WASTE TREATMENT AND HANDLING
PROCESSES ANNUAL REPORT

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ANNUAL REPORT*

by

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September 1974

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SUMMARY

Laboratory scale studies were conducted to evaluate pyrolysis and gasification processes for the volume reduction of combustible components found in typical alpha waste mixtures. Volume reduction obtained on pyrolysis of a simulated alpha waste mixture at 700°C was by an approximate factor of 2, while volume reduction of simulated feed with air was by a factor of about 20. It was also found that gasification of simulated wastes produced an inert stable ash, while pyrolysis produced a combustible carbonaceous residue. Both gasification and pyrolysis (700°C) of simulated wastes produced about 40% by weight of condensate. The lab studies also showed that fluxes of NaOH and Na₂CO₃ were effective in retaining chloride in the residue during pyrolysis of PVC. Results obtained on pyrolysis of individual components typically present in alpha wastes are presented in this report.

Design of a larger scale pyrolysis-incineration system to handle low level radioactive solid wastes was begun in March 1974, and is continuing at the time of this report. Work during this period concentrated on gathering the pertinent information essential to the design of this facility. Various similar processes were studied, including incineration, pyrolysis, and coal gasification systems, in order to identify the equipment used and its operability. Based on these studies, information from equipment manufacturers, and unique process requirements of the system, preliminary equipment selections were made. A general list of design criteria was developed for use in conceptual and detailed system design. Finally, a flowsheet of the pyrolysis-incineration system was prepared.

INTRODUCTION

Low level radioactive solid wastes, the so-called alpha wastes, are defined as those solid materials that contain plutonium or other long lived alpha emitters in concentrations greater than 10 μ Ci/kg, but have low enough external radiation levels to allow direct handling without supplementary shielding (surface dose rates of less than 10 mrem/hr). The criterion of

10 $\mu\text{Ci/kg}$ was selected since this is the upper range of alpha-emitting isotopes in naturally occurring deposits. Wastes containing less than 10 $\mu\text{Ci/kg}$ can presumably be disposed of in carefully selected burial grounds, whereas the alpha waste must be stored, or processed, to offer maximum assurance of permanent containment.

Alpha wastes, which arise mainly from fuel preparation and fabrication plants and, to a lesser extent, from fuel reprocessing plants⁽¹⁾, consist of a wide variety of solid materials. They include items made from cloth, wood, paper, plastics, rubber, glass, metal, and ceramic as well as salts and sludge from treatment of liquid waste streams and filters from gas cleanup systems. Densities of the uncompacted wastes vary from about 0.03 g/cm^3 to 3 g/cm^3 . It is estimated that approximately 1/2 to 2/3 of these wastes by volume are combustible and potentially can be incinerated to achieve volume reduction factors of about 50 and weight reductions of about 20. The average content of transuranic elements in the wastes is about 9g/m^3 .⁽²⁾

It is estimated that in the year 2000 alone, the generated volume of alpha wastes will be about $240,000 \text{ m}^3$.⁽³⁾ Fuel losses with the alpha wastes are estimated to total about 0.9%. It is extremely important that research and development be done to find efficient methods for reducing the volume of these wastes and recovering the fuel values in the wastes. Pacific Northwest Laboratory is currently developing a gasification process which is considered very attractive as a process for reducing the volume of alpha wastes and converting them to a stable ash. Current studies concern use of the process for the treatment of municipal solid wastes to reduce their volume and to recover energy from them in the form of a low Btu gas. The process appears capable of gasifying municipal wastes without the need for additional exhaust gas scrubbers and electrostatic precipitators to attain minimum air pollution standards.

This report presents some results of laboratory scale studies on pyrolysis and gasification of typical alpha waste components. Efforts on design of an experimental facility for gasification of alpha wastes are discussed.

The principal objective of the study is to develop alternative methods for safe and economical volume reduction of alpha wastes as a precursor to long-term storage, or to further processing for actinide recovery. The

efforts to date have been concerned primarily with program planning, scoping studies, and facility design. A report on program planning has been issued.⁽⁴⁾

CONCLUSIONS

The following conclusions can be made based on the results of the scoping studies:

- Volume reduction for pyrolysis of some waste components was less than desired. A 23% volume increase resulted from pyrolysis of neoprene rubber at 700°C. A volume reduction of 48% was obtained for pyrolysis of simulated wastes.
- Increase of PVC pyrolysis temperature from 500°C to 700°C increased the volume reduction from 49% to 62%.
- Variation of feed size had little, if any, effect on pyrolysis yields and volume reduction. Effects on volume reduction that were noted were attributable to variation in the waste density with particle size.
- Fluxes of Na_2CO_3 and NaOH effectively retained chloride in the residue during pyrolysis of PVC. The NaOH flux also markedly influenced the yield of gases, and use of both fluxes resulted in the presence of an aqueous phase in the condensate.
- Residue from pyrolysis of the different waste components was stable in argon and burned in air starting at temperatures between 390°C and 460°C, as determined by differential thermal analysis.
- A 95% volume reduction was obtained by gasification of simulated waste.
- Results indicated that channeling through the refuse bed occurred during gasification of simulated waste. Compaction of the top of the bed by ramming reduced the amount of channeling.
- Processing rates that were obtained suggest that gasification of 200 kg/hr of waste can be accomplished in a unit with a grate area of 0.5 m² and a height of 3 to 4 m.
- Oils and condensate produced in the process could be destroyed through direct heating by partial combustion with air, by indirect heating, or by combination with another process such as acid digestion or molten salt incineration.

- Results indicate that a unit with a continuous agitator for waste distribution and packing and a continuous grate for ash removal would eliminate channeling that occurs during waste gasification.
- Gasification of waste is a relatively simple process for complete conversion of waste to an inert form with a volume reduction of about 95 percent.

