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MASTER

RADIOACTIVE ACID DIGESTION TEST UNIT
NONRADIOACTIVE STARTUP OPERATIONS

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by

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Introduction

Acid digestion is a process being developed at the Hanford Engineering Development Laboratory (HEDL) to reduce the volume of alpha-contaminated combustible waste while converting the waste into a stable nonreactive residue. Typical waste materials, such as hypalon, neoprene, latex, PVC, polyethylene, ion exchange resin, paper and wood are digested as depicted in Figure 1.

These materials are added to hot (250°C) sulfuric acid where they are rapidly charred. Concentrated nitric acid is subsequently added to oxidize the charred material to carbon dioxide gas. Exhaust gases are treated to recover and recycle nitric and sulfuric acids. A small amount of unreactive residue containing the plutonium remains in the acid.

Following initial testing in a nonradioactive pilot plant, a radioactive demonstration plant has been constructed at Hanford to process combustible alpha waste generated from Hanford operations. The plant, the Radioactive Acid Digestion Test Unit (RADTU), was completed in September, 1977 and has been undergoing nonradioactive shakedown tests since that time.

Summary

The Radioactive Acid Digestion Test Unit (RADTU) will process 5 kg/hour of combustible solid waste. It is designed to handle almost all solid combustible waste found in plutonium processing with plutonium contamination levels up to and including scrap. The RADTU is designed with special safety features to safely contain high masses of fissile materials and to safely handle unusual materials and reactive chemicals which may find their way into the waste.

Nonradioactive operating experience to date has been very satisfactory. RADTU has been operated for extended runs on both a 24-hour per day basis as well as on a one shift per day basis. Some minor operating problems have been encountered as expected in a shakedown operation. In general, solutions to these have been readily found. These are discussed in some detail in the following sections.

Process Description

RADTU is currently capable of processing 5 kg/hour of combustible waste to a dry, nonreactive solid. The only materials exiting the process are the residue and the filtered off-gas. No secondary liquid waste stream is generated. The major process elements of RADTU, shown in Figure 2, are Feed Preparation, Digestion, Solids Recovery, and Off-Gas Treatment. Design features and initial operating experience of this system will be discussed in the following slides.

Feed Preparation

Waste is brought to the RADTU in 200 liter drums where it is assayed using a random driver coincidence counter (Figure 3). It is then hand sorted and shredded in a low speed counter rotating shredder (Figure 4). The shredded waste feeds from a feed hopper of constant cross sectional area onto a flighted belt conveyor. The hopper is designed so that waste will not bridge as it feeds onto the conveyor. The conveyor belt contains pleats which minimizes the waste falling off the belt (Figure 5) and flights across the pleats allow the delivery of a small increment of waste at a time to the extrusion feeder. This prevents bridging across the small extruder feed chamber.

Operating experience on the feed system has been very satisfactory. Initially, there was some problem with galling of metal parts in the extruder cylinder. The problem was solved by using a softer metal (bronze) in the ram head follower and by slightly relaxing the tolerances of the follower. The extruder (Figure 6) now has over 100 hours of operation without problems.

Digestion

The acid digester is shown schematically in Figure 7. Extruded waste falls onto a 5 cm deep tray filled with hot sulfuric acid. The tray is made of foamed silica and is 150 cm long by 75 cm wide. Acid is maintained on the tray at 2.5 cm depth by means of an overflow at one end. Waste is prevented from overflowing the tray by use of an underflow weir. Figure 8 shows the waste introduction points, the tray, the recirculating acid, and the underflow weir. Acid overflows the tray and is returned to an annular heating vessel where its temperature is controlled at 250°C by heating with an electric heating mantle. A gas lift is used to recirculate acid back onto the tray. This system has no moving parts. Nitric acid and makeup sulfuric acid are added to the annular vessel. No operating problems have been encountered from digester operation. A tendency of waste to build up at the feed end of the tray has been noted and techniques are being tested to improve distribution of the waste on the tray in order to increase the throughput capability of the digester.

The digester has been operated at 5 kg waste/hour. It was initially designed for 100 kg/day throughput in order to process 300 Area waste at Hanford. HEDL has since been asked to process all Hanford area glovebox wastes and capacity increase studies are underway.

Off-Gas Treatment

The digester off-gas system consists of three packed columns and associated heated HEPA filters as shown in Figure 9. The first column performs multiple functions. Air is injected into the off-gas stream prior to entering the column. This serves to oxidize NO_x to NO_2 so that it can be absorbed in the column. NO_2 also oxidizes SO_2 to SO_3 for absorption in the same column. At the same time, recirculating acid in this column is cooled and serves to condense and scrub water and HCl from the off-gas stream. The result is a mixed acid product (about 6-1/2 to 7 molar), mostly nitric and sulfuric acid with a small amount of HCl. This mixture serves as the feed for an acid fractionator.

After passing through the first column, the off-gases are heated with hot air injection and filtered through heated HEPA filters before passing into the second column. This arrangement is shown in Figure 10. The HEPA filters are a spacerless type with a packed glass seal. After several hundred hours of operation, they were tested and found still operating at high efficiency.

To date, no problems have been encountered with heated HEPA filter operation with the exception of an air heater burning out when air flow was inadvertently stopped with the power still on the heater.

Fresh water is added to the second column to scrub out residual acid. This weak acid stream is then fed to the first column. Gases passing through this tower are again heated, filtered, and exhausted to the building ventilation system. The liquid from the first column passes through a retention tank to the fractionator shown in Figure 11 where water is evaporated to strengthen the acid to 12 to 14 molar combined nitric and sulfuric acid. The off-gas from the fractionator consists mostly of water vapor and essentially all the chloride from the feed in the form of equimolar mixture of HCl and HNO_3 . The strong acid (approximately 13 N) from the fractionator then is recycled to the digester.

The net input and output to the system is as follows: raw waste feed, makeup nitric acid at 1 to 2 kg of HNO_3 per kg of waste, and a very small amount of sulfuric acid. The only waste streams exiting from the system are gases and the residue from the process. If chloride is present in the waste, it is exhausted as HCl gas. There is no liquid waste stream. The major product is the waste residue which is inert and is suitable for immobilization in glass. Any plutonium in the residue can be readily extracted with dilute nitric acid.

During early runs, nitric acid recovery from the system was low. It was found that this was caused by insufficient oxygen in the off-gas to oxidize NO to NO_2 . The problem was solved by substituting air for nitrogen

in the gas lift in the product vessel. This introduced air at the start of the process and has promoted better oxidation of NO to NO₂ for subsequent nitric acid recovery. Nitric acid consumption is now within previous estimates and should stay within 1 to 2 kg of HNO₃ per kg/waste.

Sulfuric acid carryover from the digester in the RADTU system was higher than that experienced in nonradioactive pilot plant operation. This problem was partially solved when additional air was added to the system, but higher sulfuric acid mist concentrations were still found in the RADTU off-gas.

