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MASTER

HW-65587

ALI-54

## HALOGEN COLLECTOR TEST PROGRAM

PREPARED BY ARTHUR D. LITTLE, INC.

MARCH 1, 1960

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**HANFORD LABORATORIES**

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HANFORD ATOMIC PRODUCTS OPERATION  
RICHLAND, WASHINGTON

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HALOGEN COLLECTOR TEST PROGRAM

By

Arthur D. Little, Inc.  
Cambridge, Massachusetts

March 1, 1960

HANFORD ATOMIC PRODUCTS OPERATION  
RICHLAND, WASHINGTON

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FOREWORD

A statement of information received by General Electric Company following preparation of ALI-54 is necessary to interpret this document correctly. The vendor supplying one-inch activated charcoal and molecular sieve filters has advised that those delivered to Arthur D. Little Company were actually 1/2-inch filters. To clarify test results further, the program specifications have been included as Appendix D of this report. These specifications were published originally at Hanford as HWS-6750. This appendix describes the Halogen Collector Test Program scope and facilities and contains significant test data.

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## I. SUMMARY

### A. PURPOSE

The Halogen Collector Test program was undertaken to provide pilot-plant data on laboratory-proven methods of removing trace quantities of radioactive iodine from an air stream.

### B. SCOPE

The equipment was designed to operate with halogen collector units of standard air filter size, fabricated to specifications prepared from laboratory data. The equipment was operated so that the iodine removal efficiencies, loading capacities, and flow engineering data could be determined for each halogen collector tested. Tests were conducted according to Specifications for Halogen Collector Test Program, Project CGI-791 (HWS 6750), dated March 18, 1959, on the following halogen collectors: silver-plated copper-ribbon bed, 2" deep; activated-charcoal bed, 1/2" deep; activated-charcoal bed, 1" deep; molecular-sieve bed, 1" deep; and sodium thiosulfate bed, 1/2" deep. The tests were conducted at 70° and 160° F and at 70 and 95% relative humidity. The iodine injection rate specified was changed from 0.1, 1.0, and 10.0 ppm during the testing to 0.01, 0.1, and 1.0 ppm. The analytical method was a tracer technique using I-131 added to I-127.

### C. CONCLUSIONS

Efficiency tests were performed on each halogen collector. Only the 1/2" and 1" activated-charcoal collectors achieved a high iodine removal efficiency over a sustained period at the various operating conditions. Because of the high capacity of the charcoal, loading tests on these filters were discontinued without having achieved a breakthrough. The molecular-sieve collectors attained a high efficiency for a brief period. They began to lose their effectiveness when their great affinity for water produced a breakthrough. The sodium thiosulfate collectors did not attain a high efficiency and failed after a short testing period. The silver-plated copper-ribbon bed attained high efficiencies until cumulative iodine loading reached 12 grams for four filters. At this point, the iodine removal efficiency began to decline steadily. The second set of silver-plated copper beds never attained satisfactory removal efficiencies. This was believed due to the migration of a tar, used in manufacture, onto the mesh, during the degreasing operation.

### D. RECOMMENDATION

The 1/2" and 1" activated-charcoal-bed filters are best suited for use at the proposed conditions.

## II. DESIGN OF PROGRAM AND TEST FACILITY

### A. PRELIMINARY DATA ON SELECTED HALOGEN COLLECTORS

No laboratory tests were performed at the site on the selected halogen collectors. Previous testing on the silver-plated copper-ribbon bed indicated iodine removal efficiencies of 99% at 20°C with a gas flow of 60' to 240' per minute. Stable iodine was used at concentrations of 10 - 30 ppm. Laboratory tests on activated charcoal were conducted on 12" beds of 8 - 14 mesh charcoal. The efficiencies were indicated to be 99%, with over 90% of this adsorbed by the first 3/8" of bed depth. No migration through the bed was noted. Velocities of approximately 170' per minute were used for the gas stream. Linde molecular-sieve material was subjected to the same test conditions as the activated charcoal. Although the iodine removal efficiency was indicated to be 99.9%, only 55% of the iodine was retained in the first 3/8" of bed depth. A slow, steady downstream migration of iodine through the bed was also noted.

