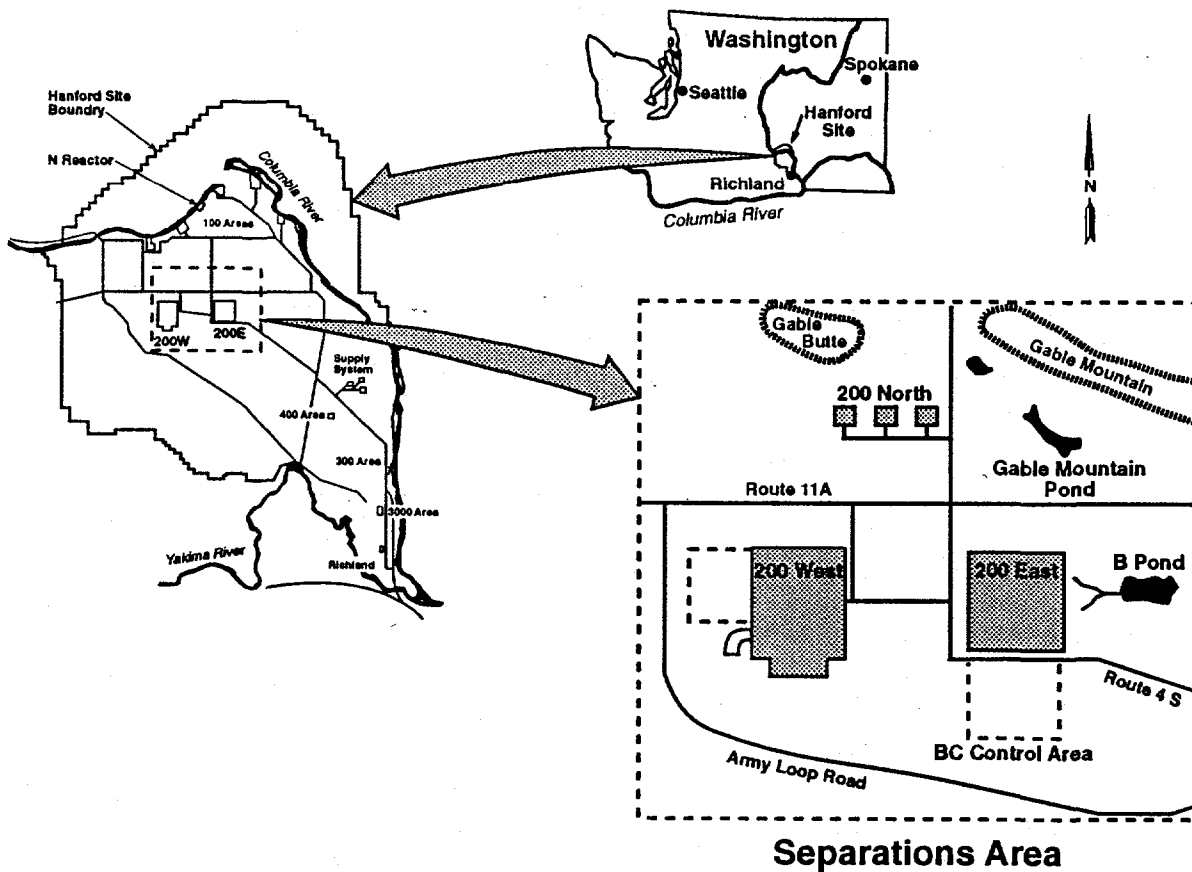
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1.0 INTRODUCTION

The 1,400-km² Hanford Site is located in southeastern Washington State, near the Columbia River. Figure 1 shows the location of the Hanford Site in Washington State and the separations area on the Site where the waste storage tanks are located.

Spent nuclear fuel has been processed at the Hanford Site to recover nuclear material since the 1940's. As a result, radioactive waste has been generated and stored on the Site in underground tanks. Presently, 177 tanks store 155 million L of radioactive waste that has been generated over the last 40 years (Hanlon 1991).

Figure 1. The Hanford Site Area Map.



The Hanford Site tank wastes contain radioactive constituents, making tests with actual waste difficult. Therefore, a nonradioactive waste simulant was prepared in the laboratory to evaluate the prototype homogenizer/ozonator's ability to oxidize organic compounds. The composition of tank 241-SY-101 was chosen for testing because it poses the highest safety concern of all the Hanford Site's organic containing tanks. The waste simulant composition of tank 241-SY-101 is shown in Appendix A.

The organic compounds added to the simulated waste may not accurately portray the actual organic species contained in the radioactive tank waste. Both radiological and chemical interactions in the waste material may have altered the original organics added.

2.0 SCOPE

This scoping study evaluated the fundamental premise of alkaline ozone oxidation to destroy surrogate organics contained in a tank 241-SY-101 simulated tank waste. Note that deficiencies in ferrocyanide analysis techniques have delayed tests with simulated wastes from tanks containing ferrocyanide. A comprehensive program based on the results of this initial study is outlined below.