A Brinks[®] mist eliminator has since been installed in RADTU and is being tested to solve this problem. The Brinks[®] unit was not required for operation in the cold pilot plant.

The net sulfuric acid consumption in the system is now very low. With acid recycle the sulfuric acid is collected in the off-gas, concentrated, and returned to the digester. Very little fresh sulfuric acid is required. (Some of the sulfuric acid comes from oxidation of sulfur in rubber and paper in the waste.)

Solid Recovery

The digester acid slurry is periodically vacuum transferred to one of the two residue pots. Here the sulfuric acid is evaporated leaving the solids from the waste digestion. These solids are mainly silicate and sulfate salts of any cations present in the feed material. The acid evaporated from these pots is returned directly to the heating vessel via the same transfer line. This system has no moving parts. Transfers are made by vacuum from the digester to the residue pots.

Support Equipment

In general, support equipment has functioned well following the initial shakedown of the equipment. The most noticeable change required

in this area was with a magnetically coupled pump used for chemical transfers. Some difficulty was experienced in starting this pump with higher viscosity fluids. The problem was solved by using a stronger magnetic coupling. Since this change, no starting problems have been encountered.

Operating Summary

A summary of shakedown test runs is shown in Figure 12. The system has been operated both continuously and intermittently for extended periods (intermittent operation included 12 hours of operation, including startup and shutdown and 12 hours on standby). Longer continuous operating runs are planned after extension of the building nitric acid piping to provide a continuous supply of nitric acid. A separate acid resistant off-gas stack was installed during February to permit RADTU operation without producing a liquid waste stream. The separate stack was used during the last two test runs together with the recycle acid system. The runs went very smoothly. The only problem encountered was the wetting of a HEPA filter with sulfuric acid mist. A Brinks[®] mist eliminator is being tested to eliminate this problem.

It was possible to set the acid recycle feed to the digester, the waste feed, and the nitric acid makeup to the digester so that no operating adjustments were required for a periods of six to eight hours of continuous operation. With this kind of operating stability and automatic control the process can be operated with a minimum of personnel.

Safety Studies

Extensive safety studies have been completed on the acid digestion process. The FSAR has been issued and approved. The RADTU has been designed with restrictive geometry to operate safely with high fissile masses (a minimum critical mass of 3.2 kg Pu²³⁹). The inadvertent introduction of reactive chemicals as well as normal waste materials was considered in the design. The digestion system has been designed to safely contain pressurizations up to a full scale vapor phase detonation even though such an occurrence is virtually incredible.

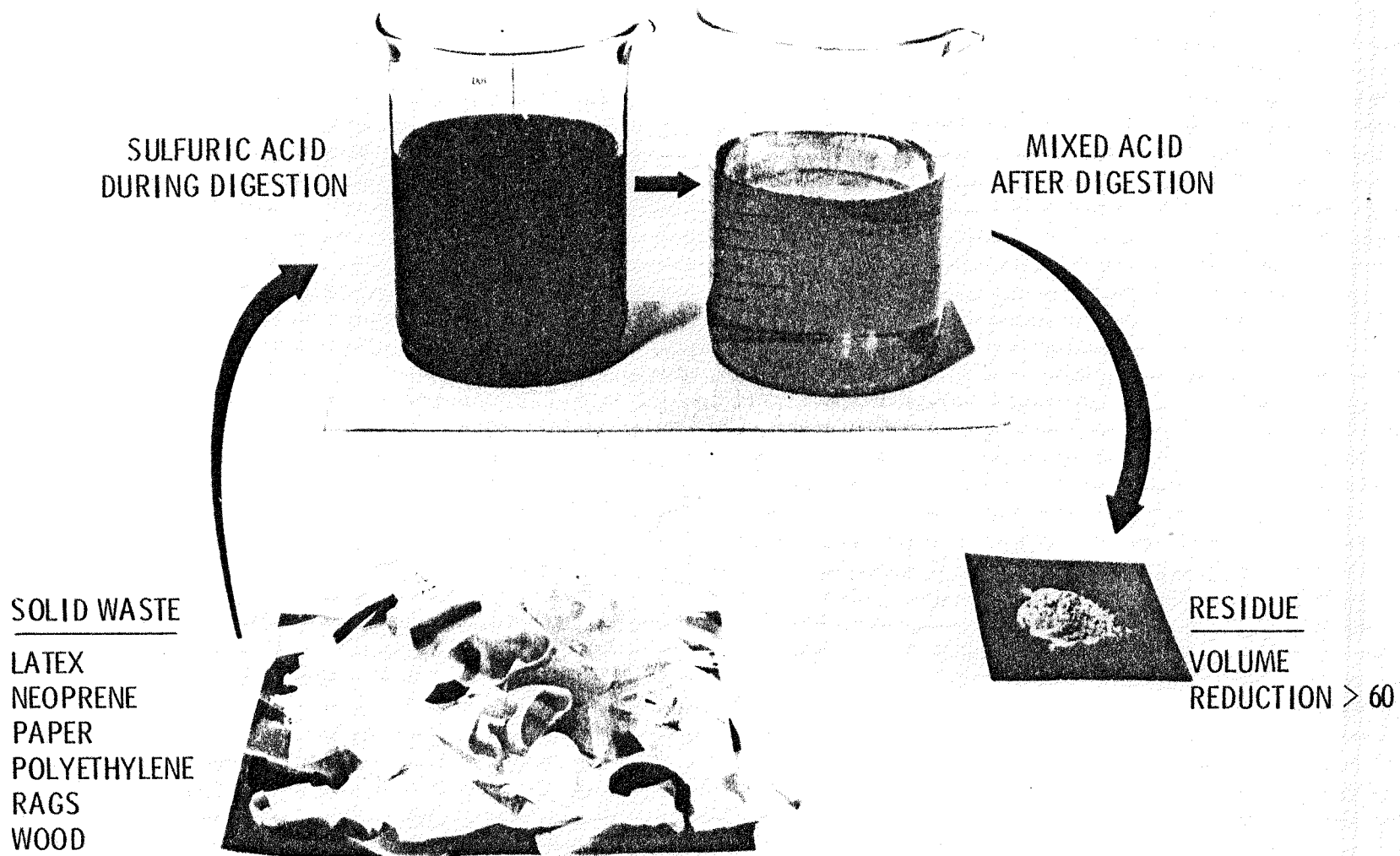
Development Schedule

Hot operation with Low Specific Activity (LSA) waste is scheduled for September, 1978. This will be followed by six to eight months of two shift operation. During this same period, tests will be conducted and an assessment made of the equipment change required to increase the throughput capacity of RADTU. Meanwhile, plutonium recovery equipment will be designed and procured for installation in RADTU at the end of this period. RADTU will be shutdown for several months to install plutonium recovery equipment as well as residue immobilization equipment. Some modification is also expected to be made to increase the throughput capacity to handle glovebox waste from all of Hanford. This is not expected to be a major modification since both the head end and off-gas systems are over sized.

Following these changes, RADTU will be restarted and operated with both waste and scrap from Hanford operations.