RECOMMENDATIONS

The potential for gasification of alpha waste as a process for rapid and efficient conversion of the waste to an inert form of small volume justifies the following recommendations for continued study of the process:

- A small process development unit should be built with a waste distributor and a continuous grate to demonstrate the channeling behavior of the fixed bed system during processing of typical alpha waste material. The unit should be at least 0.3 m in diameter and 2 m in height.
- The unit should be operated to find conditions that lead to efficient operation without channeling (such as addition of cellulosic material to the waste) and to quantify and characterize the condensate stream.
- Combining of different processes with fixed bed gasification to yield an optimum overall process should be investigated.
- Methods for handling the ash from the process and converting it to a low leachable form should be investigated.

DISCUSSION

BENCH-SCALE RESEARCH AND DEVELOPMENT

Equipment to use for the identification of products and determination of yields from pyrolysis and gasification of typical alpha waste components was designed and fabricated for the study. The equipment description and results of the experiments are presented in the following sections.

Description of Experimental Equipment

Components of the pyrolysis equipment constructed for the scoping experiments are pictured in Figure 1. The reactor is fabricated from 2 in., Schedule 10, 304 L stainless steel pipe, 14 in. long. The pipe is lined with an alumina tube (shown separately in Figure 1) which gives an

FIGURE 1. Pyrolysis Equipment for Scoping Experiments

effective inside diameter of 1.78 in. Use of a ceramic liner allows operation of the reactor over a wide range of thermal conditions, including slagging, if necessary. Alumina rods, 1/4 in. in diameter, spaced 3/16 in. apart, serve as a support for the material in the reactor and are left on the bed support plate separating the reactor and ash receiver. Ash, or molten slag, can pass between the rods from the reactor into the receiver. The receiver is fabricated from 5 in., Schedule 10, 304 L stainless steel pipe and pipe cap to give a 6 in. depth. An alumina liner is placed in the receiver to give the flexibility of receiving a molten slag without corrosion of the receiver. A relief valve that opens at 15 psig is used to prevent over pressurization of the system in the event that gas exit lines are plugged.

The feed hopper is an 11 in. long, 2 in. Schedule 40 pipe separated from the reactor by a 2 in. slide valve (not shown in Figure 1). A flange cover is used to seal the feed hopper while the slide valve is open to the reactor. A rod passes through a seal in the flange cover to ram feed into the reactor and to distribute the feed in the reactor. The rod at full insertion into the reactor extends to the bed support plate. The feed hopper is purged with N_2 before the slide valve is opened to the reactor to admit feed.

Thermocouples are inserted through the reactor wall to the inside of the alumina liner to determine internal temperature at one inch from the bottom of the reaction zone and one inch from the top of the reaction zone. In addition, thermocouples are used to record temperatures in the ash receiver and the inlet and outlet gas lines. A schematic of the assembled reactor showing the location of thermocouples and gas lines is presented in Figure 2.

Figure 3 pictures the equipment as assembled for operation. The ash receiver is placed in the resistance furnace (1000°C operating temperature) and the reactor is wrapped with glass tape for heating to temperatures of about 800°C. Gases from the reactor pass through a water cooled condenser tube (1/2 in. tubing surrounded by a water jacket) to remove condensibles, a caustic bubble scrubber to remove halides and sulfides, and a wet test

FIGURE 2. Schematic of Assembled Reactor

FIGURE 3. Assembled Pyrolysis Equipment

meter to determine the volume. The gases are then dried by passing through a column of drierite and analyzed by chromatography according to the procedure described in the following section.

Description of Experimental Procedures

Characterization of materials that are present in alpha waste mixtures was completed before experiments on pyrolysis were initiated. These characteristics (of latex rubber, tissue paper, rags, polyvinyl chloride, tygon, neoprene, and polyethylene) were obtained through the following procedures:

- Moisture content was obtained by drying a weighed quantity of material in a drying oven for 2 hr at 100°C and determining the weight loss.
- Ash content of the material was determined from the weight loss in the sample upon heating to 950°C in air for 2 hr.
- An adiabatic calorimeter pressurized to 30 atm with oxygen was used to determine the heat of combustion (calorific value).
- Each material was subjected to differential thermal analysis (DTA).
- Bulk density was obtained by weighing the quantity of material required to fill a 500 ml graduated cylinder. This changed with feed size, but while the variations are not tabulated, they were determined for each run to allow calculation of volume reduction.

Tests were also run on a simulated feed mixture, defined for this study as a combination of the individual constituents evaluated in the experimental program, and not a simulation of an actual waste stream. Such a simulation would be somewhat futile since day-to-day combinations are likely to vary considerably. Therefore, the experimental simulated feed composition is used in an attempt to define the interaction of waste constituents that were tested individually, in order to ascertain if the effect of variation of feed composition could be predicted.

The actual pyrolysis experiments were conducted in the equipment shown in Figure 3. The reactor and ash receiver (Figure 2) were heated to the desired temperature for pyrolysis while purging the system with nitrogen. After reaching the desired temperature, the top flange on the feed chamber was removed (slide valve closed), and a quantity of material sufficient to

generate about 12 l of gas was added to the feed chamber. A N_2 purge was then used to flush air with the sealed feed chamber. Next, the slide valve was opened, the material was rammed into the heated reactor, and the slide valve was closed. The volume of gas generated during pyrolysis was determined, and the gases were collected for analysis on a Hewlett-Packard gas chromatograph.

The above procedure was repeated three times for each material tested, with the condensate and residue that resulted from the three additions of material being recovered and weighed. The caustic scrub solution was sent to an independent analytical laboratory for determination of chloride and sulfur content, and the residue was examined for thermal stability (by DTA) and calorific value.

Experiments on gasification with air were conducted on a mixture of materials tested during pyrolysis. Initially, the reactor (Figure 2) was charged with 40 g of carbonaceous residue obtained from pyrolysis of the feed mixture at 700°C. The reactor was then heated to 500°C while purging the system with N_2 . Air flow to the reactor was started to ignite the char and sustain the gasification process, and external heating of the reactor was discontinued. Feed material was then added to the reactor in the manner described above in 50 g batches approximately every 20 min. At the end of a run, N_2 was substituted for the air flow to quench the reaction, and the contents of the reactor (ash, char, unreacted feed) were collected and weighed.