- Tests are needed using actual tank waste to verify the results obtained testing nonradioactive simulants.
- Offgases created from destruction of organics should be sampled and analyzed for constituents and flammability potential (explosion potential in reactor from high concentrations of oxygen and possibly lower concentrations of volatile organic).
- Additional analysis of the treated product is needed to identify metal valence changes and organic byproducts.
- Larger scale tests are needed to determine equipment size for a production facility.

Work on these tasks has been started and scheduled for completion in fiscal year 1993.

3.0 EXPERIMENTAL

Testing was performed typically using 100 mL of nonradioactive simulated waste diluted with 300 mL of water (or in some cases 400 mL of water). The waste slurry was placed into the reservoir depicted in the process laboratory flow diagram (Figure 2) and recycled through the ozone reactor at 0.8 ± 0.01 L/min. A high recycle rate was used to facilitate obtaining mass transport and kinetic data. The ozone generator continuously supplied a

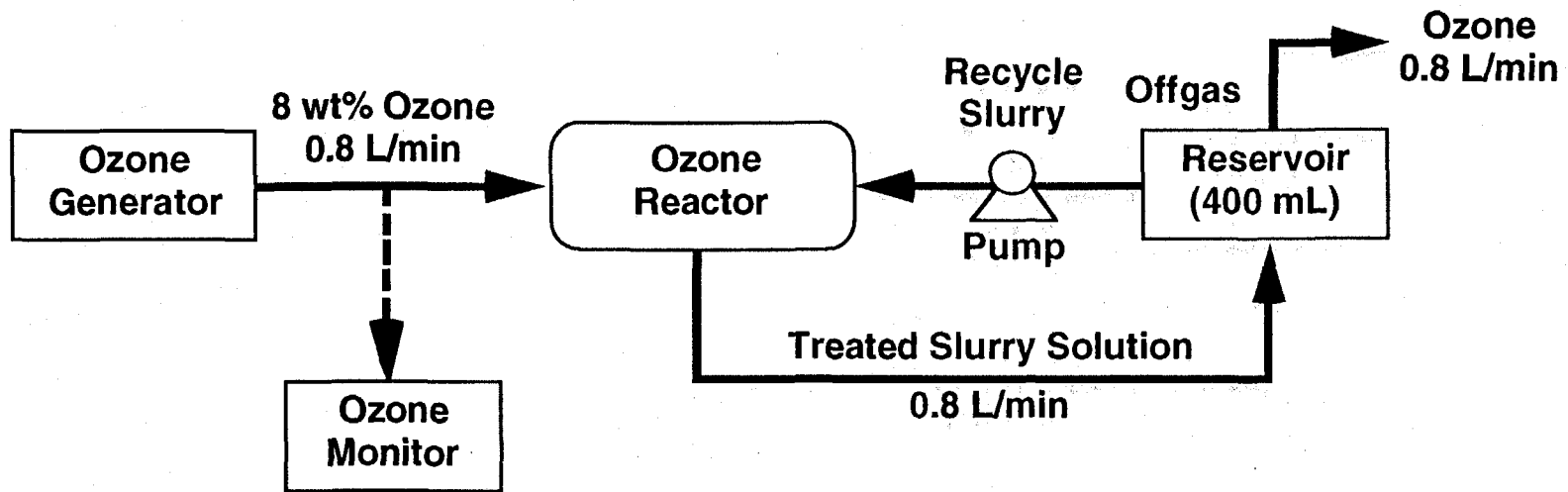


Figure 2. Ozone Process Laboratory Flow Diagram.

stream of 8 ± 0.3 wt% ozone carried in oxygen at 0.8 ± 0.05 L/min (i.e., 4.5 g ozone/h). The length of tests varied from between 8 and 20 hours. The ozone gas concentration was measured before entering and after exiting the reactor. Cooling water was supplied to the reactor's mixing shaft to help maintain the simulant temperature at 30 ± 5 °C. The free hydroxide concentration was 0.1 to 0.2 molar. Adjustment of pH was used only to replenish hydroxide consumed by oxidation of organic. Total solution volume was measured to compensate analytical results for evaporation of water.

Samples (5 mL) of the treated simulant were taken every hour. Total organic carbon (TOC) analysis was used to measure the amount of organic oxidized. Chemical oxygen demand (COD) was used to indicate the total amount of ozone oxidation and to indicate ozone use.

To determine the feasibility of using ozone to oxidize tank waste organics, a laboratory scale continuous-stirred tank reactor (CSTR) was employed. Figure 3 depicts the 50-mL-capacity CSTR used for testing.

