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OPERATION OF THE RADIOACTIVE ACID DIGESTION TEST UNIT (RADTU)

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Abstract

The Radioactive Acid Digestion Test Unit (RADTU) has been constructed at Hanford to demonstrate the application of the Acid Digestion Process for treating combustible transuranic wastes and scrap materials. The RADTU with its original tray digestion vessel has recently completed a six-month campaign processing potentially contaminated non-glovebox wastes from a Hanford plutonium facility. During this campaign, it processed 2100 kg of largely cellulosic wastes at an average sustained processing rate of 3 kg/hr as limited by the water boiloff rate from the acid feeds. The on-line operating efficiency was nearly 50% on a twelve hour/day, five day/week basis.

Following this campaign, a new annular high rate digester has been installed for testing. In preliminary tests with simulated wastes, the new digester demonstrated a sustained capacity of 10 kg/hr with greatly improved intimacy of contact between the digestion acid and the waste. The new design also doubles the heat transfer surface, which with reduced heat loss area, is expected to provide at least three times the water boiloff rate of the previous tray digester design. Following shakedown testing with simulated and low-level wastes, the new unit will be used to process combustible plutonium scrap and waste from Hanford plutonium facilities for the purposes of volume reduction, plutonium recovery, and stabilization of the final waste form.

1. INTRODUCTION

The acid digestion process is a chemical oxidation process for converting combustible organic wastes to gaseous effluents and stable solid residues. The digestion medium is sulfuric acid, heated to about 250°C. The sulfuric acid carbonizes and partially oxidizes the waste; but the primary oxidant is nitric acid, which is added continuously to the digester at a rate proportional to the waste addition rate. The composition of the off-gas depends on the waste composition but typically consist of CO_2, H_2O, and HCl (from oxidation of wastes containing chlorinated plastics) and SO_2, NO, N_2, and H_2O from decomposition of the acids. The off-gases must be treated to remove entrainment and the bulk of the corrosive acidic vapors. The residue is separated from the sulfuric acid medium by periodically withdrawing a portion of the acid slurry and distilling the acid back to the digester.

Development of the acid digestion process began at the Hanford Engineering Development Laboratory (HEDL) in 1972.[1] The process was scaled from the initial 1 to 5-ℓ laboratory-scale experiments to a 200-ℓ spherical glass digestion vessel in a nonradioactive pilot plant called the Acid Digestion Test Unit (ADTU), Figure 1. The testing in this unit confirmed the basic feasibility of the process and established the following advantages and disadvantages of the system:

Advantages:

° It handles a wide variety of waste materials, including leaded gloves.

° The reactions are readily controlled by adjusting the waste and nitric acid feed rates or the digestion temperature.

° It is a low temperature process that retains plutonium in a water-soluble, non-refractory form.

° Generation of tars, soot, and flyash are avoided.

Disadvantages:

° Relatively low processing rates.

° Limited materials of construction.

° Presence of a neutron moderating liquid which limits the design options and plutonium inventory for criticality safety.

Appropriate design features can take advantage of the positive factors and minimize disadvantages in a system for treating particular radioactive wastes. The application wherein the process has its greatest overall advantage is the treating of combustible wastes or scrap having a high transuranic content.

Consequently, the ADTU digester vessel was modified to a dimensionally favorable tray digester (Figure 2) for an evaluation of its applicability to plutonium-contaminated wastes. In this digester, waste is added periodically to a horizontal tray containing a 25-mm depth of hot sulfuric acid recirculated by ~~gas~~ air lift from a separate, annular heating vessel. Carbonized waste leaves the tray through an underflow weir draining to the heating vessel, where the digestion is completed by the continuous addition of nitric acid. Satisfactory demonstration of this concept led to the construction and operation of the Radioactive Acid Digestion Test Unit (RADTU), which is the subject of this paper.

2. RADTU DESCRIPTION

A detailed description of the RADTU facility has been presented in a previous paper,[2] and the following discussion will be limited to a brief summary of its major features. The facility is shown schematically in Figure 3. It was constructed in 1977 in an existing plutonium facility at Hanford and involved the following modular systems:

- A waste storage and assay area (not shown) where incoming wastes were stored and assayed for plutonium content. An assay system capable of analyzing low-density wastes for both multigram and trace (below 10 nCi/g) plutonium levels is currently in use.
- A feed preparation module where wastes were hand sorted to remove noncombustibles and shredded by a low-speed shredder into approximately 3-cm pieces. Shredded wastes were weighed and dumped into a conveyor that discharged them into the hopper of a hydraulically-operated ram feeder. The ram feeder compressed and extruded the waste through a 10-cm-diameter pipe into a Teflon-lined expanded spool piece where a blast of compressed nitrogen blew apart the compacted waste and distributed it onto the tray of the tray digester. The conveyor, feeder, and nitrogen blast were synchronized by an adjustable timer to provide a consistent but variable waste flow to the digester.
- The digestion module, which contains the tray digestion vessel, an annular heating vessel, a wire-mesh mist eliminator, and emergency overflow vessels. All vessels used geometrically-favorable dimensions as a major factor in criticality control.
- A solids recovery module where the waste residues were withdrawn periodically from the digester to heated pots where they were dried at 400°C. The acid vapors were returned to the digester off-gas system for recovery.

- An off-gas treatment module where the digester off-gases were condensed and scrubbed with cooled, recirculating acid in the presence of added air to oxidize and recover the SO_2 and NO as dilute sulfuric and nitric acids. The off-gas leaving the primary scrubbing tower was heated above the dew point and passed through parallel high efficiency (HEPA) air filters to remove entrained radionuclides. The off-gas was then passed through a secondary scrubber to remove residual SO_2 and NO_x before being filtered again and discharged to the facility stack.
- An acid fractionation module where the recovered dilute acids from the scrubbers were concentrated for recycle to the digester. The fractinator off-gases contained most of the water and hydrochloric acid generated during the digestion process, along with unrecovered nitric acid and NO_x; it was heated and filtered through HEPA filters before discharge to a separate RADTU stack.

All of the modular systems described above, except for the assay module and the secondary off-gas scrubber, were installed in gloveboxes for contamination control. Other modules included a liquid storage and transfer module and a chemical makeup module.

The equipment within the modules was constructed of austinitic stainless steel (low temperature process lines and vessels), glass-lined steel (the digester vessels and the acid fractionator), tantalum (the digester tray), and Teflon (gaskets, sleeves, and special fittings). Tantalum-covered borosilicate glass foam inserts (Figure 4) were also installed in the digester tray vessel heads to provide shock wave attenuation in the unlikely event of a deflagration or detonation[3] and also below the tray for neutron absorption. A similar neutron absorber originally installed above the tray was removed because it interfered with waste distribution on the tray. The central portion of the annular heating vessel also contained a borosilicate foam glass insert for neutron absorption and to prevent liquid intrusion.