Analysis was done with a Hewlett-Packard model 7624A gas chromatograph of the gases obtained in the pyrolysis and gasification experiments. The off-gas composition is such that obtaining complete resolution of all components on a single column in a reasonable length of time is difficult. Therefore, the dual-columns-in-series arrangement, shown schematically in Figure 4, was adopted; the components of the product gas are completely separated on two columns in series, with a switching valve between the columns to allow the second column to be bypassed. The first column in the series is a 10 ft x 1/8 in. stainless steel Porapak Q^(a) (50-80 mesh), and the second column is a 10 ft x 1/8 in. Molecular Sieve 5A^(b) (60-80 mesh).

^aTrademark, Waters Corporation

^bTrademark, Linde Corporation

FIGURE 4. Schematic of Dual Column Gas Analysis

The switching valve allows the CO₂, ethane, ethylene, and propane and higher boiling compounds to be eluted directly from the Porapak column to the detector, thus bypassing the molecular sieve column required for separation of oxygen and nitrogen.

The carrier gas used has a composition of 8.5% H₂ and 9.15% He. This mixture increased the sensitivity to H₂ by a factor of two over what could be obtained with pure He as the carrier gas.

Calibration curves were prepared using analytically certified gas mixtures prepared by the Matheson Company. All peak areas were integrated by a Hewlett-Packard Model 3370 electronic integrator. A sample chromatograph obtained with the equipment is shown in Figure 5. A time of about 10 min is required for each sample analysis.

Experimental Results

The objective of the pyrolysis and gasification experiments was to evaluate the effectiveness of pyrolysis and gasification for the volume reduction of alpha waste components and mixtures. Thus, process evaluation required gathering of information on several quantities: weight reduction, volume reduction, gas volume and composition, condensate weight, residue properties, processing rate, and materials of construction. Not all of the above quantities were completely characterized in the scoping experiments.

Test results on the feed material characterizations are given in Table 1, and the percentage composition of the simulated feed is summarized in Table 2. Table 3 presents experimental conditions used in the study, and also lists the seven different components (plus simulated feed) that were tested.

The experiments were designed to evaluate the effects of feed size, pyrolysis temperature, and fluxing agent on yields of gas, condensate and solid residue. Air addition was used in experiments not listed in Table 2 for evaluation of fixed bed gasification of the simulated waste.

FIGURE 5. Sample Chromatograph

TABLE 1. Characteristics of Feed Materials

<u>Feed Material</u>	<u>Wt% Moisture</u>	<u>Wt% Ash</u>	<u>Calorific Value</u>		<u>Bulk Density</u> <u>g/cm³</u>	<u>Size, in.</u>
			<u>kcal/g</u>	<u>Btu/lb</u>		
50 wt% paper 50 wt% rags	3.4	0.77	3.98	7170	0.079	3/4
Latex Rubber Tubing	0.099	1.42	10.42	18,760	0.462	+1/5-5/8
Polyvinyl Chloride	0.043	1.83	4.98	8970	0.744	3/16 rod 5/8 long
Tygon	0.044	-0-	6.13	11,030	0.402	+1/4-5/8
Neoprene	0.16	28.5	4.83	8690	0.776	+1/4-5/8
Polyethylene	-0-	0.013	11.00	19,790	0.356	5/8 squares 1/8 to 1/4 thick

TABLE 2. Simulated Feed Composition

<u>Constituent</u>	<u>% By Weight</u>
Rubber Tubing	15
Neoprene	15
PVC	20
Tygon	10
Polyethylene	10
Tissue Paper	15
Rags	15

TABLE 3. Experimental Conditions for Scoping Studies

Exp. No.	Feed	Size, In.	Temp, °C	Gas Addition	Flux
1	Rubber Tubing	-5/8+1/4	700	None	None
2	PVC	↓	↓	↓	↓
3	Tygon	↓	↓	↓	↓
4	Polyethylene	↓	↓	↓	↓
5	Neoprene	↓	↓	↓	↓
6	50 wt% Paper- 50 wt% Cloth	↓	↓	↓	↓
7	PVC	-1/4+14 Mesh	↓	↓	1gNa ₂ CO ₃ /g PVC
8	↓	↓	↓	↓	1.7gNa ₂ CO ₃ /g PVC
9	↓	↓	↓	↓	0.75gNaOH/g PVC
10	↓	↓	↓	↓	1.3gNaOH/g PVC
11	↓	↓	↓	↓	None
12	Polyethylene	-1+5/8	↓	↓	↓
13	PVC	↓	↓	↓	↓
14	↓	-1/4+14 Mesh	500	↓	↓
15	Polyethylene	-5/8+14 Mesh	↓	↓	↓
16	Neoprene	↓	↓	↓	↓
17	Simulated	-5/8+14 Mesh	700	↓	↓
18	↓	↓	500	Air 1-2 l/min	↓

Sample results of differential thermal analysis (DTA), which indicates thermal stability of the different feed materials, are given in Figure 6 and 7 for tygon and neoprene respectively. The DTA for tygon shows 250°C as the endothermic decomposition starting point. Also decomposing endothermically are polyethylene starting at 420°C, tissue paper starting at 320°C, cloth starting at 320°C, and PVC starting at 260°C. Melting of polyethylene is noted by an endotherm starting at 80°C. Neoprene decomposes exothermically (Figure 7) starting at 340°C as does latex starting at 360°C. Complete decomposition of all materials is attained at 500°C.