The CSTR uses short residence times (a few seconds) because of ozone's fast decomposition rate. High-shear mixing in the reactor increases the rate of gaseous ozone's mass transport into solution. The decomposition kinetics of ozone in alkaline medium is in the order of seconds. Because of the fast ozone decomposition kinetics, the ozone/organic reaction rate is limited by mass transport of ozone into solution. In general, the mass transport can be increased by either gas/liquid mixing and/or retention time (Horvath et al. 1985; Bollyky et al. 1981)

An inline static mixer was used downstream of the CSTR to further increase the mass transport of ozone into solution. The static mixer comprises a section of pipe containing internal radial mixing blades that create turbulent flow. Literature indicates that inline static reactors are efficient ozone reactors (Nebel et al. 1973).

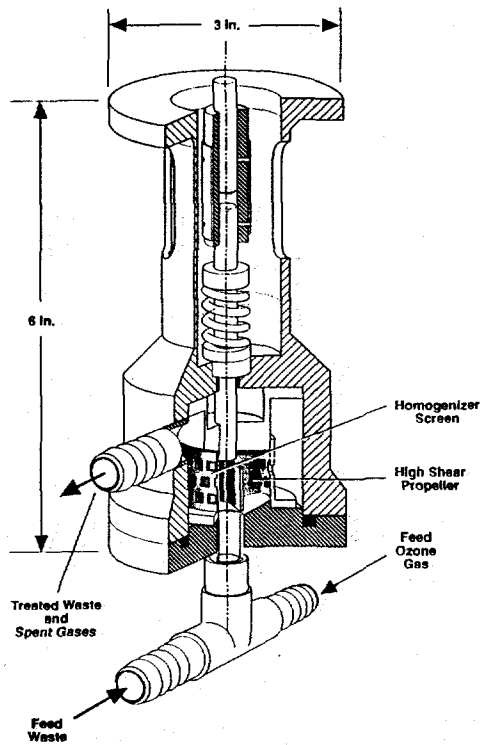
4.0 ANALYTICAL RESULTS

Figure 4 delineates the reduction in TOC from ozone treating of the following sodium salts: (1) oxalate, (2) acetate, (3) citrate, and (4) ethylenediaminetetraacetic acid (EDTA) organic. The data indicates that oxalate is not easily oxidized by ozone. The TOC is reduced by 50% for acetate and citrate, and 90% for EDTA.

The analytical data suggest that ozone is initially consumed by oxidizing nitrite to nitrate, which represents 70% of the COD. After significant nitrite oxidation, ozone begins to oxidize the surrogate organic, represented by the TOC depletion shown in Figure 4.

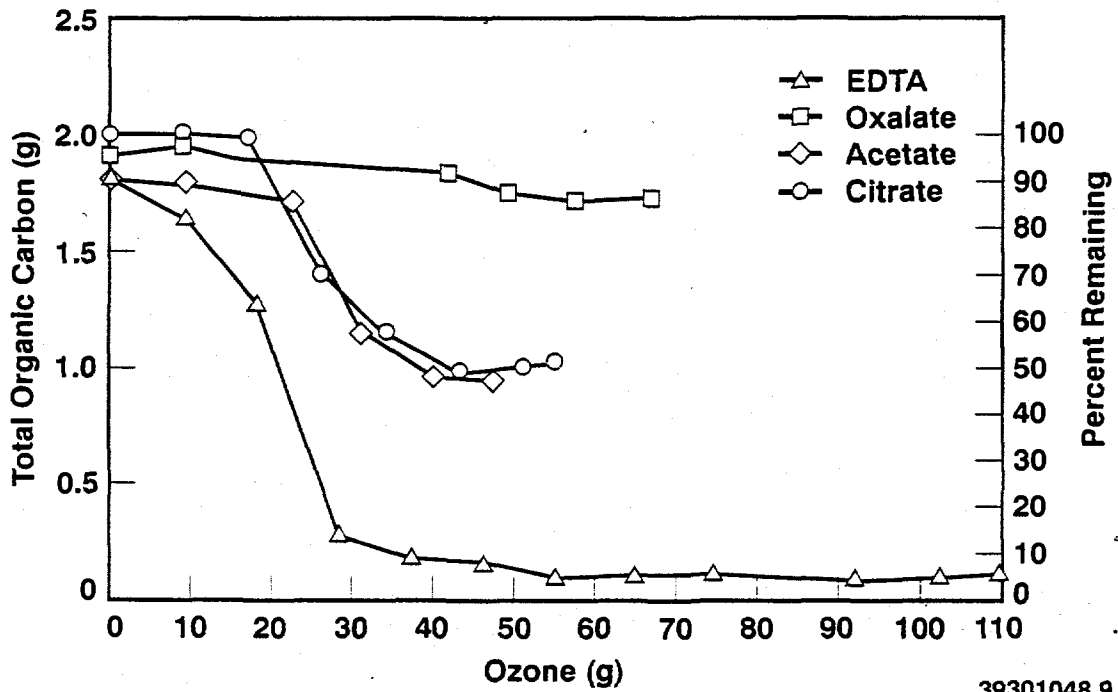
Ozone likely oxidizes the organics into carbonate and small carbon chain refractory organics. Small carbon chain organics, however, are thought not to pose safety problems regarding flammable gas generation or an organic exothermic reaction. The organic oxidation occurring in the tanks, which generates flammable gas, will presumably cease after ozonation has decomposed

Figure 3. Prototype Ozone Reactor.