3. RADTU OPERATING HISTORY

The RADTU was completed in June, 1977, and underwent shakedown testing with synthetic waste mixtures for several months before introduction of actual waste. The shakedown tests resulted in several important improvements and modifications to the digester and its auxiliary systems. These included the addition of the nitrogen jets in the ram feed system to distribute the waste charge onto the tray; the addition of a nitrogen jet to spray tray acid over the incoming waste;

doubling the number of residue evaporation pots to four to increase the residue handling capacity and installing an improved mist eliminator in the final scrubber off-gas line to reduce acid carryover into the off-gas blower. The new mist eliminator also included provisions for steam backflushing to remove stearic acid deposits that can build up over long periods of time from digestion of certain plastics.

Following the shakedown runs, the RADTU was operated on potentially contaminated, non-glovebox waste from the plutonium facility for six months before being shut down for the installation of a high rate digester vessel, described in Section 5. During this time, it processed 2100 kg of mostly cellulosic materials at an average rate of 2.7 kg/hr and a sustained digestion rate of 3 kg/hr. The resulting dried residue weighed 325 kg and had a bulk density of 800 kg/m^3.

The new high rate digester is currently undergoing shakedown testing before RADTU is returned to radioactive service. Current plans are to use the modified RADTU to process combustible plutonium scrap and waste from Hanford plutonium facilities for the purpose of volume reduction, plutonium recovery, and stabilization of the final waste form.

4. RADTU PERFORMANCE CHARACTERISTICS WITH THE TRAY DIGESTER

During the operating period with the tray digester, RADTU was operated on a five-day basis, sixteen hours per day. Waste addition was done in twelve hour periods, followed by completion of digestion and eight hours of standby while the temperature was maintained. The system was completely shut down and allowed to cool over weekends and holidays. The demonstration with both synthetic and actual low-level wastes showed that the acid digestion process can be operated on a sustained, reliable basis and that the process is very stable and easy to control.

4.1 RADTU Performance Highlights

The following highlights summarize the performance of RADTU during the six-month campaign with low-level wastes.

- Sustained digestion rates of 3 kg/hr of predominantly cellulosic wastes were achieved with instantaneous rates as high as 4 kg/hr.
- Downtime was about 51% of the operating time. Approximately 12% of the downtime was caused by low temperature (excess water in the system); 50% by equipment upgrading, repair, and replacement; 18% for cleanout of the tray; and the remainder for non-process related causes. Design changes are expected to significantly reduce the downtime in future runs.

- The digester operated for extended periods of time with little or no operator adjustment required.
- The ram feeder performed in a completely satisfactory manner during the campaign.
- Noncombustible materials and residue collected on the digester tray, and tray cleanout was required about once a month. Up to 10% of the tray residue was noncombustible hardware and glass that had not been detected during the manual sorting operation.
- The airlift used to circulate heated acid from the heating vessel to the tray performed adequately, although it contributed to heat loss between the two vessels. It was necessary to maintain the heating vessel at about 260°C to assure a tray temperature of at least 240°C. The air lift height was at about the maximum for sustained flow, and plugging problems in the inlet tube due to low flow occurred frequently.
- The residue drying operation was relatively slow and inefficient with the dilute slurry feed that was used. Some corrosion problems were noted in the transfer/off-gas line.
- Only two pump failures (both scrub recirculation pumps) occurred during the campaign.
- The acid fractionator system was operated to maximize the product (recycle) acid concentration, with a resulting recovery of about 50 to 75% of the nitric acid in the fractionator feed. Overall, about 40% of the NO leaving the digester was collected in the recycle acid.
- The major factors limiting the capacity of the digester were the available heat input to the system, which was sufficient to boil off only about 12 to 15-ℓ of water per hour from the incoming acids, and the restrictive geometry associated with the critically-safe tray digester which hindered the acid-waste contact.
- Three potential failures (surface cracks and minor spalling) of the glass-lined steel equipment were observed at the end of the campaign. Two were caused by localized mechanical stress; the third was the result of too much heat flux through a surface covered with settled sludge. All of these are correctable with appropriate design and operating procedures. There was no observed chemical attack of the glass surfaces. Other materials of construction performed satisfactorily.

- Conversion of the dried residue to a borosilicate glass was demonstrated in a 15-cm-diameter canister. Graphite was added to volatilize sulfate as SO_2 at 900°C. The sulfate-free residue was then blended with glass formers and melted at 1100°C. The final product had a density of 2600 kg/m^3 and contained 33 wt % of residue oxides.

4.2 RADTU Material Balance Flowsheet

The average performance of the RADTU during the low-level waste runs is summarized in the material balance flowsheet shown in Figure 5. The flowsheet was derived from measured inputs, analyses of off-gases and recovered acid, and certain assumptions regarding the behavior of nitric acid and sulfuric acid in the digester and off-gas system. The following assumptions were made:

- Sulfuric acid evaporates from the digester at the rate of 0.02 mol/mol of off-gas. The remaining sulfuric acid recovered in the recycle system is derived from SO_2 produced by the digestion reaction.
- The remainder of the oxygen required to digest the waste comes from decomposition of HNO_3 to NO and N_2. The relative amounts of each decomposition product are determined by the ratio of moles of oxygen required to the moles of HNO_3 used. (Decomposition of HNO_3 to produce NO_2 occurs with excess nitric acid, but this was avoided by using just enough nitric acid to color the digester off-gas with a trace of NO_2.)
- Oxidation of NO and SO_2 to NO_2 and SO_3 was assumed to occur only in the scrubbers; only unoxidized NO and SO_2 were assumed to leave the scrubbers in the off-gas.
- The water content in the scrubber off-gas was based on the equilibrium vapor pressure at the off-gas temperature.

Typically, about 43 moles of HNO_3 and 31 moles of H_2SO_4 were fed to the digester per kilogram of waste digested. An estimated 38% of the sulfuric acid in the feed was evaporated or entrained with the off-gases; the remaining 62% contributed to the digestion reaction and left the digester as SO_2. About 87% of the nitric acid reacted with the waste to form NO, the remainder to form N_2. Virtually all of the SO_2 and sulfuric acid leaving the digester was recovered in the two scrubbers, but only 40% of the NO from the digester was recovered as HNO_3. Most of the unrecovered nitric oxide was lost as HNO_3 and NO_xCl in the fractionator overheads, but approximately 10% of it left the second scrubber unabsorbed. As discussed earlier, the operation of the fractionator was based primarily upon reducing the amount of water in the recycle acid rather than maximizing the recovery efficiency of HNO_3.