FIGURE 6. DTA for Tygon in Argon

FIGURE 7. DTA for Neoprene in Argon

Results obtained on pyrolysis of feed components are given in Table 4. Conditions used for pyrolysis include two temperatures (500°C and 700°C), three feed sizes, and two concentrations of two different fluxes. Total weight yields are generally lower than expected, especially for runs with PVC. Several factors contribute to the apparent loss of material: 1) since the surface area of the off-gas lines and condenser is quite large, removal of tars from these surfaces for weighing is extremely difficult, 2) some of the condensate is collected in the caustic scrubber solution and wet-test meter which makes determination of the weight of condensate extremely difficult, 3) the caustic scrubber appears to be somewhat inefficient for HCl removal, especially in runs with PVC where HCl should account for 58% of the weight; however, in Run 5 with tygon, HCl should account for only about 25% of the weight, 4) unknowns are present in the off-gas, as is apparent from unknown peaks in the chromatographs, and are assumed to have a molecular weight of 58 for calculation of the average molecular weight of the off-gas. Items 1 through 3 are suspected to have a major contribution to the material loss, especially in Runs 3 and 5 where extra care to recover condensate was not exercised. Unknowns in the gas phase should not effect the overall balance more than 5%. The weight residue in the reactor can be determined without much difficulty and is accurately known.

Despite the low material balances, the effect of feed size and the addition of fluxes can be ascertained by examination of Table 4 and Tables 5 and 6, which represent average gas composition. For the three different sizes of polyvinyl chloride (PVC) used in Runs 3, 11, and 13, no significant effect on gas composition (Table 5) or on material distribution (Table 4) is apparent, although slightly more entrainment of material may have occurred in Run 11 with the smallest material size as evidenced by an increase in the amount of condensate recovered. The same observation can be made with the use of two sizes of polyethylene in Runs 6 and 12, with the additional observation that significantly more methane and fewer unknowns were produced from the larger size feed, Run 12, Table 5. A slower release of high molecular weight gases (unknowns) from the larger size feed than from the smaller size feed could result in a longer residence time in the reactor for the high molecular weight decomposition products, and thus more efficient thermal cracking to produce methane. The effect of feed size on volume reduction

TABLE 4. Yields From Pyrolysis of Materials

Run	Material	Size	Temp., °C	Gas	Conden- sate	Residue	H ₂ S	HCl	Total Weight % Yield	Gas Vol. Liter/ kg
1	Paper and Cloth	-5/8 in. +1/4 in.	700	20.2	40	20.1	--	--	80.4	198
2	Neoprene			16.9	6	43.6	0.9	12.9	80.2	199
3	PVC			11.3	0.7	13.7	--	25.7	51.4	167
4	Latex			40.3	42.4	1.5	--	0.3	84.5	338
6	Polyethylene			74.6	6.5	0.3	--	--	81.4	634
5	Tygon			25.3	2.7	7.3	0.2	29.1	64.6	300
7	PVC and Na ₂ CO ₃ Flux (Stoichiometric)	-1/4 in. +14 mesh		15.3	11.4 Tar 4.4 Aq	12.6	--	18.9	62.6	179
8	PVC and Na ₂ CO ₃ Flux (100% Excess)	17.8		13.4 Tar 9.2 Aq	6.8	--	7.0	54.2	178	
9	PVC and NaOH Flux (Stoichiometric)	11.0		23.0 Tar 9.0 Aq	36.6	--	7.4	87.0	205	
10	PVC and NaOH Flux (100% Excess)	10.4		7.5 Tar 39.4 Aq	30.1	--	-0-	87.4	276	
11	PVC	10.6		11.9	14.1	--	47.5	84.1	165	
13	PVC	-1 in. +5/8 In.		11.1	5.6	15.2	--	36.6	68.5	185
12	Polyethylene			71.9	14.3	0.6	--	--	86.8	693
14	PVC	-1/4 in. + 14 mesh		8.3	19.6	17.9	--	31.7	77.5	62
15	Neoprene	-5/8 in. +1/4 in.		7.6	30.8	46.2	--	5.0	88.6	76
16	Polyethylene			30.7	63.1	--	--	--	93.8	203
17	Simulated	-5/8 in. +14 mesh	700	27.1	41.8	18.5	--	5.1	92.5	243

noted in Table 5 for PVC is caused by the difference in bulk density of the feed. The bulk densities of PVC used in Runs 3 and 13 were 0.74 and 0.65 g/cm³ respectively, which accounts for the difference in feed and residence volume divided by the feed volume. No significant incentive for close control of the feed size is apparent from the results of the pyrolysis experiments with different feed sizes.

TABLE 5. Gas Composition for Pyrolysis at 700°C of Different Waste Components of Different Sizes

Run	Waste Component	Composition, Volume %								Volume Reduction, %
		H ₂	CO ₂	C ₂ H ₄	C ₂ H ₆	CH ₄	CO	C ₃ H ₈	Unknowns	
6	Polyethylene -5/8 in. +1/4 in.	8.9	0.07	35.1	7.1	23.9	--	12.6	12.3	> 99
12	Polyethylene -1 in. + 5/8 in.	8.4	0.07	34.2	5.9	34.4		12.0	5	> 99
3	PVC, -5/8 in. +1/4 in.	40.3	0.7	12.7	6.1	27.5	2.8	6.4	3.5	62
11	PVC, -1/4 in. + 14 mesh	41.8	0.07	13.2	4.0	30.9	0.7	4.3	5	63
13	PVC, -1 in. + 5/8 in.	40.2	0.2	15	4.1	35.0	--	3.5	2	74

*Molecular weight of 58 assumed for unknowns.