39203128.1 F10

Figure 4. Ozone Oxidation of Surrogate Organic Compounds in a Simulated Tank 241-SY-101 Waste.



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the complex organics into carbonate and small carbon chain compounds. Secondly, the organic species remaining after ozonation are thought to be unable to produce any significant exothermic reaction that could propagate to create heat and gases.

The required level of organic destruction to resolve tank safety issues is not well defined; however, a grout waste feed specification of 1.5 g/L TOC (assuming a 1.0 specific gravity) is shown for reference (Hendrickson 1991). In principle, the degree of organic destruction should be linked directly to the safe storage of treated tank waste as follows:

- Quantify the maximum gas generation rate.
- Quantify the maximum exotherm caused from an organic reaction.
- Quantify grout performance using treated product, i.e., compressive strength and toxic characteristic leach procedure tests.

The effect of varying operating conditions on reactor performance are depicted in Figure 5, using EDTA as the surrogate organic. The ozone reactor performed similarly under varying operating conditions.

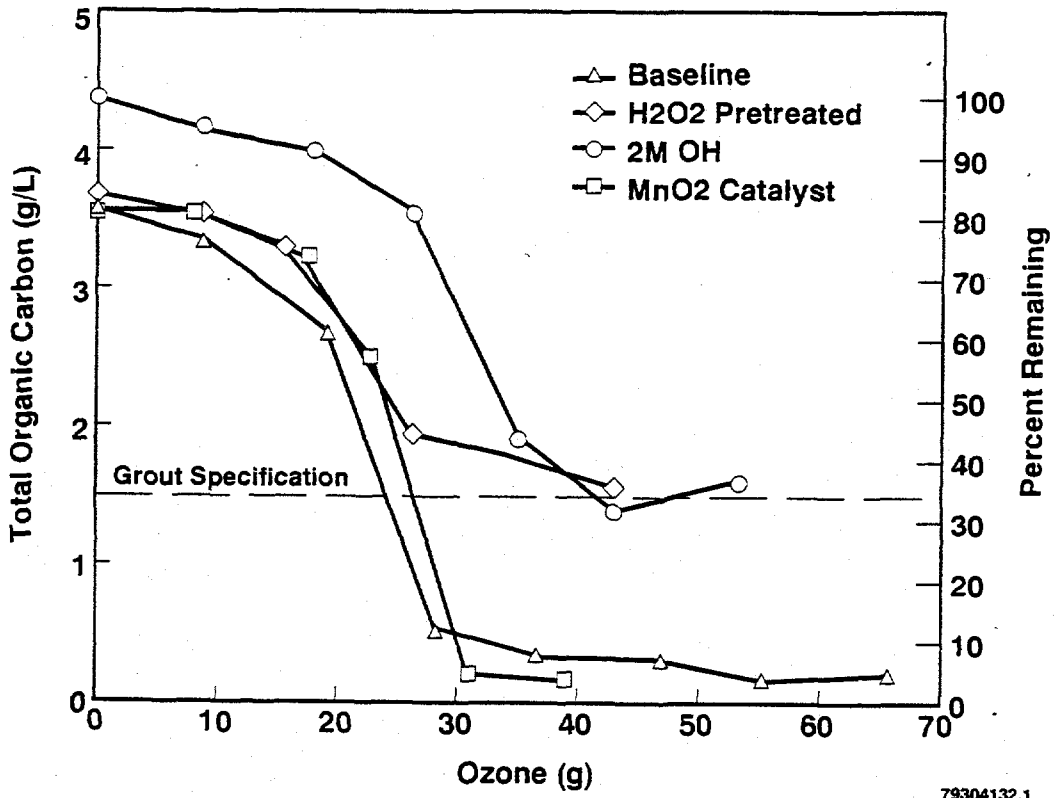
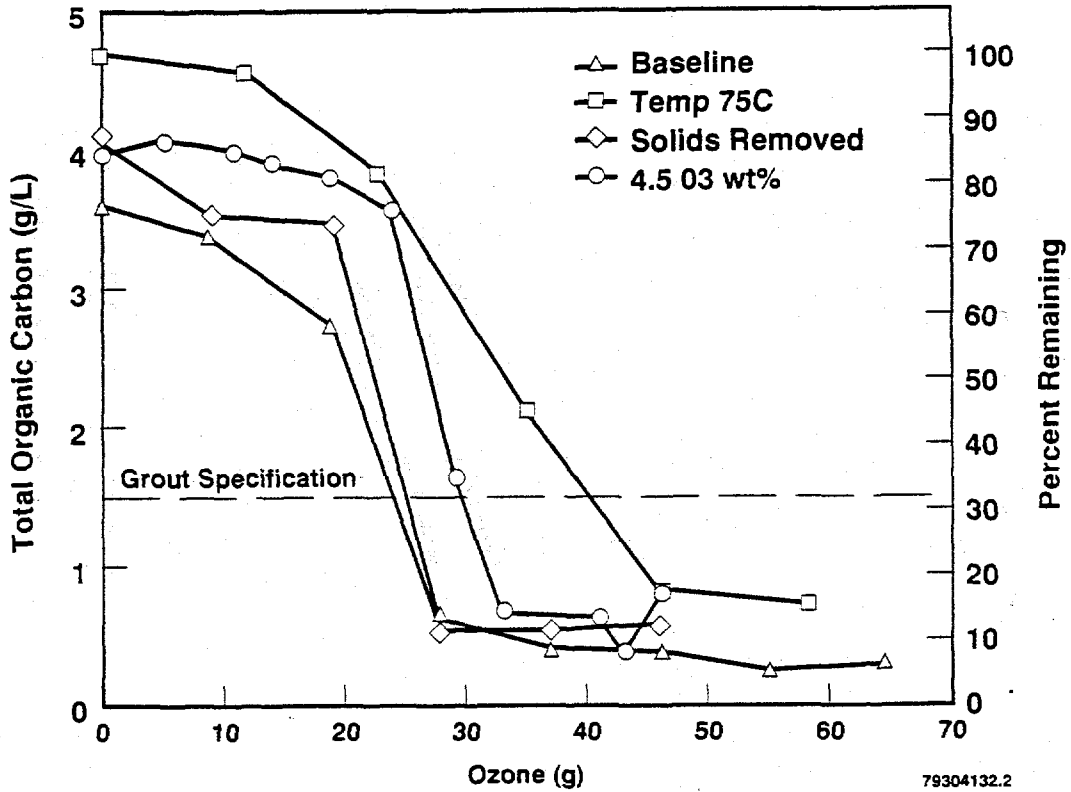
Raising the solution temperature from 30 °C to 75 °C and raising the hydroxide concentration from 0.01 molar to 2 molar appear to have most detrimental affect on ozone use. Ozone decomposition kinetics increase with temperature and hydroxide concentration, which in turn increase the amount of ozone back-reacting to produce diatomic oxygen (O_2), instead of reacting with oxidizable species in solution (Winters 1981).