4.3 RADTU Energy Balance

Theoretical heat-balance calculations were made for the acid digestion module to establish more clearly the relationship between the flowsheet parameters and the net heat input requirement. For simplicity, the overall reactions were broken into the component parts: adjusting reactant temperatures to 25°C; evaporation of the reactant acids at 25°C; decomposition of the acids to produce oxygen; reaction of the oxygen with the waste to produce CO_2, H_2O, and HCl; and heating the reaction products to the vapor off-gas temperature of 200°C.

In RADTU, the net heat input to adjust the incoming acid temperatures to 25°C and to evaporate them at that temperature can be approximated by the following relationships:

Component	Heat of Vaporization, kJ/kg
H_2O	2 640
HNO_3	790
H_2SO_4	1 200

The heat input required to decompose the acids to SO_2, NO, N_2, H_2O, and O_2 and to heat all of the products (except O_2) to 200°C is given by the following:

Reactant	Products	Heat of Decomposition, kJ/kg
HNO_3	NO, H_2O, O_2	1 790
HNO_3	N_2, H_2O, O_2	313
H_2SO_4	SO_2, H_2O, O_2	2 110

The heat of combustion of the waste with oxygen at 25°C to yield gaseous products at 200°C can be approximated as -410 ± 40 kJ/mol of oxygen required. The oxygen demand for waste materials varies from about 40 mol/kg for cellulosics and polyvinyl chloride (PVC) to about 100 mol/kg for latex rubber and polyethylene. The low-level waste mixture used in the RADTU runs had an oxygen demand of about 44 mol/kg and a resulting heat of combustion of about -18,040 kJ/kg.

The final heat input requirement is that required to heat the acid dilution water, the volatilized and unreacted H_2SO_4, and the airlift air from 25 to 200°C. The required heat input is given by the following:

Component	Sensible Heat Input, kJ/kg
H_2O	332
H_2SO_4	162
Air	177

Application of these factors to the flowsheet conditions of Figure 5 results in a net heat input requirement of 12,200 kJ/kg of waste, or 36,500 kJ/hr at the average processing rate of 3 kg/hr (see Appendix for detailed calculations). The heating vessel is provided with a nine-zone heating-mantle furnace with a total heat output capability of 29.7 kW (107,000 kJ/hr), for an overall heat input efficiency of 34%. This efficiency is unusually low for mantle-heated equipment and undoubtedly means that substantial heat losses occurred in the tray vessel and the surmounting air-cooled mist eliminator.

The preceding heat balance is dependent on the assumed reaction mechanisms, which can vary for different wastes and for different operating conditions. The results of a parametric calculation for digestion of a synthetic waste mixture in RADTU are shown in Figure 6 to illustrate the effect of different assumptions concerning the nitric and sulfuric acid utilization and the nitric acid decomposition mechanism on the required net heat input.

5. ANNULAR HIGH RATE DIGESTER

Although the tray digester proved to be an adequate solution to the criticality safety design problem, its capacity limitations led to the development of an improved annular design that both doubles the heat transfer area and provides intimate contact between the shredded waste and the acid. A schematic drawing of the new digester as currently installed in RADTU is shown in Figure 7. The tray digester vessel has been replaced with an annular vessel similar to the previous heating vessel, which has been retained. The two vessels are connected at both the top and the bottom. Circulation between the vessels is provided by an airlift in the heating vessel discharging into the top of the digester vessel. The feed system uses the same ram feeder as the original installation; a timed blast of nitrogen disperses the extruded waste as it enters the top of the annular section, and internal air lifts spray hot acid over the waste as it falls into the acid solution.

The new digester has been tested in ADTU and has demonstrated a sustained capacity of 10 kg/hr with synthetic waste materials and no recycle acid. The upper rate limit with recycle acid has not been established but is expected to be in the range of 8 to 10 kg/hr as limited by the water boiloff requirements and the available heat input to the two vessels.

Other system improvements are also being tested during the initial shakedown runs with the new high rate digester. These include improvements in the sorting and feed preparation to reduce the amount of noncombustible material inadvertantly added to the digester, use of a continuous centrifuge to concentrate the residue slurry before drying, and replacement of the heated HEPA filters between the two off-gas scrub columns with a fume coalescer to reduce the carryover of sulfuric acid to the second scrubber.

Secondary features of the RADTU test program with the new high rate digester will be the evaluation of methods to minimize noncombustible waste addition to the digester and to remove such material that does enter the system. To accomplish the first objective, the incoming waste will be screened to remove coarse particulate material, and an air classifier will be tested for separating noncombustible materials from the shredded waste. The use of the bottom interconnecting line as an easily flushed collecting trap will also be tested. (A screen has been provided in the bottom of the annular digester to keep bulky noncombustibles out of the bottom of the digester and the collecting trap.)

6. FUTURE OPERATIONS

Following shakedown testing of the RADTU with the new high rate digester, the RADTU will be evaluated for use as a production facility to process accumulated plutonium scrap and waste from Hanford plutonium facilities. Laboratory and pilot plant work is continuing to extend the applicability of the acid digestion process to other potential waste forms such as organic liquids, ~~in~~ ion exchange resins, and unusual plutonium scrap materials. An agitated trough dryer is under development as a replacement for the current pot residue dryers and will be installed in RADTU pending successful demonstration in ADTU.

7. REFERENCES

[1] COOLEY, C. R., LERCH, R. E., "The Acid Digestion Process for Treatment of Combustible Wastes", Management of Plutonium-Contaminated Solid Wastes (Proc. OECD-NEA Seminar, Marcoule, France, 1974), OECD, Paris (1975) 172.

[2] LERCH, R. E., ALLEN, C. R., BLASEWITZ, A. G., "Treatment of Alpha-Bearing Combustible Wastes Using Acid Digestion", Treatment, Conditioning and Storage of Solid Alpha-Bearing Waste and Cladding Hulls (Proc. OECD-NEA and IAEA Tech. Seminar, Paris, 1977), OECD, Paris (1978) 149.

[3] ALLEN, C. R., COWAN, R. G., GRELECKI, C. J., "Acid Digestion and Pressurization Control in Combustible Radwaste Treatment", ASME preprint 78-ME-17 (Joint ASME/CSME Pressure Vessels & Piping Conference, Montreal, 1978), ASME, New York (1978).