### B. REQUIREMENTS AND CHARACTERISTICS OF OVER-ALL TEST SYSTEM

The test system required the filtration, heating, and humidification of 4000 cfm of air in commercial equipment, and involved the injection and mixing of iodine vapor into the air stream, prior to passage of the air through a test chamber designed to contain commercial filter units. The exhaust gases from the test chamber were scrubbed with a prepared solution in a wet scrubber packed with saddles to remove any iodine remaining. Exhaust gases were discharged to the atmosphere.

Major considerations in the design were the method of iodine injection and the analysis for iodine. The iodine must be in the gaseous state at  $\pm 10\%$  of specified concentrations. The formation of iodine compounds or complexes was not permissible. The analysis for filter efficiency was carried out by use of a tracer technique, a sample of the gas being continuously withdrawn at the inlet, the exit, and between the filter banks of the test chamber. The I-131 content of these gas samples required that an accurate analysis for iodine content be carried out in a closed system, for safety reasons.

It was determined that a wet scrubber packed with saddles would be most effective in providing a clean exhaust gas.

### C. PREPARATION OF RADIOACTIVE IODINE

The requirements of the program, insofar as they affected the use of I-131, were (1) that the iodine be injected as molecular iodine, and (2) that no additional extraneous solid matter be added as carrier material. These requirements were met by conversion of the I-131 activity as received from Oak Ridge to an alcoholic tincture containing iodine in the free state, by use of a process that introduced no additional solid materials.

The method consisted of the addition of 100 mg of reagent-grade hydriodic acid,<sup>1</sup> followed by a stoichiometric quantity of hydrogen peroxide. This mixture was permitted to stand for 10 minutes, during which time the following oxidation went to completion:



At the completion of the reaction, the iodine was present in concentrated form, generally as crystals clearly visible in the small volume of solution. This mixture was then treated with 100% ethyl alcohol and diluted to a total volume of 50 ml. The solution was stored in polyethylene bottles, from which it was later transferred as needed.

### D. DESCRIPTION OF RADIOACTIVITY MEASUREMENTS

The measurement of I-131 was carried out by the use of wide-range ratemeters, coupled to standard well-type NaI scintillation counters.<sup>2</sup> The air stream was sampled at four points--(1) before the prefilter, (2) between the prefilter and the absolute filter, and (3) and (4) before and after the test filter. At each point, a volume of air was withdrawn from the stream, and the iodine was scrubbed from it by a countercurrent column containing a solution of 1 N NaOH and 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. This solution was circulated continuously through a standard gas-washing bottle inserted in the well of the counter. The volume of air was accurately measured by bellows-type meters.

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1. A special grade containing no hypophosphorous acid as preservative was used.
2. The components used in this program were Chicago Nuclear Rate-meter 1620B, Scintillation Well Counter DS5-5, and Basic Probe DS5.

Two ratemeters were used for the program. One was stationed at the first of the above-mentioned points, and was kept there continuously for monitoring the concentration of iodine in the input airstream; the second was placed on a table that could be moved to each of the other three stations during each run. All measurements were based on the concentration of I-131 in the input airstream, and were thus on a relative basis, eliminating the need to standardize each new solution of iodine.

Coupling an Esterline-Angus Recorder to each ratemeter enabled the operator to obtain a permanent record of the readings of each instrument during a run.

#### E. FACTORS AFFECTING PRECISION OF MEASUREMENTS

The plateau characteristics of each counter were determined by standard procedures, using I-131 activity in the same gas-washing bottles as those employed during the experiments.<sup>1</sup> The length of each plateau was about 150 - 200 volts, and the sensitivities of the two detectors at the center of the plateaus were different by about 3%. Choosing different relative points on the plateaus made it possible to operate the ratemeters at voltages such that identical responses would result from a standard I-131 solution placed in each well.

The ratemeters were kept on-line throughout the experiments.<sup>2</sup> This procedure helped to minimize shifts in the response of the instruments. It should be noted again that because all measurements were made on a relative basis, there was no need to calibrate the instruments beyond the point of checking the sensitivity of each ratemeter for a standard I-131 solution.

The percentage statistical fluctuation of the reading of a ratemeter can be computed from the relationship:<sup>3</sup>

$$\frac{100}{\sqrt{2 C R N}},$$

where C R = time constant in seconds, and N = counting rate in counts per second.

1. It was found that a Cs-137 source, supplied in a small plastic container, gave an apparent "double plateau" that rendered it unsuitable for establishing a suitable plateau.
2. Except for several intervals, when electrical breakdown necessitated repairs.
3. R. A. Faires and B. H. Parks, Radioisotope Laboratory Techniques, George Newnes, Ltd, London, 1958, p. 143.