Insoluble solids were removed from the simulant by centrifuge. The liquid portion was tested because of a proposed plan to perform a solid-liquid separation by water wash before ozone processing. The solids' presence appears to have little affect on ozone demand. The ozone gas concentration was reduced from 8 wt% to 4.5 wt% to determine if the higher concentration of ozone exceeds the solubility limit of ozone in solution and may have impeded ozone's mass transport. The data indicate that ozone demand is similar regardless of the ozone concentrations tested.

The simulant was pretreated using 300 mL of 30 wt% hydrogen peroxide in place of the 300 mL of dilution water, in an attempt to oxidize nitrite to nitrate, thus lowering the ozone demand by 70%. Although nitrite oxidation by hydrogen peroxide was not analyzed for in the test, it appears that the peroxide did not oxidize the nitrite and, therefore, did not significantly reduce the ozone demand. Finally, ozone consumption remained unchanged when 10 g of manganese dioxide was added to help identify possible ozone decomposition catalysts.

Figure 6 delineates the reduction of COD by ozone treatment, using EDTA as the surrogate organic. COD measures the amount of oxidation performed by ozone. The reduction of COD occurred linearly, indicating that ozone is the limiting reactant at the higher COD concentrations tested.

Figure 5. Effect of Varying Operating Parameters on Reactor Performance Based on Ethylenediaminetetraacetic Acid.



The data indicate that the ozone reactor uses >90% of the ozone to oxidize material in the waste simulant. (Appendix B contains calculations of ozone use.) Constituents that are oxidized by ozone in the simulant are nitrite, organic, and multivalent metal ions. The theoretical reduction of COD by ozone processing portrays more ozone demand than actually was analytically measured. The deviance is contributed to experimental error (Bollky et al. 1981).

Several general observations were made during testing that provide insight into the applicability of a large scale alkaline ozone oxidation process. First, if the hydroxide concentration dropped below roughly 0.01 normal, aluminum compounds precipitated, substantially increasing the total volume of solids. The inorganic carbon produced during organic oxidation consumes free hydroxide and, therefore, additions of hydroxide may be needed to prevent excessive solids generation. Second, the ozone concentration in the reactor offgas was measured to be less than 10 ppm, contingent on the hydroxide concentration remaining above pH 12. Ozone decomposition kinetics increase substantially with rising pH. In the experiment, at lower pHs the ozone concentration in the offgas increased substantially. Therefore, the simulant pH was maintained > pH 12 in all experiments.