APPENDIX

Detailed Heat Balance Calculations for RADTU Low-Level Waste Runs

Basis: 1 kg Waste

Component	kg	Heat input, kJ/kg	Total kJ
1. Vaporize incoming acid streams:			
H_2O	5.01	2 640	13 226
HNO_3	2.71	790	2 141
H_2SO_4	3.04	1 200	3 648
2. Decompose acids:			
HNO_3 to NO	2.36	1 790	4 224
HNO_3 to N_2	0.35	313	110
H_2SO_4 to SO_2	1.88	2 110	3 967
3. Oxidize waste:			
Waste	1.00	-18 040	-18 040
4. Heat unreacted feed components to 200°C:			
H_2O	5.01	332	1 663
H_2SO_4	1.16	162	188
Air + N_2	5.75	177	1 018
		TOTAL	12 145

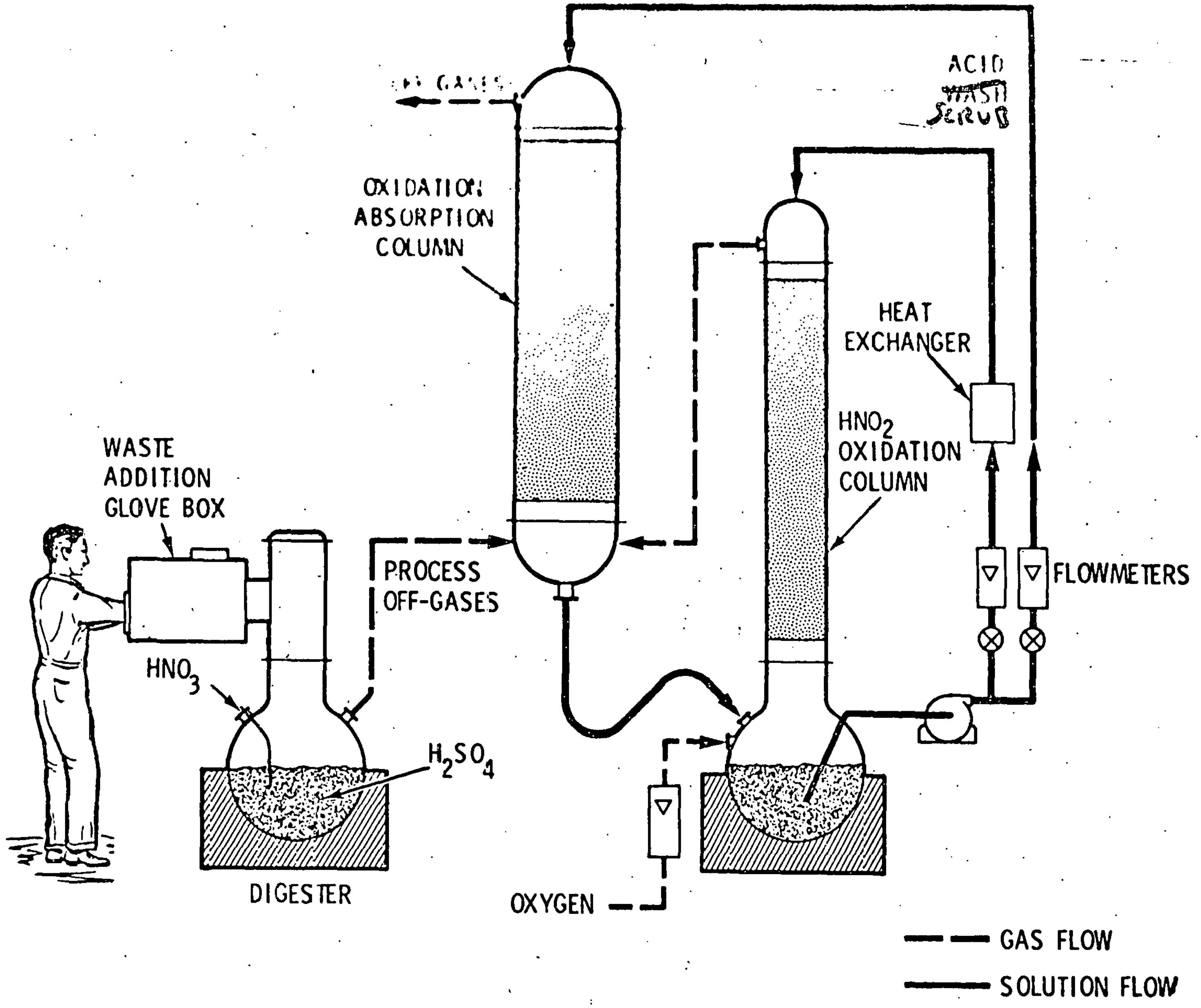

FIGURE ~~4.0-1.~~ 1 Non-Radioactive Engineering Scale Acid Digestion Test Unit (ADTU) 745760-1