Use of fluxes with PVC significantly affected the gas composition (Table 6) and the material distribution (Table 4). An actual reduction in the amount of residue is noted when Na₂CO₃ is used as the flux in Runs 7 and 8, and of course, the amount of CO₂ and CO in the product gas increases as shown in Table 6. Also, an aqueous phase is produced when the fluxes are used (Table 4, Runs 7, 8, 9, and 10). This can be explained by examination of the reaction with HCl that occurs:

TABLE 6. Gas Compositions for Pyrolysis of PVC
With and Without Fluxes at 700°C

Run	Composition, Volume %								Avg. Molecular Weight
	H ₂	CO ₂	C ₂ H ₄	C ₂ H ₆	CH ₄	CO	C ₃ H ₈	Unknowns	
7	31.4	9.6	10.9	6.5	25.1	6.1	5.4	5	20.9
8	25.5	23.7	10.4	4.4	24.4	2.9	3.6	5	24.4
9	57.1	0.8	9.2	4.1	19.6	0.6	3.7	5	13.1
10	72.5	0.9	6.0	2.9	12.1	0.2	2.4	3	9.2
11	41.8	0.07	13.2	4.0	30.9	0.7	4.3	5	15.7

*Molecular weight of 58 assumed for unknowns.

The molecular weight of the NaCl retained in the residue is less than the molecular weight of the Na₂CO₃ flux. The CO₂ and water products can both react with products of pyrolysis to produce CO. The weight of residue as shown in Table 4 is determined by subtracting the weight of flux input from the weight of residue recovered.

The Na₂CO₃ flux was not nearly as effective for chloride retention as was the NaOH flux used in Runs 9 and 10. The use of the NaOH flux significantly influenced the volume of gas generated (Table 4) and the composition of the gas, which was rich in H₂ (Table 6). The amount of residue also increased significantly (Table 4) with the use of the NaOH flux as would be expected from the following reaction:

The molecular weight of the NaCl is greater than the NaOH flux which contributes to the added weight of residue. In essence, retention of chloride by the NaOH flux increases the weight of residue whereas with the Na₂CO₃ flux, chloride retention reduces the residue weight. Water produced by the reaction with NaOH could react with the char from pyrolysis to produce the large amounts of H₂ (Table 6, Runs 9 and 10), but this is doubtful since CO (which is not present in the product gas) would also be produced. The NaOH must catalyze degradation of PVC in a manner to produce large amounts of H₂.

Results of pyrolysis experiments at 500°C with polyethylene and PVC feeds are compared to results at 700°C in Table 7. In general, the amount of conversion of the materials to a gaseous product is greater at the higher temperature while the amount of residue and condensate is less. With polyethylene at 500°C, the condensate had the fluidity of a highly viscous grease while at 700°C the condensate appeared to have the fluidity of number 10 oil. Volume reduction is greater at the higher temperature for PVC.

TABLE 7. Effect of Temperature on Pyrolysis Yields

<u>Variable</u>	<u>PVC</u>		<u>Polyethylene</u>	
	<u>500°C</u>	<u>700°C</u>	<u>500°C</u>	<u>700°C</u>
Wt% Gas	8.3	10.6	30.7	74.6
Wt% HCl	31.7	47.5	--	--
Wt% Residue	17.9	14.1	--	0.3
Wt% Condensate	19.6	11.9	63.1	6.5
% Volume Reduction	49	62	>99	>99

Volume reductions obtained by pyrolysis of different waste components and simulated waste (Table 2) are summarized in Table 8. Generally, further volume reduction can be obtained by combustion of the carbonaceous residue. A volume increase is obtained on pyrolysis of neoprene as was also noted on combustion of neoprene samples to determine its ash content.

TABLE 8. Volume Reduction From Pyrolysis of Waste Components at 700°C

<u>Component</u>	<u>% Volume Reduction</u>	<u>Volume Feed/ Volume Residue</u>
50% Paper, 50% Cloth	80	5.1
Neoprene	-23	0.8
PVC	62	2.6
Latex Rubber	99	100
Tygon	93	14
Polyethylene	>99	>100
Simulated	48	1.9

Experimental and calculated results from pyrolysis at 700°C of a simulated feed (Table 2) are presented in Table 9. Calculated results are based on experimental results obtained with individual components combined in proportion to their presence in the simulated feed. Experimental results differ significantly from the calculated results in the amount of condensate and HCl obtained. The predicted HCl yield is greater than the amount obtained experimentally, whereas the predicted amount of condensate is less.

TABLE 9. Results With Simulated Feed

<u>Variable</u>	<u>Calculated</u>	<u>Experimental</u>
Wt% Gas	26.9	27.1
Wt% HCl	14.3	5.1
Wt% Residue	17.1	18.5
Wt% Condensate	24.1	41.9
Wt% Accountable	82.4	92.6
Volume Reduction, %	52.1	48.4

The calculated accountability for pyrolysis of the simulated feed (Table 9) is only 82.4% because accountability with individual components was low (Table 4). The lost material is probably condensate which accounts for part of the discrepancy between the experimental and calculated quantity of condensate. The results indicate that interaction between components during pyrolysis generates a considerable fraction of condensate and causes some retention of HCl, perhaps in the condensate.

Table 10 shows gas compositions for pyrolysis of the simulated waste mixture as calculated from results with individual components and as obtained experimentally. More unknowns (high molecular weight hydrocarbons) are obtained experimentally than expected. This corresponds to the observation that more condensates are obtained experimentally than calculated (Table 9). Interaction between feed materials appears to inhibit thermal cracking of the hydrocarbon products.

TABLE 10. Gas Composition From Pyrolysis of Simulated Feed

Method of Determination	Volume %							
	H ₂	CO ₂	C ₂ H ₄	C ₂ H ₆	CH ₄	CO	C ₃ H ₈	Unknown
Calculated	20.0	7.8	16.2	5.0	25.6	9.8	6.6	9
Experimental	24.0	4.4	12.4	5.5	20.6	9.0	6.1	18

Stability of char residues obtained from pyrolysis of the different feed components at 700°C was determined by differential thermal analysis (DTA) of the chars in an inert gas and air. Results obtained in argon and in air are shown in Figures 8 and 9, respectively, for char from pyrolysis of tygon. All char residues gave similar results. The char is stable in argon as shown in Figure 8 and starts burning at between 390°C and 460°C in air, Figure 9.