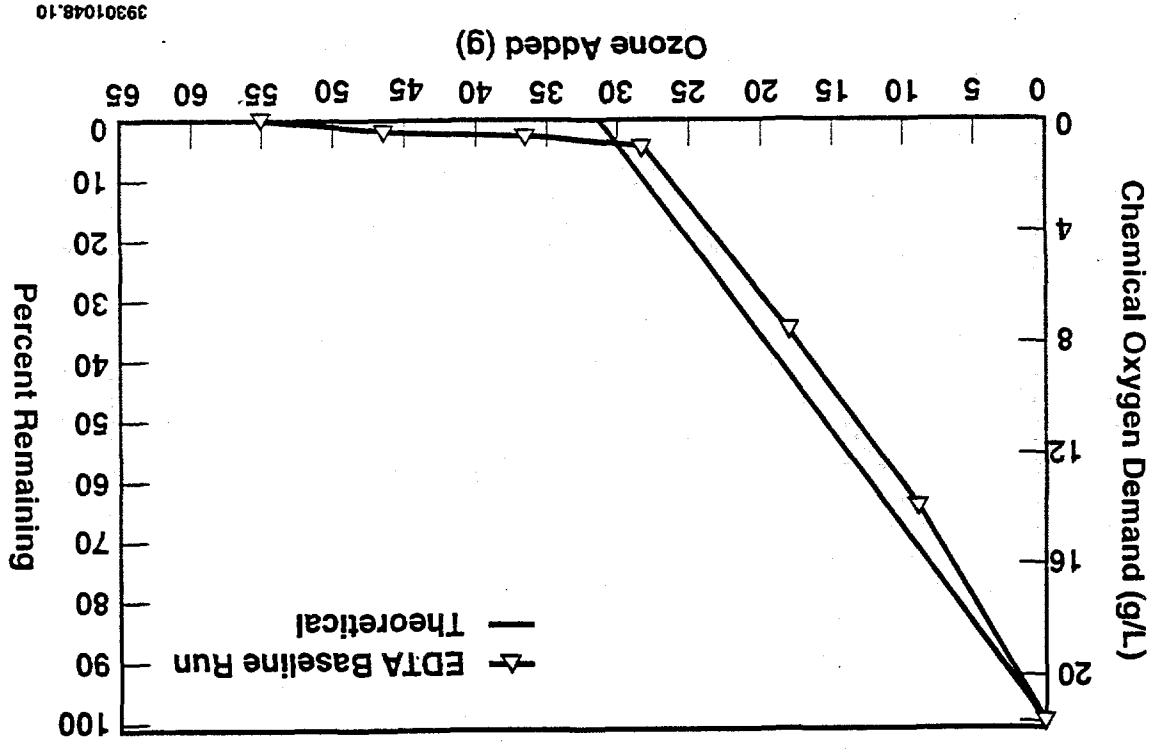


Figure 6. Ozone Reduction of Chemical Oxygen Demand Based on Ethylenediaminetetraacetic Acid.

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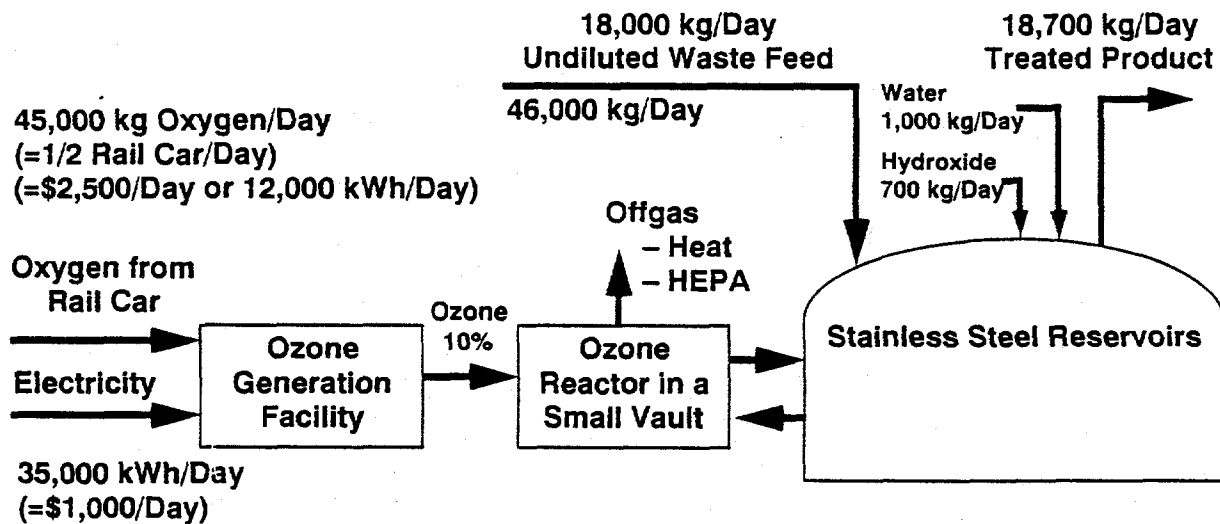
5.0 APPLICATION OF RESULTS