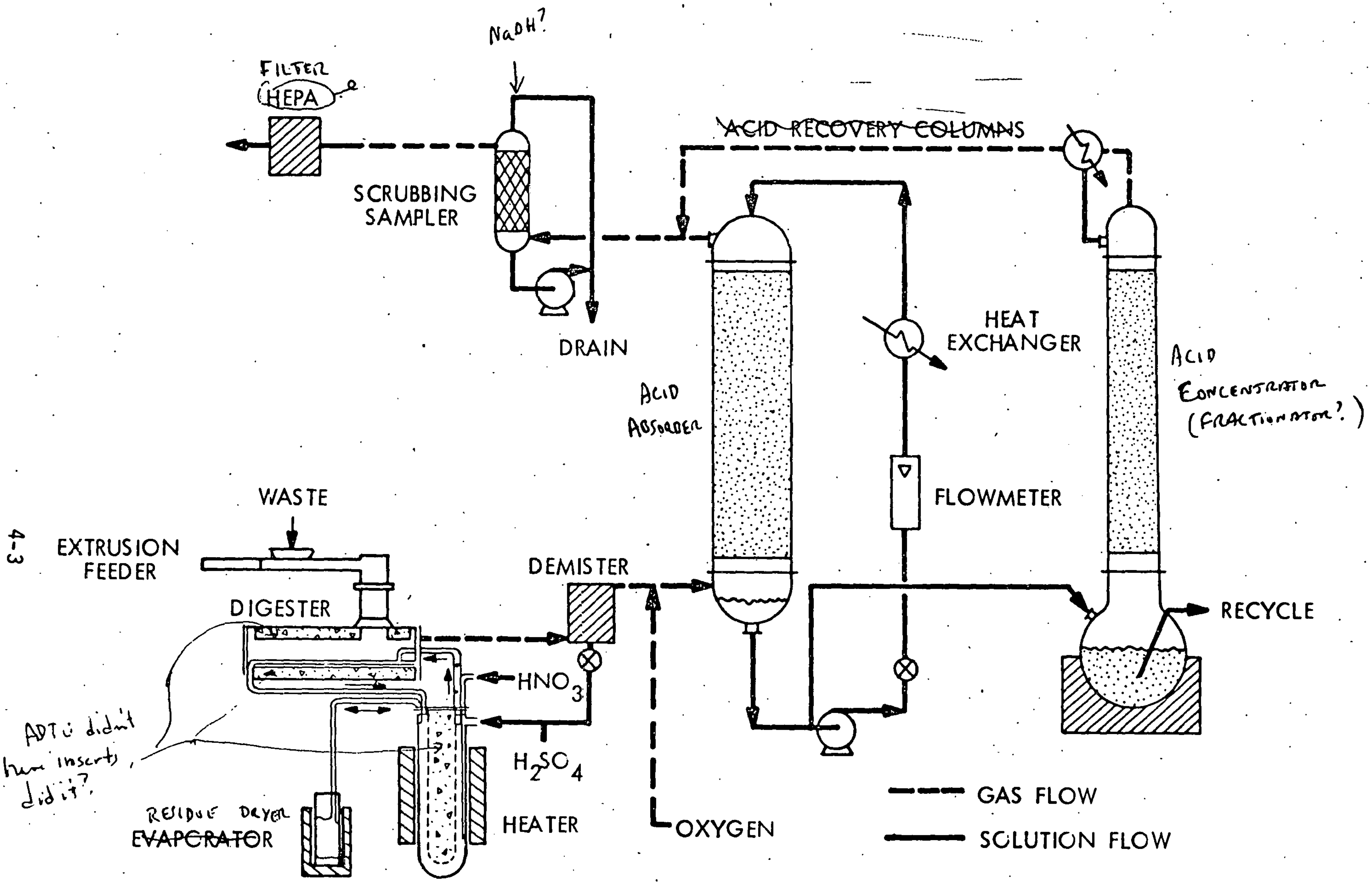

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FIGURE ~~4.0-2~~ 2. Modified