An experiment was conducted in which simulated alpha waste of composition given in Table 2 was gasified with air. Char from pyrolysis of simulated feed at 700°C was added to the reactor and was heated to 500°C. Air was then admitted at rates varying from 1 liter/min to 2 liter/min, and the power to the heater was cut. After burning the char for about 1 hr, simulated feed was added to the reactor in 50g batches at intervals of approximately 20 min. The gas generated was monitored continuously for CO concentration and intermittently for composition of all gases. Table 11 shows the composition of the product gases at 2 different times during the run with 2 liter/min air flow, and Figure 10 shows the CO concentration as a function during the same time.

TABLE 11. Product Gas Composition For Air Gasification of Simulated Waste Mixture

<u>Time After Startup, min</u>	<u>Volume % Concentration</u>				
	<u>H₂</u>	<u>CO₂</u>	<u>O₂-Ar</u>	<u>N₂</u>	<u>CO</u>
23	3.7	14	0.9	77.2	4.2
60	3.1	14	1.1	78.0	3.8

FIGURE 8. DTA of Char (From Pyrolysis of Tygon) in Argon

FIGURE 9. DTA of Char (From Pyrolysis of Tygon) In Air

FIGURE 10. CO Concentration in Gas Generated From Pyrolysis of Simulated Waste

Nitrogen concentrations are high (Table 10), which indicate some channeling occurred. Oxygen concentrations are low, particularly since most of the response is caused by the presence of Ar, perhaps 90% of the total concentration indicated by the gas chromatograph peak. Oxygen depletion was probably by reaction with combustible components in the gas phase.

Channeling in the refuse bed will result in a decrease in CO concentration as a result of both reaction of CO with oxygen that by-passes the combustion zone and less production of CO by reaction of char with oxygen and CO₂ in the combustion zone. Figure 10 shows that shortly after ramming the refuse bed, the CO concentration increased. The lag between the ramming and the CO concentration increase probably represents the time required for the gas to pass from the reactor to the continuous CO analyzer. The results indicate that channeling can be diminished by ramming, or agitation, of the top of the refuse bed.

A total of 250g of simulated feed was processed during the run. About 67g of a thick, viscous condensate was obtained, in addition to the solids that remained in the reactor at the end of the run (15g ash, 76g char, and 26g unreacted feed) and the gases produced. A volume reduction of 95% was achieved. Average processing rates were about 90 kg/hr m² at an air flow of 2 liter/min. Longer runs (at least 6 hr) are required to establish the processing rate for each airflow rate.

FACILITY DESIGN

The objective of the work discussed in this section was to perform the necessary preliminary investigations required for the detailed design of a pyrolysis-incineration system. Included in these investigations were (a) studies of processes similar to this alpha waste system, (b) discussions with manufacturers of equipment needed in this system, (c) the preparation of a list of design criteria for the system, (d) the construction of a preliminary flow diagram, and (e) the conceptual design of a pyrolysis-incineration reactor. Each of these tasks is summarized in the following paragraphs.

Similar Processes

In the early stages of this work, a number of processes similar to the pyrolysis-incineration system were studied to become familiar with the types and arrangements of hardware used and its operability. Some of these processes include:

Nuclear Waste Incineration System, General Electric
Contaminated Waste Recovery Facility, Atlantic Richfield Hanford Company
Garrett Pyrolysis Process, Garrett Research & Development Company, Inc.
Rocky Flats Incinerator, USAEC
Harvard University Air Cleaning Laboratory
Radioactive Waste Incinerator, U.S. Army Nuclear Defense Laboratory
Morgantown Gas Producer, U.S. Bureau of Mines
Wellman-Galusha Gasifier, McDowell-Wellman Company
Sasol Lurgi Pressurized Gasifier, South African Coal, Oil & Gas Corp.

In order to take a closer look at a number of these processes, four sites were visited in July. They included General Electric, Rocky Flats (Dow Chemical), U.S. Bureau of Mines, and the McDowell-Wellman Company. Some observations made from the study of these processes were:

- Incoming solid wastes require close inspection to remove as much undesirable material as possible before further processing.
- The average size of waste that is fed into a reactor can be 2 to 4 inches for a batch feed input, but should be around 1 inch for a continuous feed process.
- For our application, the most desirable type of shredding apparatus for the solid waste appears to be the counter-rotating system with cam-type blades and an automatic reversing feature to prevent jamming.
- Horsepower requirements for shredding of the solid wastes will be in the range of 0.8 to 1.5 horsepower-minute per pound.
- The feed hopper used for interim storage of shredded solid wastes will most likely be sealed at its inlet to prevent air leakage into the process.

- Screw feeding of waste into the reactor seems most promising of all techniques investigated at this point. The screw feeder will be a straight barrel, constant-pitch device, since the success of either a tapered screw or a variable-pitch screw has been questionable.
- A negative pressure air flow system is preferable to a positive pressure system, when considering the feeding of radioactive-bearing solid wastes.
- A modified version of the grate used in the Wellman-Galusha gasifier looks satisfactory to our needs.
- Careful design of the valving used at the ash removal area of the reactor will be necessary to prevent excessive leakage and wear. Numerous processes have experienced problems in this area.
- The most acceptable materials of construction for the grate system for ash removal are Inconel, Hastelloy C, and Hastelloy X. For the areas in contact with the off-gas stream, the most acceptable materials of construction appear to be the higher stainless steels in the 300 series (314, 316) and Hastelloy C.
- Allowance for expansion of the pyrolysis-incineration reactor due to thermal cycling is essential.
- A cyclone separator can be used to remove the majority of dry particulates in the off-gas stream. A fairly constant gas flow rate will be required to insure efficient particulate removal.
- Water scrubbers used to date for particulate removal have been, on the whole, unsatisfactory. It would be desirable to eliminate this component from our system if at all possible. The water scrubber causes a saturated off-gas stream which is a major cause of premature HEPA filter failure.
- Along with the shredder, the off-gas cleanup system is the other most critical item in a facility of this type. A system which eliminates the tars very early and runs completely dry seems most desirable. Methods other than aqueous scrubbing for chloride removal should be examined.