As a rule, the final readings were made with the time-constant setting of 40 seconds. From this we can see that a counting rate of 10,000 per minute would result in a statistical fluctuation of about 1%.

The accuracy of the meter on the ratemeter is  $\pm 1\%$  of full scale, and we believe that in most instances this is the limiting factor in obtaining a more precise reading. The statistical fluctuation is essentially eliminated by means of taking the reading from a strip chart, where an average reading can be quickly determined.

For low count rates of the order of 1500 cpm, where the 3000-cpm scale is used, a variation of  $\pm 30$  cpm is possible. Where count-rate increases are small during a test it is apparent that this meter inaccuracy can account for a considerable portion of the variations observed in the data. Since a determination is dependent upon the relative count rates before and after a filter or collector, the variations of a particular efficiency is a function of the variation of count rates before and after the filter. Because of this  $\pm 1\%$  accuracy of the meter, much effort was spent on keeping the background count to a minimum--which of course would minimize the effect of this meter accuracy.

#### F. EFFECT OF BACKGROUND

During the experiments, the accumulation of activity on the filters resulted in a gradual rise of the background of the well counters. This background rose from about 350 cpm to 1800 or 2000 cpm, and efforts to reduce it were rather ineffective. Additional shielding amounting to 2" thicknesses of lead around the sides and on the top of the main shield reduced the background from 2000 to about 1200 cpm. Additional lead sheets were placed around the base of the detector support, but even this did not produce any marked lowering of the background. It is suggested that some thought be given to preventing this effect in any future studies.<sup>1</sup>

#### G. SELECTION AND DESIGN OF IODINE DETECTION SYSTEM

Approximately 0.1% of the gas stream was withdrawn at each of the inlet, exit, and intermediate gas-sampling connections. Each sample passed through a column packed with stainless steel pall rings.

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1. If the detector were temporarily moved about 60' from the filters, the background could be reduced to normal values.

The free iodine was fixed in solution as sodium dithionite and sodium iodide. A sodium thiosulfate--sodium hydroxide solution was used in the scrubbing columns. When a continuous sample was taken throughout the test at each sampling station, a comparison of the I-131 content of any two adjacent samples indicated the efficiency of the filter between them. The comparison was made by the use of scintillation counters. A glass probe or miniature gas washing bottle was inserted in the re-circulation system in the scrubbing columns. This probe was set into the well of the scintillation counter, and the count rate of each sample was recorded. A water-filled probe was used to find the background reading at each station. Subtraction of the background from the recorded count rate produced a corrected count rate of the sample. A flow sheet of the analytical system is shown in Figure 15A (in Appendix A). Sample calculations are shown in Appendix B.

The efficiency of this analytical system was tested by the arrangement of two scrubbers in series. The exhaust gas from the first scrubber became the feed for the second. This system proved that the analytical system was at least 99.9% efficient in removing iodine from the air sampled.

#### H. SELECTION OF METHOD OF IODINE INJECTION AND DESIGN AND CONSTRUCTION OF APPARATUS

The iodine injection rates, based on the specifications of the test program, varied from 0.1 ppm to 10.0 ppm. Three methods of injection were originally proposed: the injection of iodine vapor through a capillary tube; the injection of iodine as HI; and the injection of iodine dissolved in alcohol as a tincture solution.

Injection of iodine through a capillary tube was rejected because of the difficulty of designing a system flexible enough to operate at all desired concentrations. At flow rates of 0.1 ppm, approximately 150' of capillary tubing would have been required to provide a pressure drop high enough for effective metering and control.

Injection of iodine as HI was rejected because of problems very similar to those associated with injection as iodine vapor. It had the additional disadvantage of requiring the formation and decomposition of a compound, HI.

The injection of iodine as a tincture solution in alcohol was the method selected for use. A flow rate of 1200 ml per hour was required with this method and it was possible to maintain adequate control at this

injection rate with commercial pumping equipment. Since the iodine was to be injected as vapor, combustion tests were performed on sample solutions. They proved that all iodine present was vaporized as free iodine. No other iodine compounds or complexes occurred. This proof is given in Appendix C.