In summary, operating experience with RADTU has been very satisfactory. Some minor operating problems have been encountered. The solutions to these have been relatively straightforward and it is expected that the plant will meet or exceed all objectives.

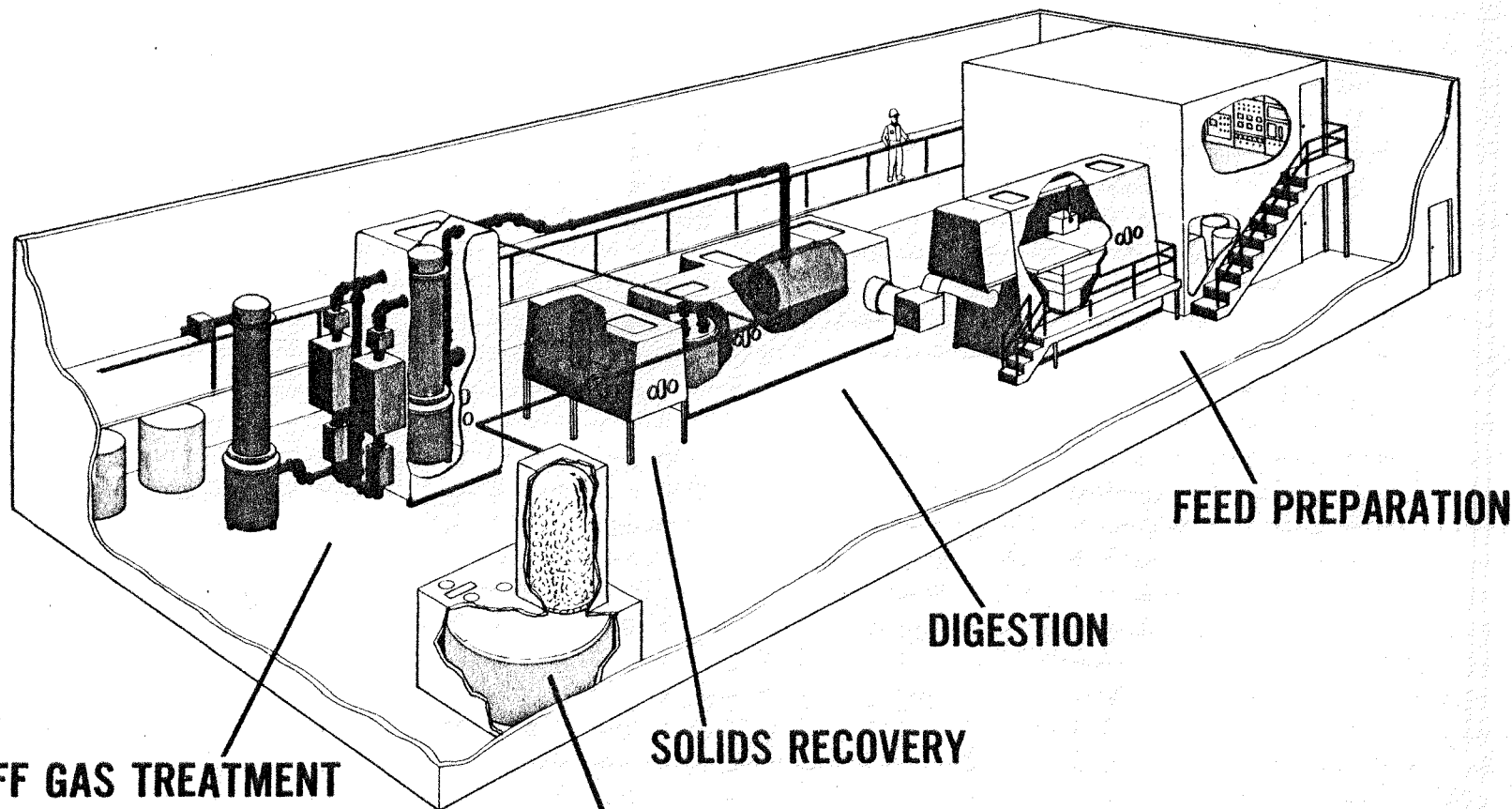
ACID DIGESTION OF SOLID WASTE



VOLUME: 60 ML
WEIGHT: 10 GR

FIGURE 1

RADIOACTIVE ACID DIGESTION TEST UNIT (RADTU)