Estimated scale-up of the analytical data shown in Figure 7 indicate that 70,000 kWh/d of power and 90,000 kg/d of oxygen carrier gas would be required to treat 20 million L of 3:1 diluted tank waste per year (i.e., a 20-gal/min plant operating 180 d/yr). Additionally, a 4,000-L capacity CSTR is estimated from direct scale-up of the 50-mL laboratory CSTR. Note that obtaining mass transport properties, similar to the laboratory reactor, on a larger scale is optimistic (i.e., scaling by a factor of 80,000 from the laboratory reactor to a full-scale reactor). In lieu of larger scale tests, the estimated 4,000-L capacity CSTR provides only a rough minimum estimate of equipment size. (Appendix B contains calculations of scale-up from the analytical data.)

An ozone generation facility would supply ozone to a CSTR likely located in a vault. Waste would be transferred from a feed reservoir, contacted with the ozone in the reactor, and routed to a product reservoir. The offgas from the process would be heated to destroy unreacted ozone and volatile organics, filtered to remove particulates, and vented to the atmosphere. Water and hydroxide additions may be used to makeup for water evaporation and hydroxide consumption caused by processing.

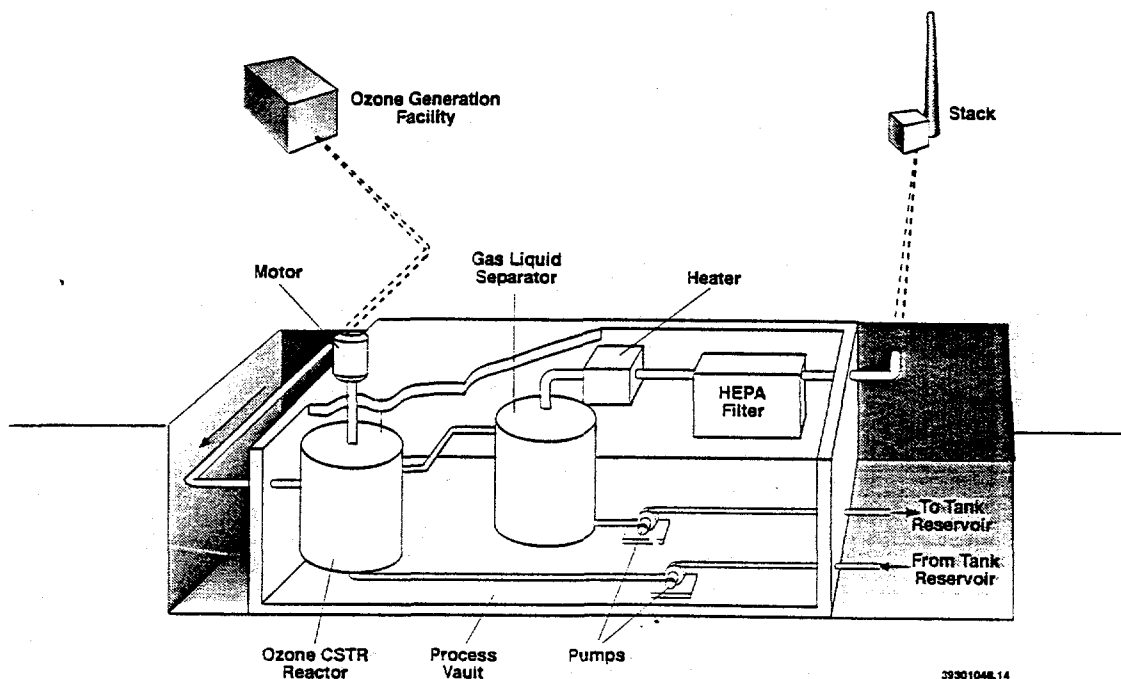
Conceptually, the ozone CSTR could be located in a small vault, adjacent to the ozone generation facility and waste reservoirs, as depicted in the simplified facility layout shown in Figure 8. The layout depicted provides one possible concept of how an ozone process might be designed.

Figure 7. Preliminary Full-Scale Ozone Process Mass Balance.



Note: Data obtained from bench scale tests using nonradioactive simulated tank waste.

Figure 8. Simplified Ozone Facility Layout.



6.0 CONCLUSION

Scoping tests show that alkaline ozone oxidation can destroy surrogate organics contained in a tank 241-SY-101 nonradioactive waste simulant into inorganic carbonate and smaller carbon chain refractory organics. Estimated scale-up of the analytical results indicates that the reactor and ancillary equipment are conceptually compact, facilitating a process vault concept.