Non-Radioactive Acid Digestion Test Unit (ADTU).

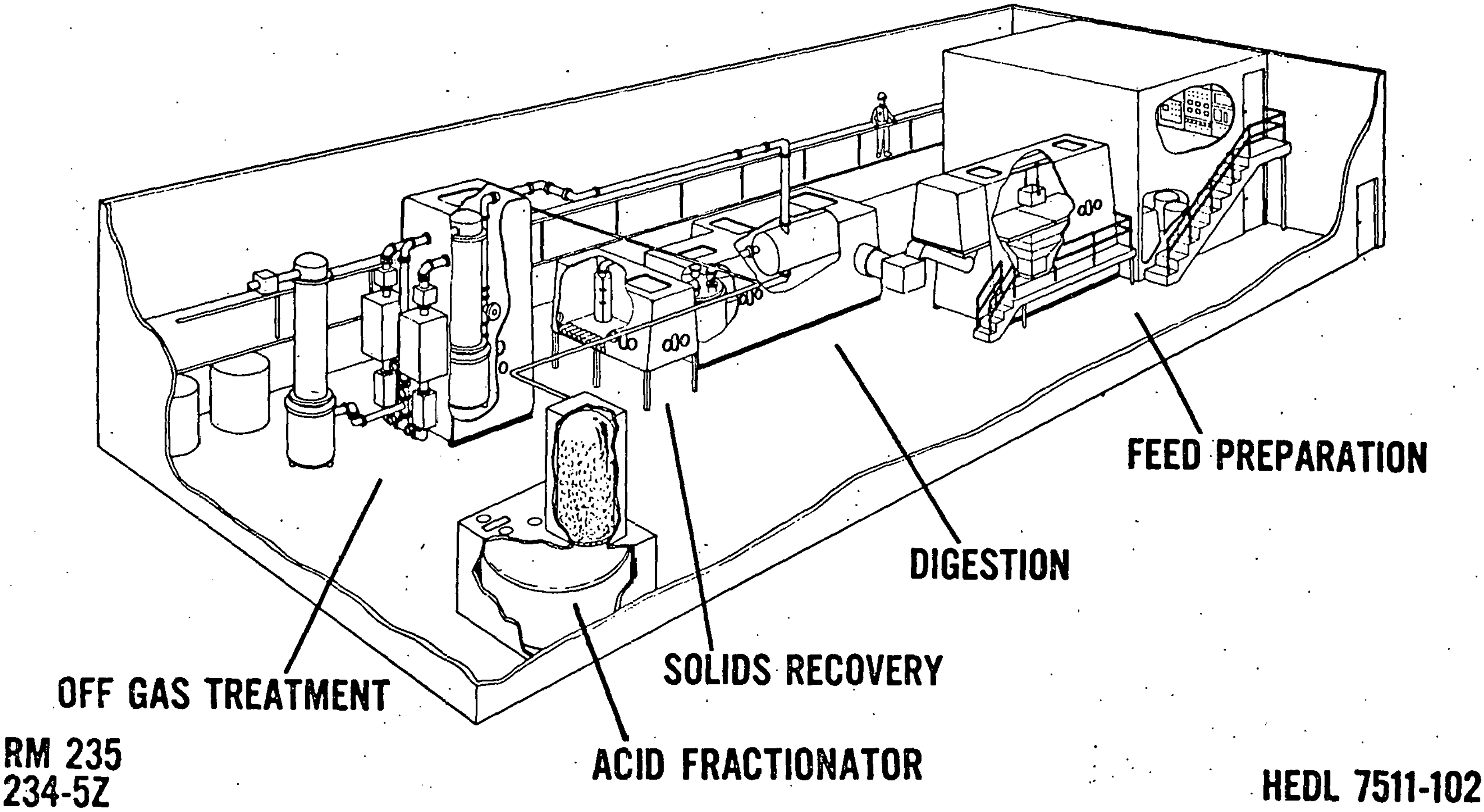
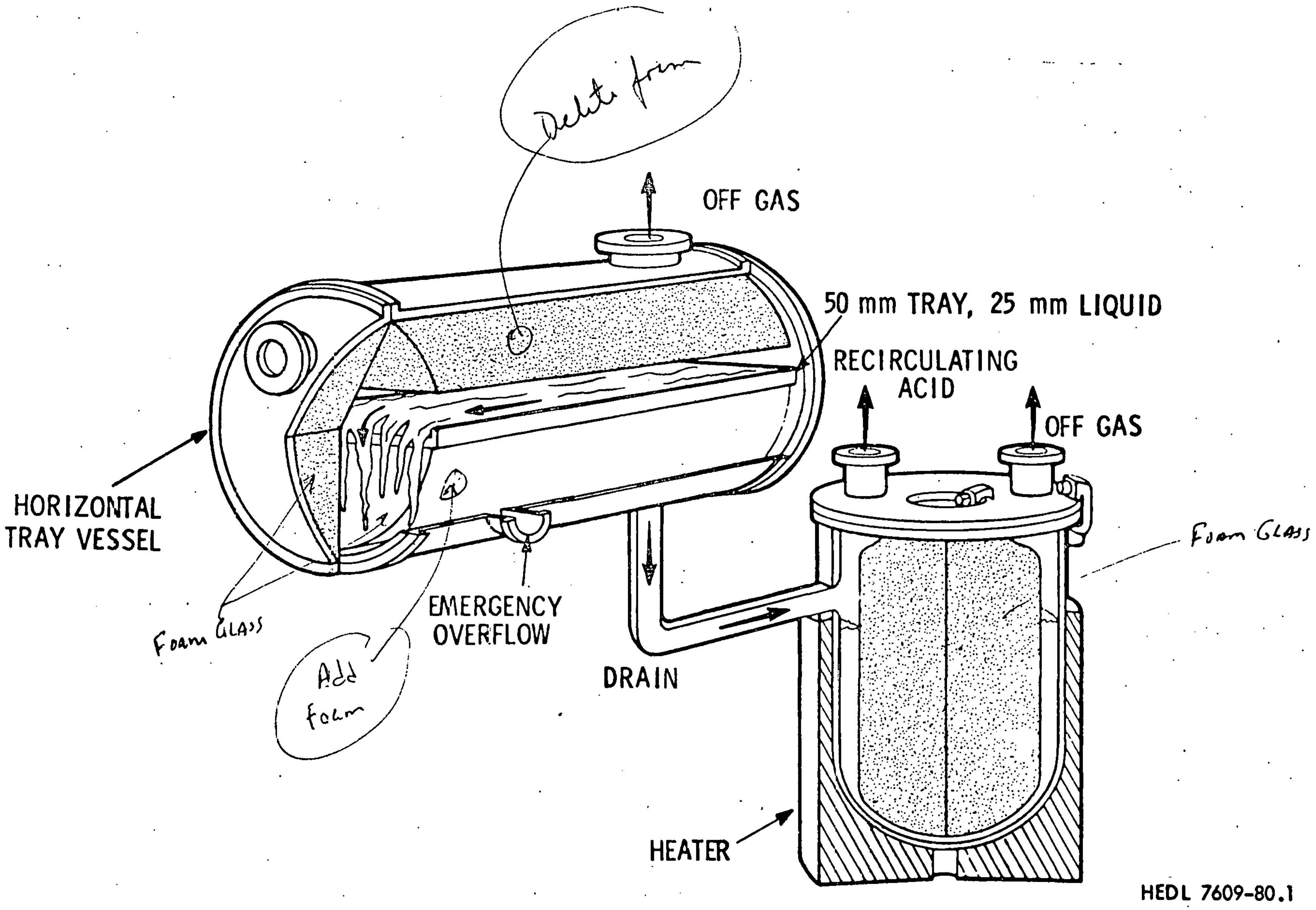