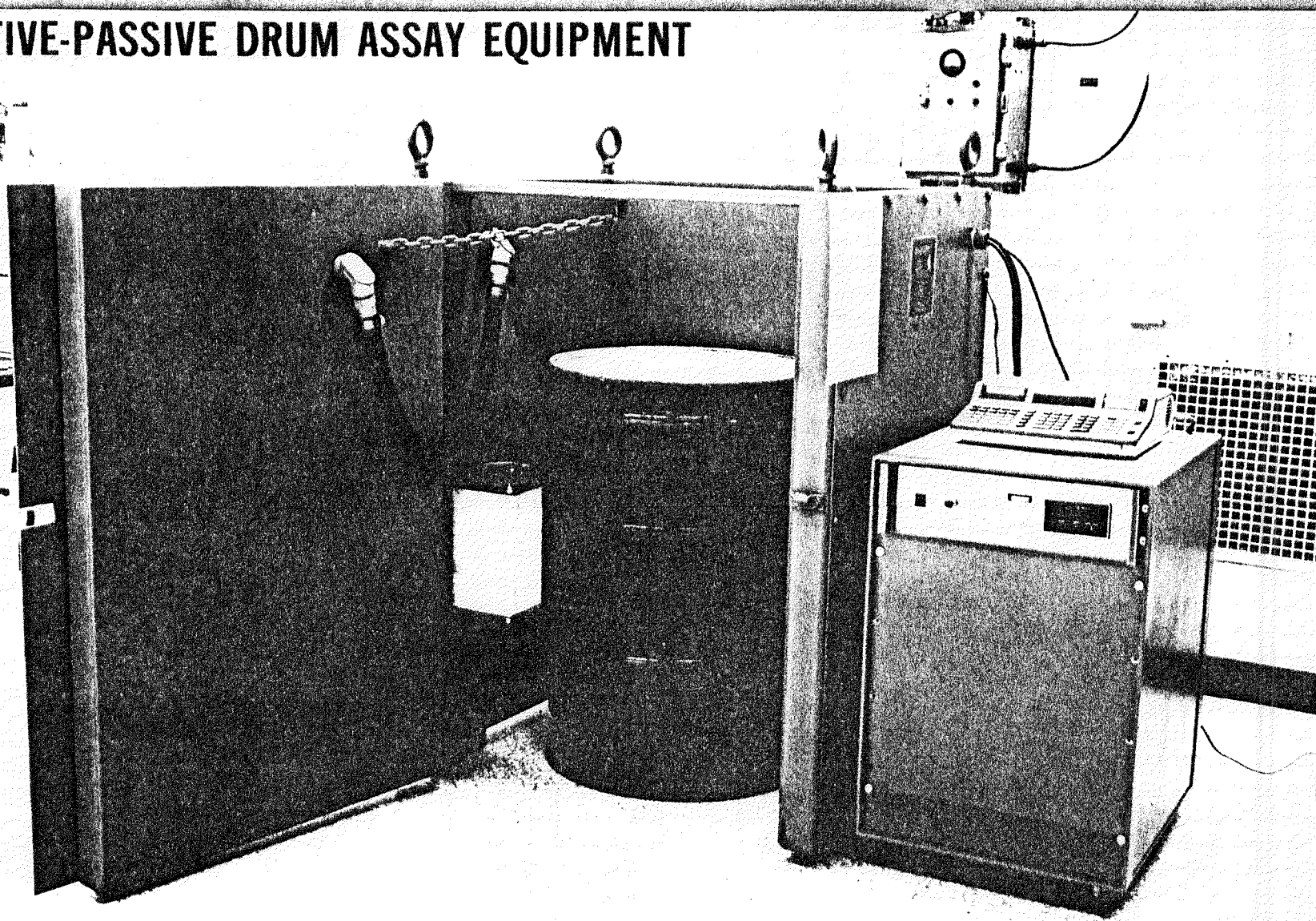


RM 235
234-5Z

HEDL 7511-102

FIGURE 2

ACTIVE-PASSIVE DRUM ASSAY EQUIPMENT



HEDL 7804-191.2

FIGURE 3

SHREDDER WITH COUNTER ROTATING BLADES

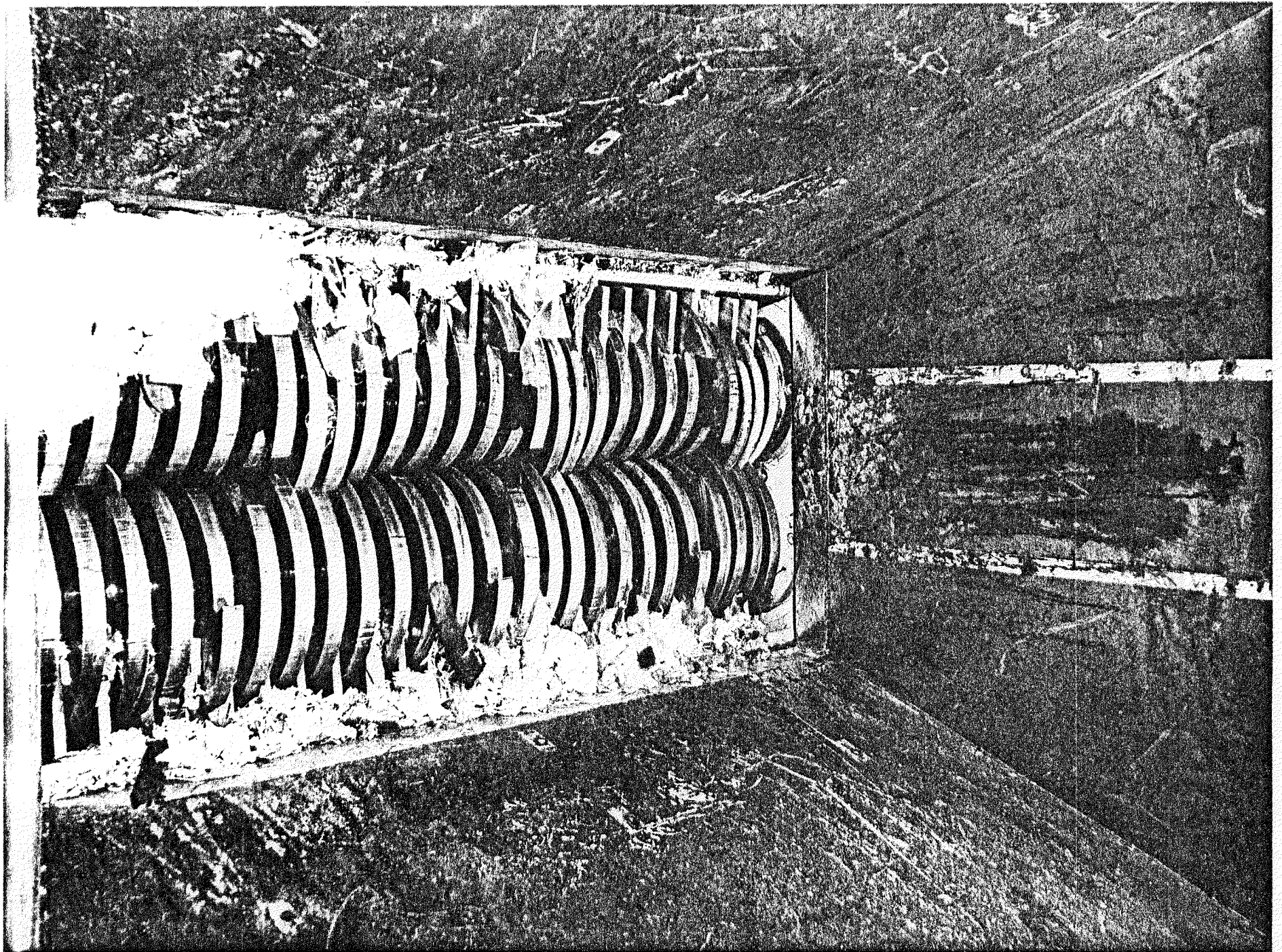