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APPENDIX A

PROCEDURE FOR MAKING TANK 241-SY-101 WASTE SIMULANT

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**APPENDIX A
PROCEDURE FOR MAKING TANK 241-SY-101 WASTE SIMULANT**

The simulant recipe below is representative of tank 241-SY-101 which is presently releasing flammable gas, thus rendering the tank an unresolved tank safety issue. The composition was derived from the recent window C analytical results obtained from core sampling the tank.

The following procedure will be used to make the waste simulant:

- Mix the following acids into a 4-L beaker.

Chemical	Molarity	mL
HNO ₃	16	245.
HCL	12	34.
HF	27	1.

- Add the following chemicals to the acid solution.

Chemical	g
NaAlO ₂	150.
FeSO ₄ -7H ₂ O	0.25
Ca(NO ₃) ₂ -4H ₂ O	6.25
Cr ₂ O ₃	1.16
ZnO	0.18
NiSO ₄ -6H ₂ O	0.45
NaH ₂ PO ₄ -H ₂ O	12.7
Mo(OH) ₃	0.21
Sr(NO ₃) ₂	0.21
CsCl	0.84

- Slowly add NaOH to pH 9 to 12.

4. Add the following chemicals to the solution:

Chemical	g
Na_2CO_3	62.4
NaOH	100.
KOH	8.21
$\text{NH}_4(\text{OH})$	1.67
NaNO_2	330.

5. Add ONE of the following organic compounds, depending on the test.

Organic compound	Test sequence	g
Na_4EDTA	1	57.
Oxalate	2	103.
Citrate	3	49.
Glycol	4	58.

6. Add water to make 1 L.
7. Blend the solution at 60 °C for one hour.

The simulant composition is listed in Table A-1.

Table A-1. Undiluted
Tank 241-SY-101 Simulant
Composition.

Chemicals	Concentration (gmol/L)
Na	14.5
NO ₃	3.97
SO ₄	0.0026
NO ₂	4.78
CO ₃	0.59
OH	2.63
Cr	0.15
Fe	.0009
K	0.15
Mo	0.0015
Zn	0.0022
Al	1.83
Cl	0.41
Ca	0.27
F	0.01
NH ₄	0.048
Ni	0.025
PO ₄	0.092
TOC	1.54
Sr	.001
Cs	.005

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APPENDIX B

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APPENDIX B

Calculate ozone use (= mole COD/mole O₃) based on Figure 6.

Given:

- @ 0 g O₃ feed at 10.85 g COD in simulant
 also
 @ 30 g O₃ feed at 0.44 g COD in simulant

Slope of the line is:

$$\frac{(10.85 \text{ g COD} - 0.44 \text{ g COD}) / (16 \text{ g/mol COD})}{(0 \text{ g O}_3 - 30 \text{ g O}_3) / 48 \text{ g/mol O}_3}$$

$$\Rightarrow 104\% \text{ O}_3 \text{ use} \leftarrow$$

(∴ conservatively assume > 90%)

Calculate scale-up of process

Given:

Process rate	20 x 10 ⁶ L/yr (diluted waste)
Operation	180 d/yr
Gas density	1 L O ₂ /1.3 g O ₂
O ₃ req. from experiment	40 g O ₃ /500 mL diluted treated product
O ₃ Concentration	10 wt.% O ₃ (experiment used 8 wt%)
Electricity to make O ₃	0.8 kWh/kg O ₂ feed
Laboratory reactor volume	50 mL
Laboratory gas flow rate	48 L/h

$$\frac{(20 \times 10^6 \text{ L waste/yr}) (40 \text{ g O}_3 / 0.5 \text{ L waste}) (100 \text{ g O}_2)}{(180 \text{ d/yr}) (10 \text{ g O}_3)}$$

$$\Rightarrow 88,000 \text{ kg O}_2/\text{d} \leftarrow$$

(∴ conservatively assume 90,000 kg O₂/d ←)

Calculate scale-up of process (continued)

$$(1 \text{ L gas}/1.3 \text{ g O}_2)(88 \times 10^6 \text{ g O}_2/\text{d})(1 \text{ d}/24 \text{ h})$$

$$\Rightarrow 2.8 \times 10^6 \text{ L O}_2/\text{h}$$

Direct ratio between laboratory equipment and full-scale plant

Experimental	-	Full scale
$\frac{48 \text{ L/h}}{0.05 \text{ reactor volume}}$	=	$\frac{2.8 \times 10^6 \text{ L O}_2/\text{h}}{X \text{ reactor volume}}$

$$X = 2938\text{-L full-scale reactor capacity}$$

(∴ conservatively round to 4,000 L)

Power Requirements:

$$(88,000 \text{ kg O}_2/\text{d})(0.8 \text{ kWh/kg O}_2)$$

$$= 70,400 \text{ kWh/d}$$

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