- The flow rate of the off-gas stream should be kept to a minimum. Methods other than excess air addition should be considered for the cooling of the off-gas stream.
- The HEPA filters currently in use in most installations of this type have a relatively short life and handle relatively low flow rates. A new filter has been recommended by G.E. personnel. It is a Flanders "Superflow" filter and will handle a larger flow rate over a long time period before pressure drop becomes excessive. These should be investigated further.

A concluding comment is that the technology for handling solid wastes is still in its infancy. In designing our process, each component should be designed and selected carefully, based on the design criteria and not on the component currently in use. The one main advantage of our visits to and studies of these various processes was to enable us to identify the problems in current hardware, which, in part, will help us in formulating our specific design criteria.

Manufacturer Contact

The second task undertaken was the contact with manufacturers handling the equipment which is required in our process. The following is a partial list of manufacturers contacted. It is anticipated that the selection of purchased components will come from this list.

<u>Product</u>	<u>Manufacturer</u>
Shredders, Choppers	Shred-Pax, TEB Ltd. Addison, IL
	Industrial Shredder and Cutter Co. Salem, OH
	Convey International, Inc. Roseburg, OR
	Tele-Com Industries, Corp. Oneida, NY
	W-W Grinder Corp. Wichita, KS
	Riety Manufacturing Co. Santa Rosa, CA
	Star Machinery Seattle, WA

<u>Product</u>	<u>Manufacturer</u>
Shredders, Choppers - Continued	Williams Crusher & Grinder St. Louis, MO Titan Engineering, Inc. Indianapolis, IN
Cyclone Separators	Joy Manufacturing Co. Seattle, WA J. A. Courtney Co. Portland, OR Manning Packing & Supply Portland, OR Aget Manufacturing Co. Adrian, MI Anderson IBEC Strongsville, OH Wright-Austin Co. Detroit, MI Donaldson Company, Inc. Corad Division Minneapolis, MN Hammond Machinery Builders Kalamazoo, MI Demco, Inc. Oklahoma City, OK Murphy Rogers, Inc. Huntington Park, CA The Western States Machine Co. Hamilton, OH
Air Lock Valves	Joy Manufacturing Co. Seattle, WA Prater Industries, Inc. Chicago, IL Wm. W. Meyer & Sons, Inc. Skokie, IL Detroit Stoker Co. Monroe, MI.
Hoppers, Feeders, Conveyors	J. O. Roisum Company, Inc. Boise, ID United Conveyor Corp. Chicago, IL Syntron Homer City, PA

<u>Product</u>	<u>Manufacturer</u>
Hoppers, Feeders, Conveyors - Continued	Meyer Machine Co. San Antonio, TX Link Belt Co. Seattle, WA Kalamazoo Conveyor Co. Kalamazoo, MI Joy Manufacturing Co. Western Precipitation Division Los Angeles, CA
Absolute Filters	Joy Manufacturing Co, Seattle, WA
High Temperature Seals	Borg-Warner Corp. Seattle, WA
Gas Burners	Johnson Gas Appliance Co. Cedar Rapids, IA The Barber Manufacturing Co. Bedford Heights, OH
Refractories, Glass Lined Pipe	Babcock & Wilcox Seattle, WA The Ceramic Coating Co. Newport, KY
Stack Sampling	Preferred Equipment Engineers, Inc. Seattle, WA
Exhausters, Blowers	The Spencer Turbine Co. Hartford, CT Aerovent, Inc. Piqua, OH Buffalo Forge Co. Buffalo, NY

Design Criteria

Discussions with manufacturers and with companies operating or designing gasification processes similar to this one were considered when compiling a list of design criteria for the pyrolysis-incineration process. The following is a list of these design criteria. This listing is not meant to be complete and is a compilation of the information gained to date.

1. The capital cost of the facility should be reasonable.

Design Criteria - Continued

2. The process utilized for the volume reduction of solid waste is one of pyrolysis-incineration. With minimal modifications, the hardware used for the process should be adaptable to the use of molten salts for ash removal.
3. The process will be carried out in a single, vertical vessel which is between one and two feet in diameter and four to eight feet of working length.
4. At least a portion of the inside walls of the reactor vessel will be lined with two layers of cast refractory, one to 980°C and the other to 1760°C.
5. The vessel will be designed with sufficient expansion joints to prevent failure due to thermal cycling.
6. Provisions will be made for the insertion of any fluxing agents to be used in the process.
7. The process will take place at near atmospheric pressure and at temperatures ranging from 100°C to 1400°C.
8. The vessel and peripheral hardware will be designed to minimize heat losses to prevent the need for excessive heat generation.
9. The facility should operate efficiently and with few moving parts, should be as automatic as possible, and switching in the operation of the facility should be interlocked to minimize operator error.
10. An inventory of radioactive materials will be kept at all necessary points in the process.
11. The vessel will be constructed to allow for ease of inspection and maintenance.
12. Adequate test ports will be provided.
13. The potential build-up of radioactivity in the walls of the reactor vessel will be investigated; any concentration points will be avoided.
14. The facility will be adaptable to a glove box operation.

Design Criteria - Continued

15. The reactor will operate under negative pressure. All openings into the reactor and other locations will be gasketed with high temperature sealing materials.
16. The unprepared waste must be thoroughly inspected by hand and by metal detection equipment to insure the removal of tramp metal and other noncombustibles.
17. The metal detection equipment will have a magnetic field of about 5000 gauss.
18. The waste will be shredded to a size of about 2.5 to 10 cm.
19. The shredder or chopper selected will be capable of accepting some small amount of tramp metal.
20. The mass flow rate of solid waste will be from 11.3 to 45.3 kg per hour.
21. Means of monitoring the proper mass flow rate of solids and residence time in the vessel will be provided.
22. The overall volume reduction of the solids in the process is expected to be from 10- to 50-fold.
23. The top level of waste in the reactor vessel should be distributed and compacted by a mechanical member.
24. The discharge of ash from the reactor vessel should not affect the continuous operation of the facility. The ash discharge system will be removable.
25. The grate in the vessel will be designed to insure a smooth and uniform ash flow.
26. Overall air flow for the system will be from the surrounding area to a glove box and from this glove box into the process, when radioactive wastes are used.
27. The air flow rate through the system should be as minimal and as uniform as possible to insure acceptable and consistent operation of the cyclone separator.