FIGURE 4

RADTU WASTE FEED AREA

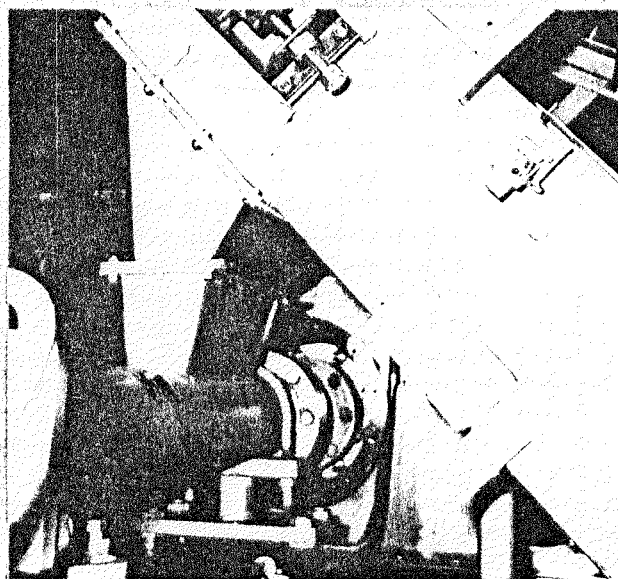
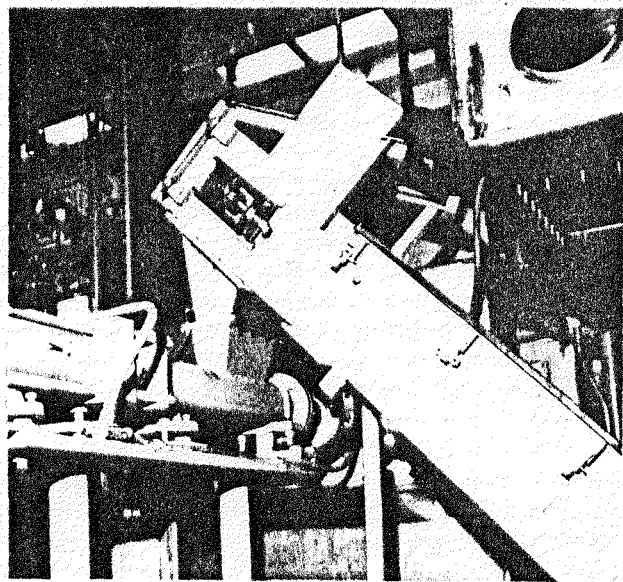
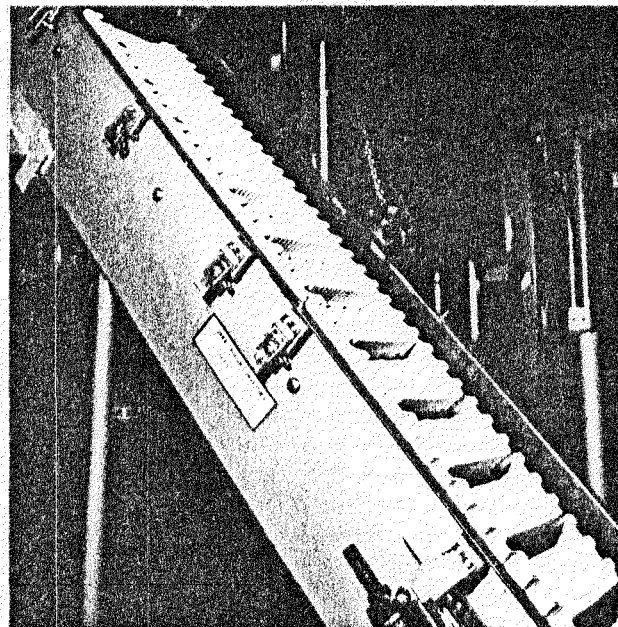
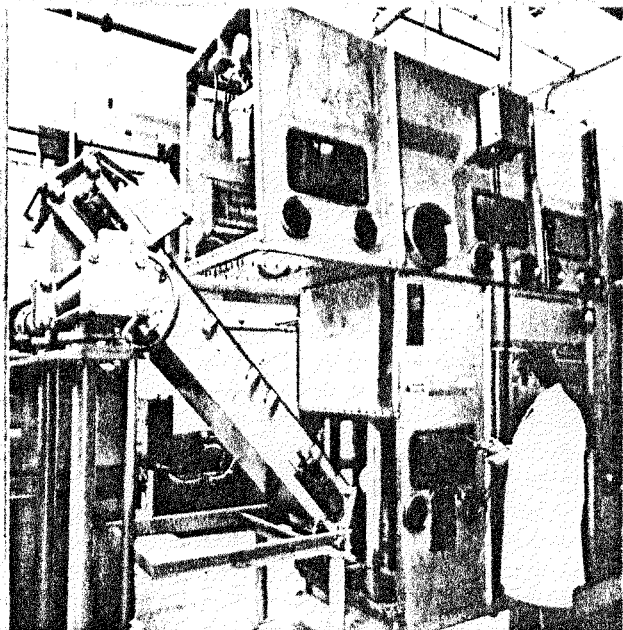