FIGURE ~~5.0-3.~~ 3 RADTU Facility Layout.



4 Schematic of Digester System Showing Borosilicate Foam Glass Inserts.
FIGURE ~~6.1-1.~~ ~~Normal Digester Operation~~.

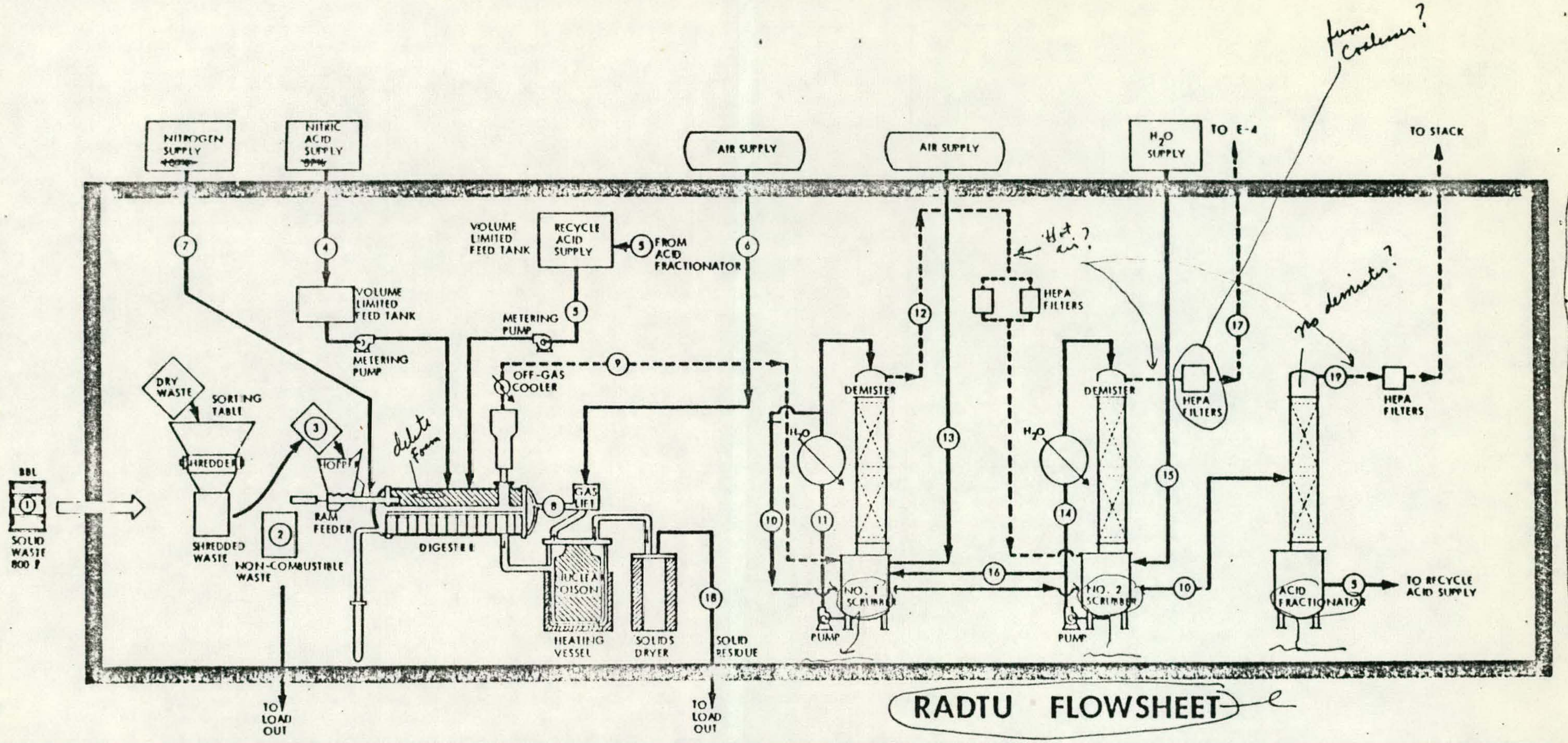


FIGURE 3. RADTU FLOWSHEET

RADTU FLOWSHEET e

LINE NUMBER	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
DESCRIPTION	DRUMMED WASTE	NONCOMB. WASTE	COMBUSTIBLE WASTE	NITRIC ACID	RECYCLE ACID	GAS LIFT AIR	NITROGEN JET	DIGESTER LIQUID	DIGESTER OFF-GAS	NO. 1 SCRUB. DISCH	NO. 1 SCRUB. RECIRC	NO. 1 SCRUB. OFF-GAS	DILUTION AIR	NO. 2 SCRUB. RECIRC	NO. 2 SCRUB. WATER ADD.	NO. 2 SCRUB. DISCH	NO. 2 SCRUB. OFF-GAS	SOLID RESIDUE	FRACTIONATOR OFF-GAS
RATE UNITS	Kg/DAY	Kg/DAY	Kg/DAY	Kg/DAY	Kg/DAY (LITERS/DAY)	Kg/DAY	Kg/DAY	LITERS/MIN.	Kg/DAY	Kg/DAY (LITERS/DAY)	LITERS/MIN.	Kg/DAY	Kg/DAY	LITERS/MIN.	Kg/DAY	Kg/DAY	Kg/DAY	Kg/DAY	Kg/DAY
QUANTITY OR RATE	126	20	106*	316	761 (544	526	49	RECIRC.	1760	1634 (1362)	20	1110	400	20	517	552	1075	15	873
PRESSURE, GAGE	0	0	0	0	20 PSIG	5 PSIG	250 PSIG	0	-7 IN OF H_2O	20 PSIG	20 PSIG	-9 IN OF H_2O	0	20 PSIG	0	20 PSIG	-20 IN OF H_2O	0	-15 IN H_2O
TEMPERATURE °C	25	25	25	25	70	25	25	230	110	70	40 - 70	40	25	30	20	30	30	30	104°C
COMPOSITION -WT. %																			
Paper									SO_2 = 6.9%	H_2O = 69%									HNO_3 = 12.3%
Polyethy		GLASS = 35%	85% PAPER	HNO_3 : 57%	H_2SO_4 = 40%	N_2 = 75%	N_2 = 100%	H_2SO_4 = 91%	H_2O = 36.2%	H_2SO_4 = 18%		NO = 2.7%	N_2 = 75%		H_2O = 100%	HNO_3 = 6.7%	CO_2 = 17.3%	$CaSO_4$	HCl = 0.2%
PVC		METAL = 65%	POLYETHYLENE 10%	H_2O : 43%	HNO_3 = 12%	O_2 = 23%		H_2O = 9%	H_2SO_4 = 6.6%	HNO_3 = 12%		CO_2 = 16.8%	O_2 = 23%			H_2O = 92.2%	N_2 = 70.3%	SiO_2	H_2O = 87.5%
GLASS			5% PVC		H_2O = 48%	Ar = 1%			HCl = 0.1%	HCl = 0.1%		Ar = 1.1%	Ar = 1%			H_2SO_4 = 0.7%	O_2 = 8.3%	$ZnSO_4$	
METAL			(6% ASH)						Ar = 0.4%			O_2 = 9.4%				HCl = 0.2%	NO = 1.1%	$Fe_2(SO_4)_3$	
H_2SO_4									NO = 6.5%			HCl = 0.1%					H_2O = 1.7%	$Ti(SO_4)_2$	
HNO_3									CO_2 = 10.6%			H_2O = 1.5%					Ar = 1.1%		
H_2O									N_2 = 25.8%			N_2 = 68.1%							
SO_2									O_2 = 6.9%			SO_2 = 0.3%							