Design Criteria - Continued

28. The life of the absolute filters used in the process will be reasonably long.
29. The system will be designed to prevent the generation of any secondary waste.

Flow Diagram

Based upon the design criteria developed during this period, a preliminary flow diagram of the process was prepared. A step-wise procedure will be used to explain this process and the reader may refer to a schematic of this process shown in Figure 11.

The solid wastes will be brought into the facility either in boxes or in 55 gallon drums. These boxes will be emptied on to an open conveyor belt (in a hood). All the waste will then be inspected by hand to remove the majority of the undesirable materials. The waste remaining will then pass through a metal detection device to locate any small pieces of tramp metal; the undesirable materials will subsequently be removed. The waste with its containers will be fed into a shredder described in the comments under discussions of similar processes, shredded to the proper size, and then conveyed in a closed conveyor to a feed hopper. The feed hopper will have an airlock valve at its inlet to prevent air in-leakage. The feed hopper will be of sufficient size to insure a smooth and continuous flow of shredded waste, and at the base of the feeder, there will be an agitation mechanism to prevent bridging. The waste will then pass through a metering device to a straight barrel, constant-pitch screw feeder, which will transfer the waste into the reactor vessel.

Once in the reactor, the top level of the waste will be distributed and compacted to insure proper flow and to prevent any channeling. The waste will exist at four levels in the reactor. On the top will be the unreacted waste which is being distributed and compacted. Below that will exist the pyrolysis zone; the required heat for which is provided by a combustion zone below the pyrolysis zone. The bottom zone of the reactor is the ash. The ash is removed from the reactor through a rotating grate and airlock valve. Downstream of the valve, the waste is either directly deposited in final storage containers or is shipped to an actinide recovery

FIGURE 11. Flow Diagram of Pyrolysis Process

system. Air is bled in at the bottom of the reactor and the off-gas from the process is exhausted out the top of the reactor. A more complete description of the reactor concept follows.

The off-gas stream from the reactor is still under investigation, and a final conceptual design of the gas cleanup system has not been formulated. However, one concept that has merit will be described here. The off-gas is passed through a burner directly downstream of the reactor where oxygen is mixed with the off-gas at a volumetric rate of about 10% of the off-gas flow rate. A portion of the mixture is then burned, thereby hopefully destroying the tars in the off-gas stream. To date, no tests have been conducted to demonstrate the feasibility of this technique for a representative off-gas stream, but the destruction of tars by partial combustion in the Winkler coal gasification process has been successfully demonstrated. Once the destruction of tars has taken place, the off-gas is cooled prior to filtering by a cyclone separator. If properly designed, the cyclone separator will remove a large majority of the particulates from the off-gas stream. The off-gas stream is then passed through a water scrubber to remove the finer particulates and any unacceptable halides. From the observations made in previous studies, a water scrubber is not really desirable. If the halides can be released from the system, or if another method is found to remove them, the water scrubber can possibly be eliminated. The off-gas stream then passes through a bank of absolute filters, is mixed with excess air and burned in a gas afterburner.

Reactor Design

Conceptual design for the pyrolysis-incineration reactor occurs as a separate task and is described here in detail. (Refer to Figure 12 for a schematic of this reactor.) The reactor will be approximately 18-24 in. inside diameter and around 10 ft high. The exterior of this cylindrical vessel will be constructed of carbon steel. The vessel will be lined with two layers of refractory in the working zones. The inside layer will be designed for service up to 1760°C, and the outer layer to 480°C. The walls of the vessel will be fitted with an appropriate number of test ports for use during the operation of the reactor.

Solid waste will be fed into the top of the vessel through a screw feeder. Once in the reactor, the upper zone of waste material will be compacted and distributed with a mechanism which extends through the top of the vessel. This device to be constructed of Hastelloy C or Hastelloy X, consists of a perforated plate on a vertical shaft that has members extending downward from the plate. The plate with extensions is rotated and translated vertically on the top of the material bed, thus compacting the entire bed and distributing the top of the bed. The plate is rotated with a variable speed drive and is vertically translated through a ball screw and servo motor. This design allows the plate to move up to down and also in an oscillating mode of variable frequency. It is anticipated that this flexibility will be necessary to adapt the mechanism to different waste types and flow rates.

At the lower end of the reactor, an ash discharge-air inlet system is shown in Figure 12. This system consists of a rotating grate, a grate drive, air inlet, collection hopper, slide valve, and a rotary air lock valve. The ash rests on top of the continuously rotating grate (also to be constructed of Hastelloy C or Hastelloy X) which channels the ash down through the center of the unit. This grate is turned by a variable speed drive which is coupled to the grate by a set of bevel gears. Air is allowed to filter up through the grate via distributed openings. The ash particles which are too large to pass through the grate are forced to the outside where they are crushed between the rotating grate and a cast metal wear ring, which is easily replaceable due to the anticipated wear caused by the highly abrasive ash. The crushed ash then passes down the outside of the grate to a collection hopper where it joins the smaller ash particles which passed through the center of the grate. The collection hopper will be only slightly tapered to insure flowability of the ash. Below the hopper will be a slide valve which is normally open. This valve will be used only when maintenance is required on the rotary airlock valve which passes the ash from the reactor to a collection container. By operating in this manner, the slide valve can be closed and the rotary valve removed without affecting the continued reaction in the vessel itself.

FIGURE 12. Schematic of Pyrolysis-Incineration Reactor

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