FIGURE 5

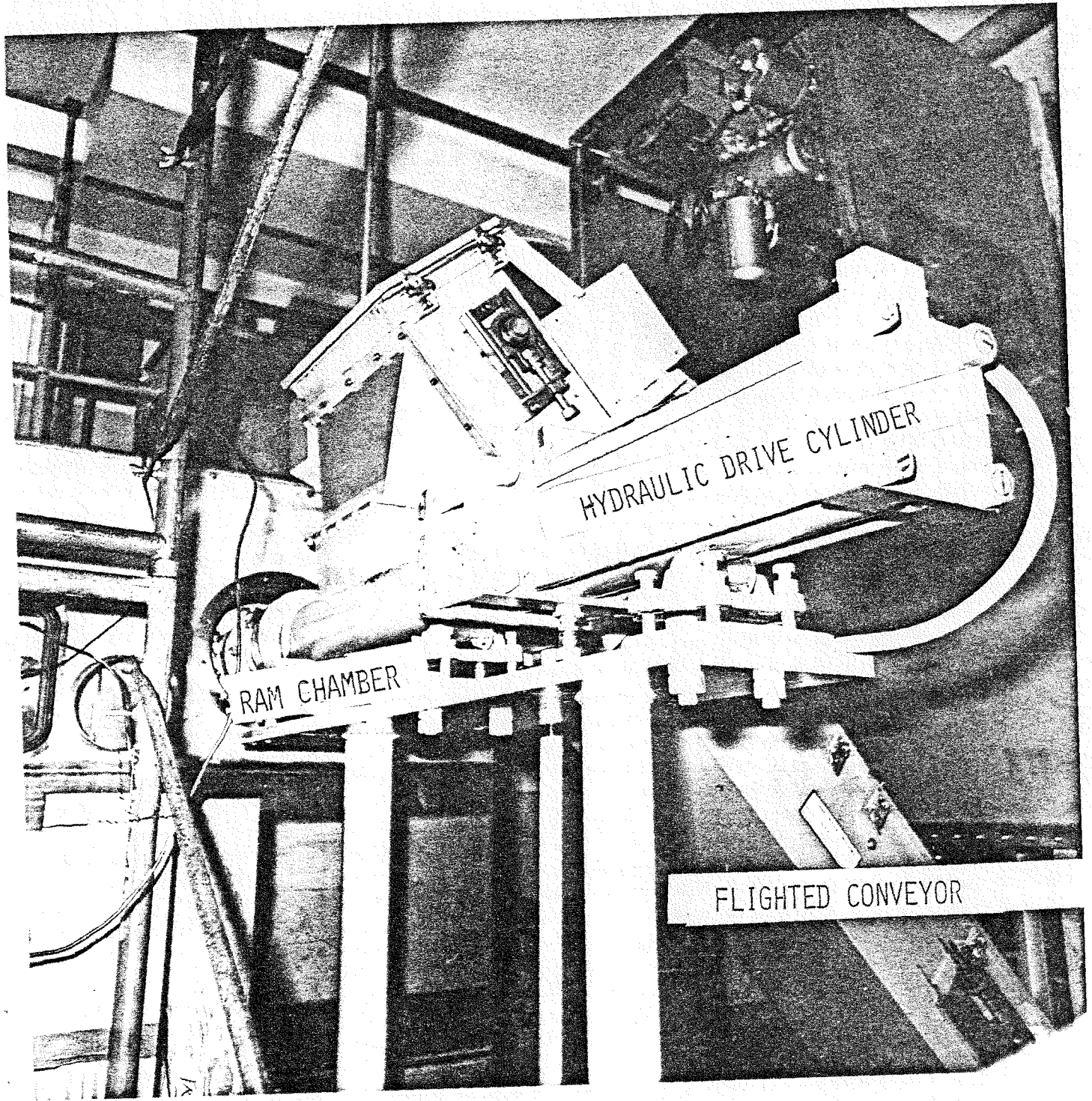
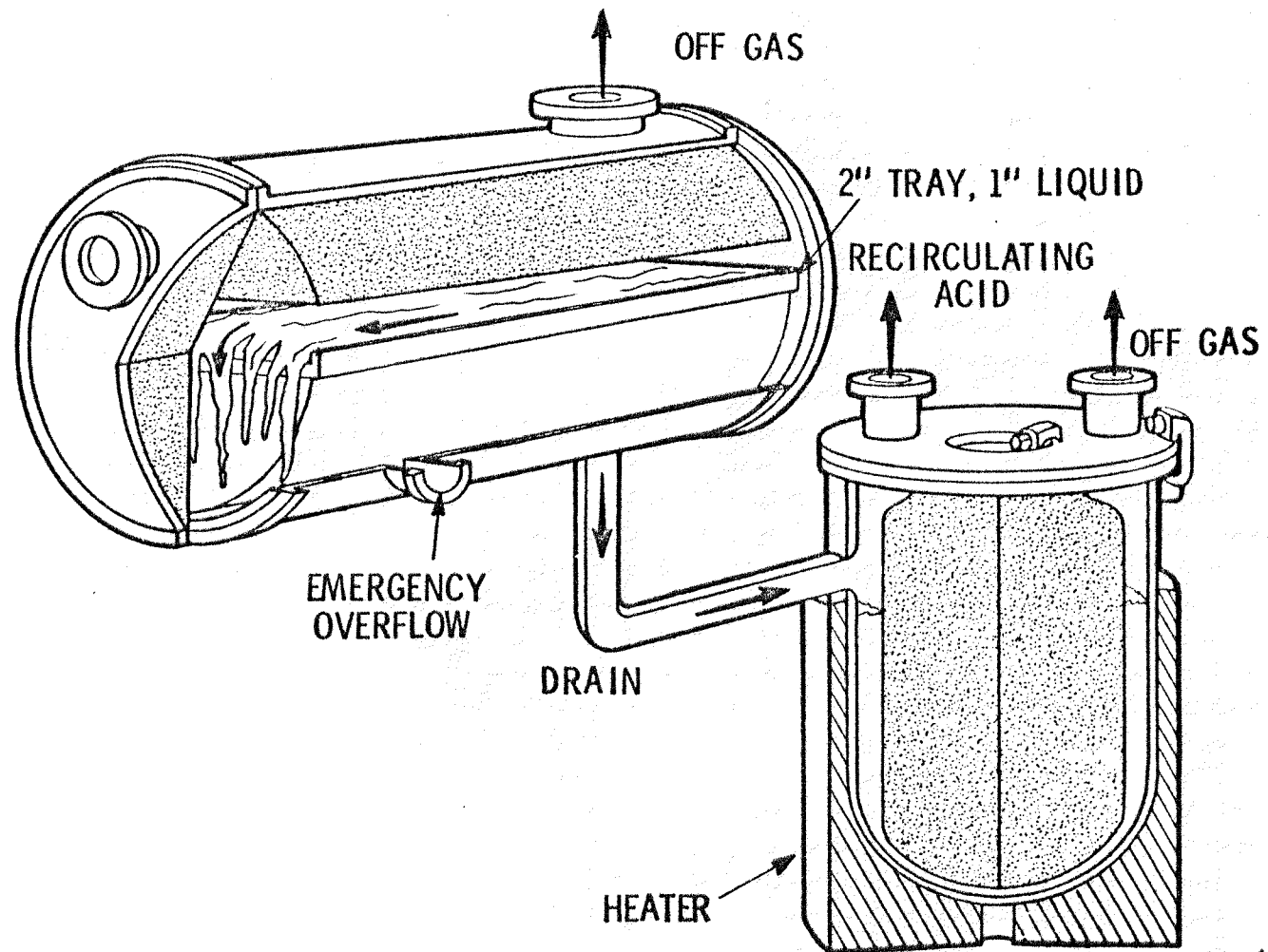