HCl

NO

N_2

O_2

Ar

FIGURE 5 MATERIAL BALANCE FLOWSHEET FOR RADTU LOW-LEVEL WASTE CAMPAIGN

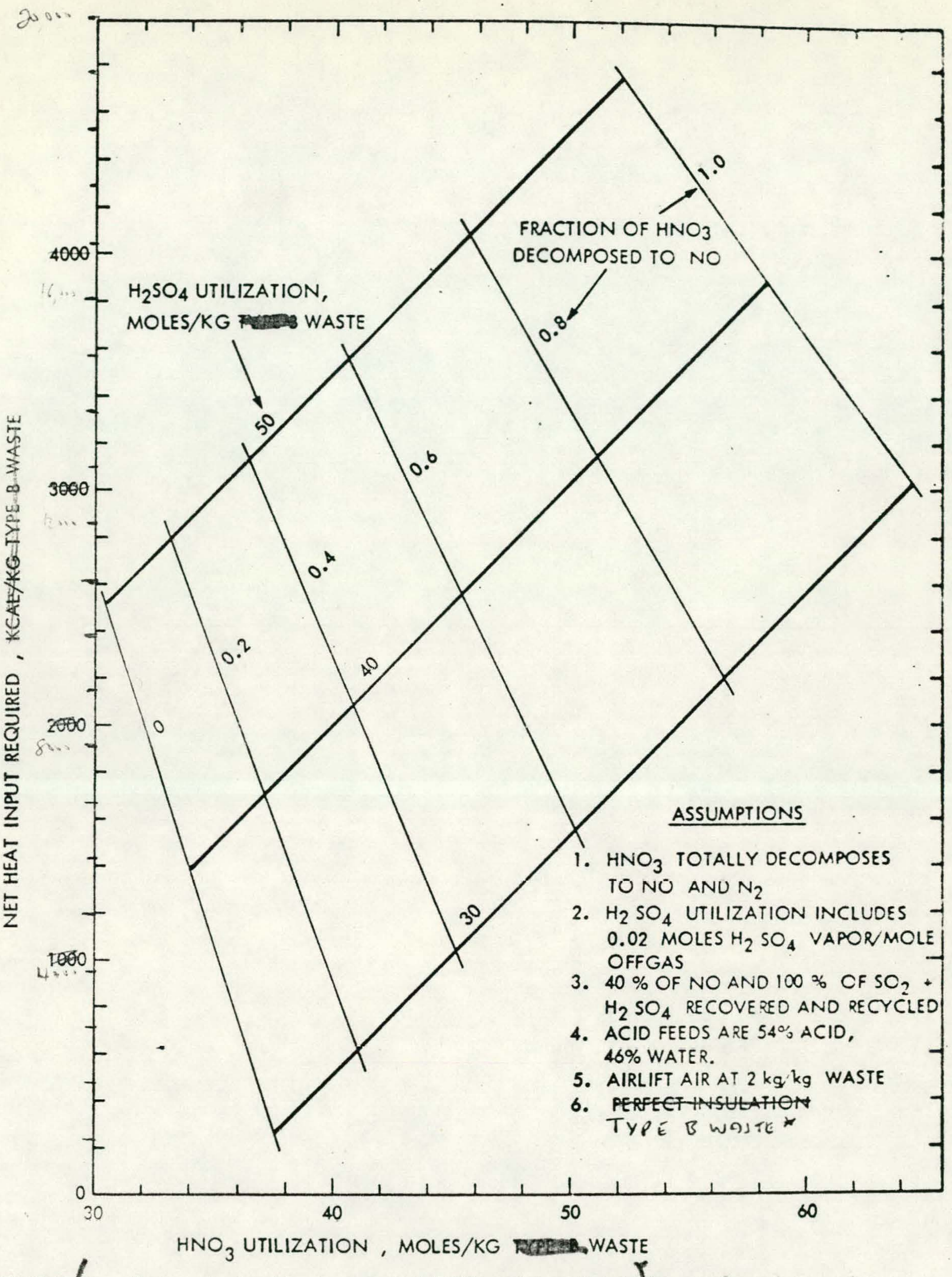


FIGURE 6. Parametric Study of Acid Digestion Heat Input Requirements as a Function of Acid Utilization and Reaction Stoichiometry.

* Type B waste = 40% cellulose
5% latex
10% neoprene
10% Hypalon
20% PVC
15% Polyethylene

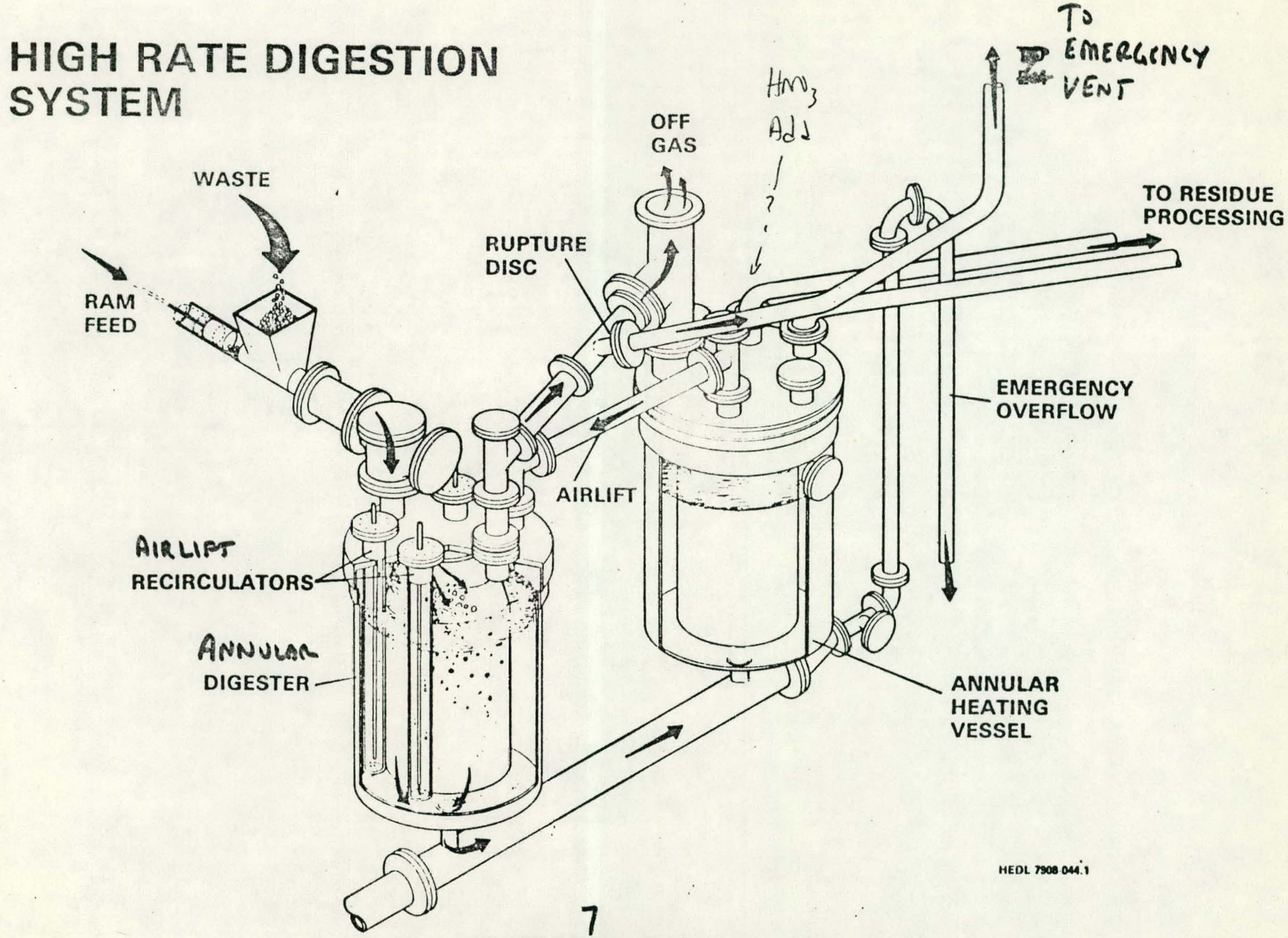


FIGURE ~~18~~7. High Rate Digester System