FIGURE 6
WASTE EXTRUDER FOR FEEDING DIGESTER

RADTU DIGESTER CONFIGURATION



HEDL 7609-80.1

FIGURE 7

RADTU DIGESTER WITH END REMOVED

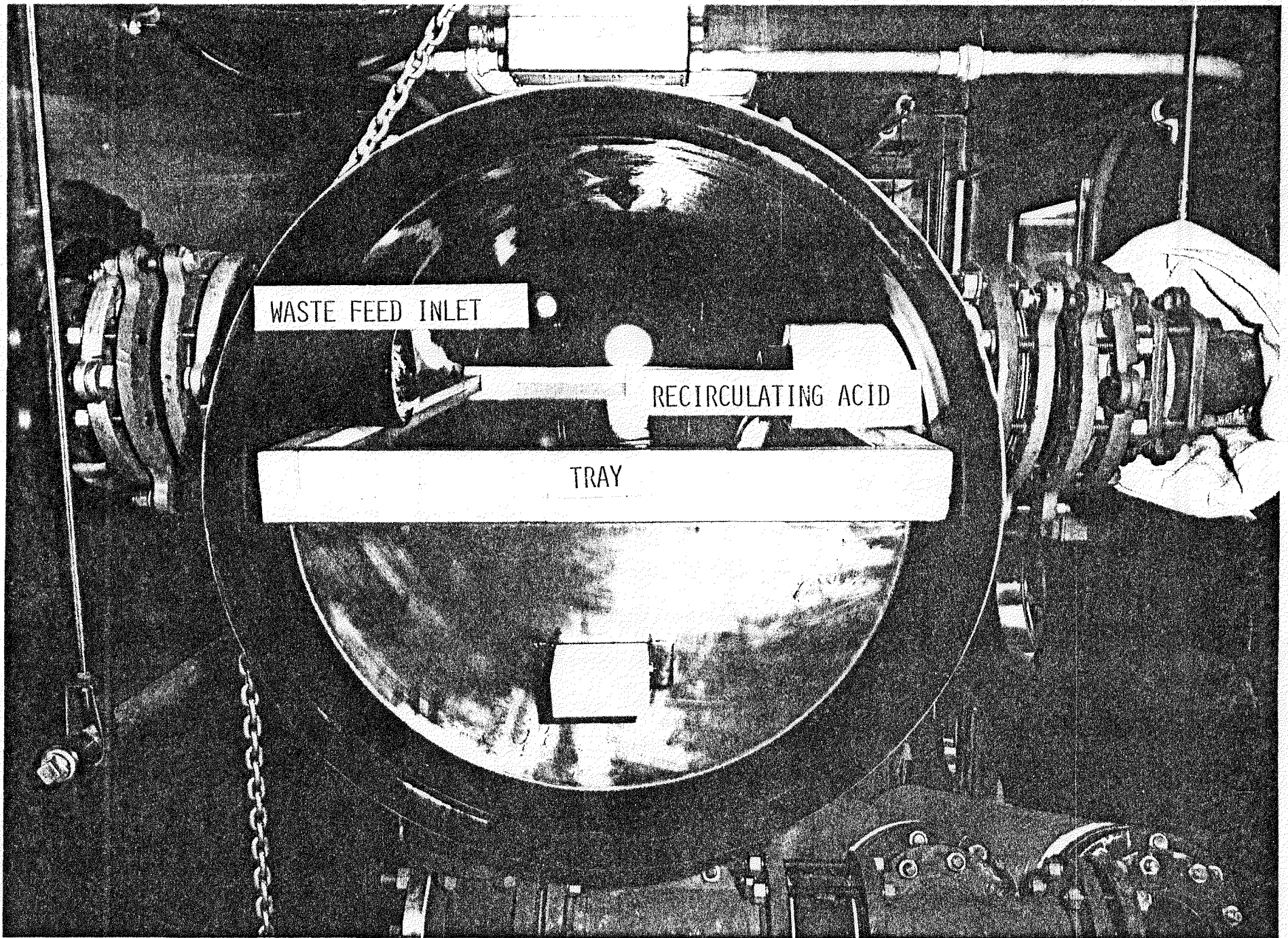
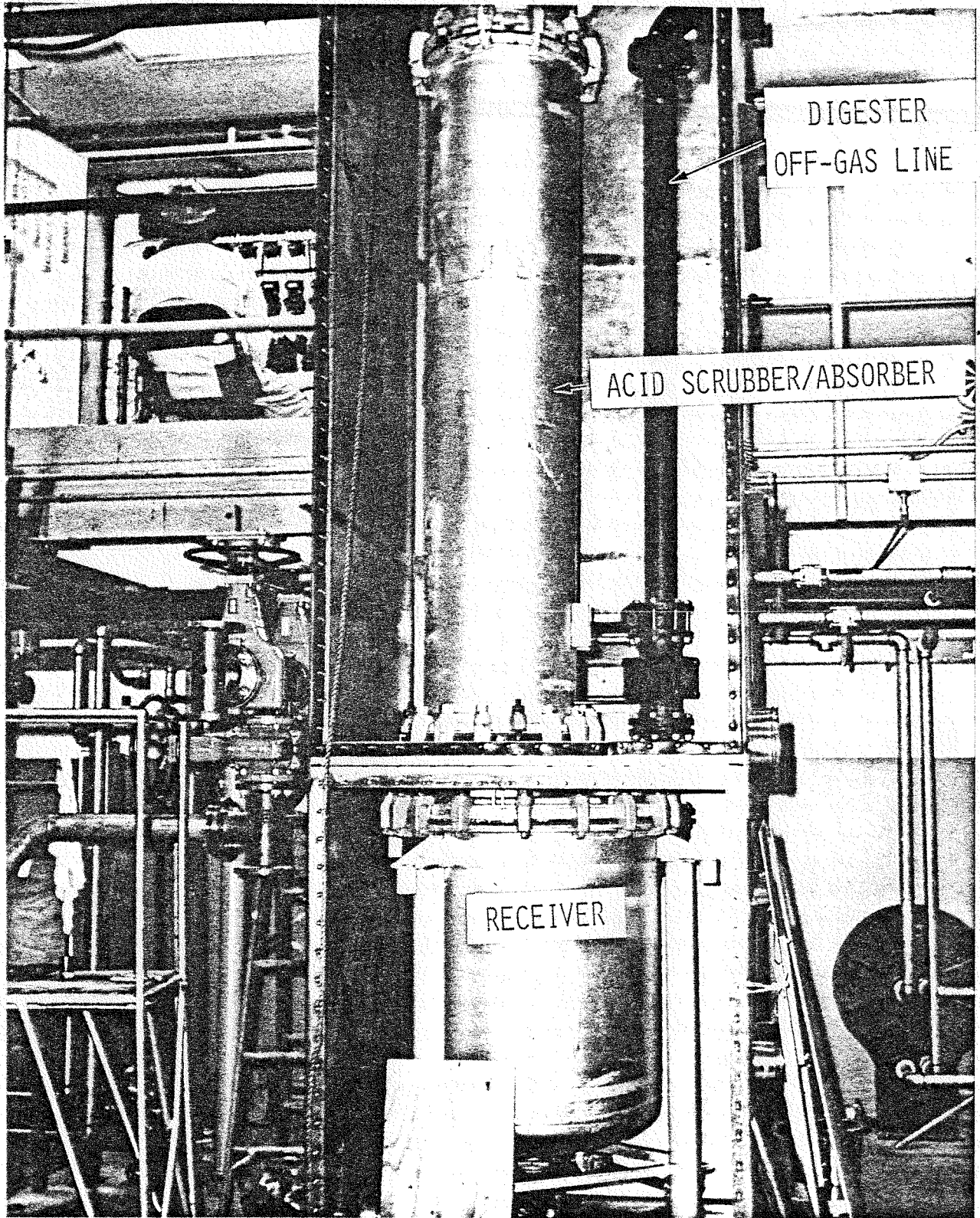


FIGURE 8



FIRST OFF-GAS COLUMN AND HOOD DURING FIELD ASSEMBLY

FIGURE 9

**CONDENSING ACID ABSORBER AND
HEATED HEPA FILTER**

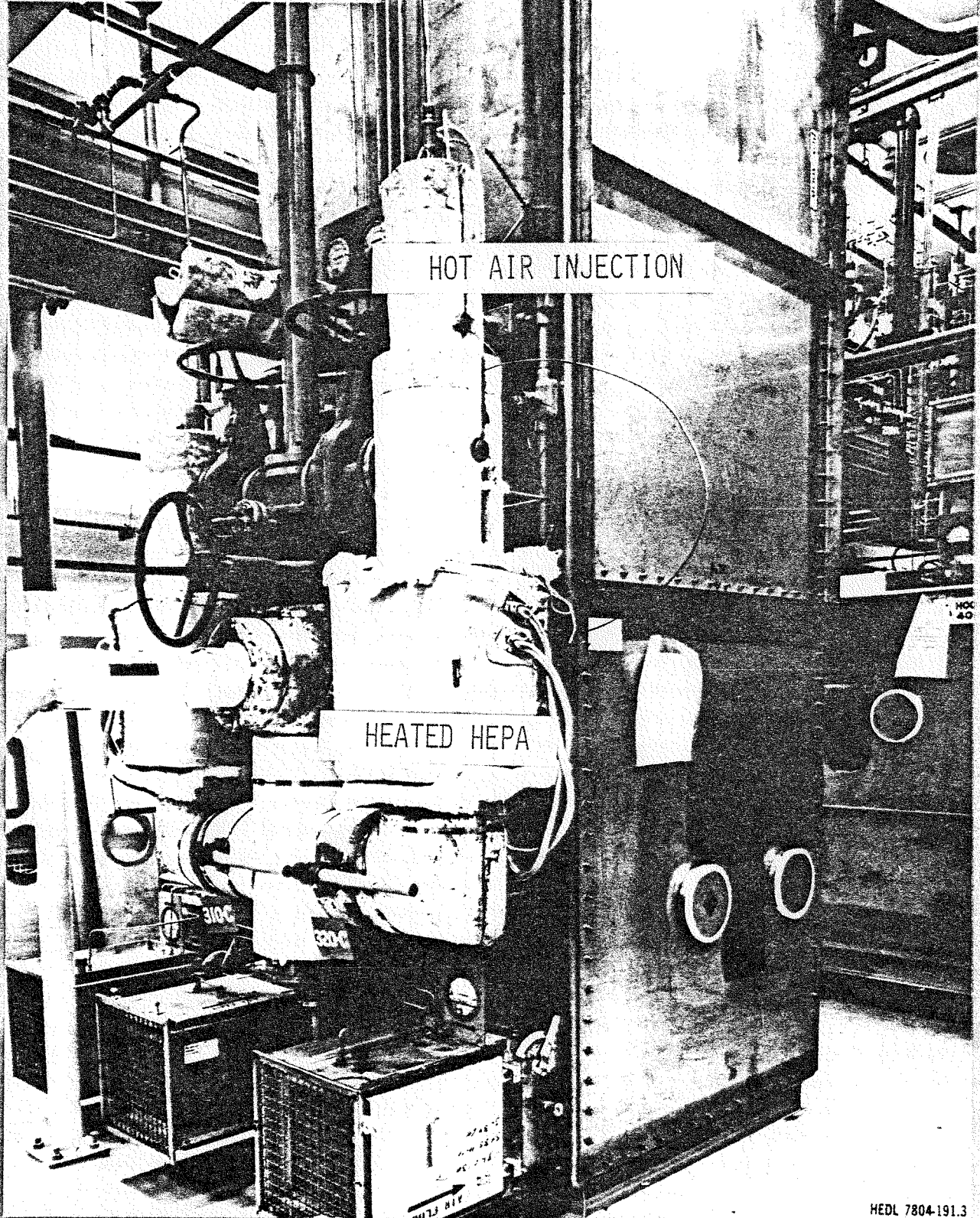
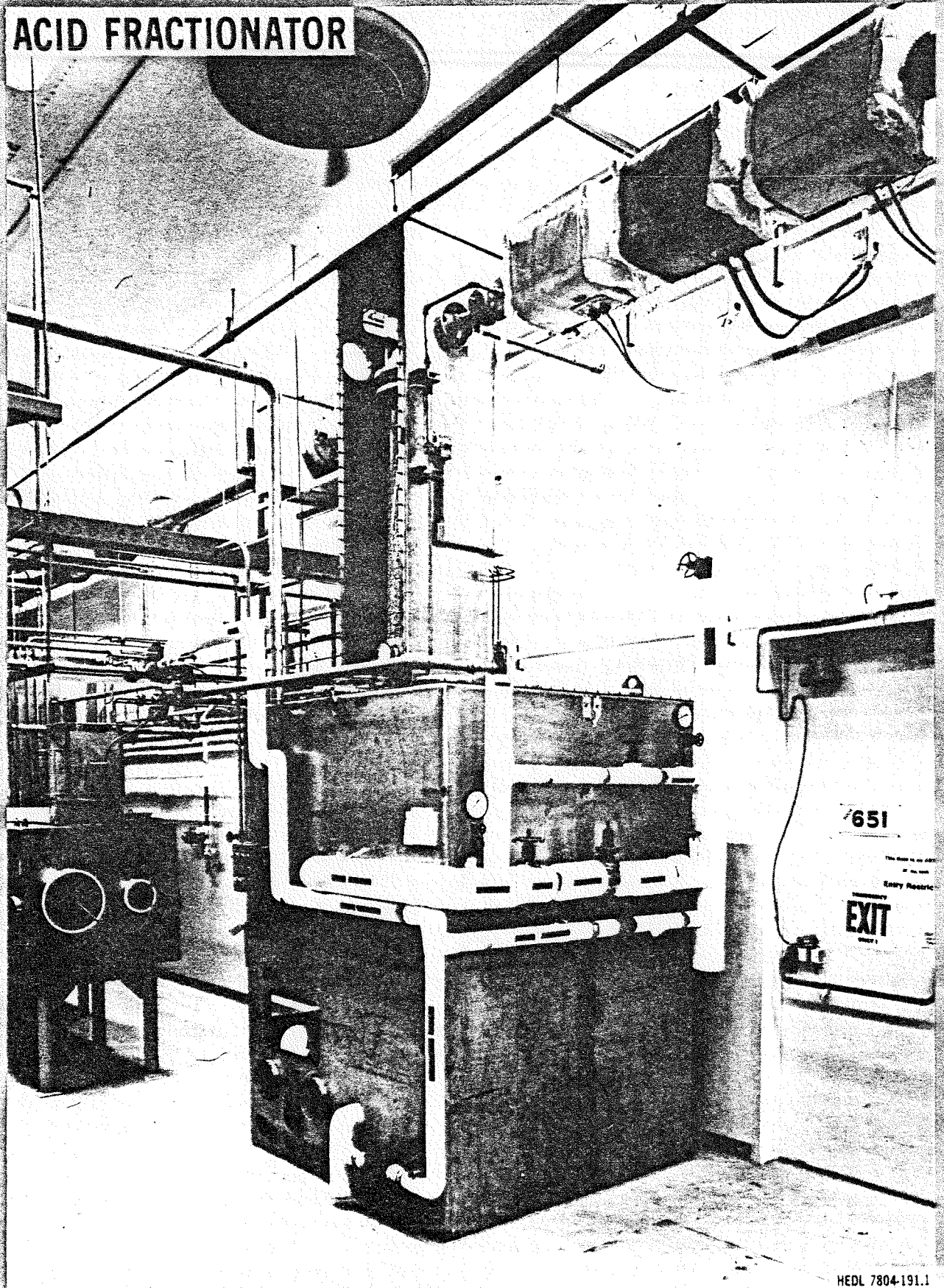


FIGURE 10

ACID FRACTIONATOR



HEDL 7804-191.1

FIGURE 11

FIGURE 12

<u>DATE</u>	<u>MODE</u>	<u>TIME/HOUR</u>	<u>WASTE ADDED</u> KG
NOVEMBER-DECEMBER, 1977	OCCASIONAL	23.4	62
JANUARY, 1978	INTERMITTENT (12 HRS ON/12 HRS OFF)	42.5	176
APRIL, 1978	CONTINUOUS	34.6	98
MAY, 1978	CONTINUOUS	28.5	90
MAY, 1978	CONTINUOUS	<u>35.5</u>	<u>111</u>
	TOTAL	164.0	537