Tests of various ignition systems indicated that the iodine-alcohol mixture burned well when ignited by a large, hot wire gauze, which was required to maintain the flame. A 200-mesh platinum-wire gauze was used for this purpose. Tests with a brightly polished stainless steel plate could not detect carbon formation. The system selected involved the atomization of the iodine-alcohol solution by a spray nozzle prior to burning. A propane torch introduced briefly between the nozzle and the shielding hood was used for ignition of the spray. The screen was located about 4" downstream of the nozzle.

Although the iodine injection concentrations were changed to 0.01 ppm to 1.0 ppm during the tests, the total pumping rate remained the same and the injection system did not require modification, since solution concentrations rather than pumping rates were changed.

## I. SIGNIFICANCE AND DETERMINATION OF SCRUBBER FACTORS

A scrubber factor can best be defined as a factor, included in the calculation of efficiencies, which takes into account the minor variations in construction, installation, and operation of the four analytical scrubbers. If all four had been exactly identical, the factor for each would have been one. When these factors were introduced into the calculations, the relative count rates were adjusted to equivalent rates for scrubbers with a factor of one. Such things as the angle of approach and the length of tubing for transporting the scrubbing solution from the scrubber to the counter seemed to have had an effect on this factor.

So that the factors might be determined, the system was run under actual test conditions with no filters in the test section. Each scrubber sample was therefore of the same concentration, assuming adsorption or desorption of iodine on the walls of the system to be negligible. The scrubber factors were calculated from the amount by which the relative count rates diverged from the average.

The initial factors were determined at the start of the program and are given below.

<u>Scrubber</u>	<u>Factor</u>
1	0.963
2	1.190
3	0.988
4	0.927

After more experience was gained in operating the scrubbers, it was decided to re-evaluate these factors. These re-evaluated factors were determined after completion of the efficiency tests and were used in recalculation of the scrubber efficiencies. All efficiencies shown were calculated with the following new scrubber factors:

<u>Scrubber</u>	<u>Factor</u>
1	0.986
2	1.023
3	1.106
4	0.972

### III. TEST EQUIPMENT

The over-all equipment layout is illustrated in Figures 1A and 2A. A flow sheet of the system is shown in Figure 13A.

Air enters the system from outside the building through a galvanized steel duct, 20" x 40". Inside the building, the duct is enlarged to 20" x 44". The outside opening is covered by a steel grate, or bird catcher. Dampers are provided to allow the air to be drawn from inside the building.

The entering air has its dust content reduced to below 0.005 mg per cubic meter by two sets of filters. There are four roughing filters, 20" x 20" x 2", made of expanded aluminum mesh covered with super filter coat, a material to which dust particles will adhere. They have an average dust arrestance of 70% at flow rates up to 1200 cfm. They are housed in a duct section 20" x 44" x 18.5" long. There are four Micretain filters, contained in a duct section 42" x 50" x 36" long and followed by a tapered section to reduce the dimensions to the original 20" x 44". The Micretain filters are 24" x 24" x 11-1/2". The material used is a glass-asbestos filter medium. They have an efficiency of 95-99% at a flow rate of 1000 cfm. Both sets of filters are housed in galvanized steel ducts.

The air is humidified and preheated in a segmented, galvanized steel duct, 20" x 44" x 81" long. It contains parallel steam coils for heating the incoming air, a steam injection grid to control the relative humidity, and a steam injection pipe to provide additional steam at more severe conditions. Automatic control valves are provided for one steam coil and for the steam injection grid. In cold weather, a bypass damper and the primary steam coil are used to temper the incoming air to prevent freezing in the main steam coil, the primary coil being maintained under full steam pressure. The steam is drawn from a 220-psi steam line and reduced to 96 psi. A water injection system provides the means of desuperheating in the steam coil and injection pipes. Desuperheating is necessary in warm weather. Plexiglass windows in the downstream end of the humidifier provide visual observation of the gas stream. A demister is located downstream of the plexiglass windows.

The size and shape of the duct is changed to a 20"-10 circle. The material used from this point on is stainless steel, type 316. The iodine vapor is introduced about 6' downstream of the beginning of the stainless steel duct and after a 90° elbow.

for withdrawal of a continuous gas sample are provided before and after each bank of filters. The velocity gradient was measured across the face of the filter in this section at the start of the program and was found to be essentially uniform.

Continuous samples withdrawn from the filter-holding section pass through one of four analytical scrubbers. Each scrubber consists of a packed column, a centrifugal pump, a condenser, and a gas meter. The column is packed with stainless steel pall rings. The scrubbing solution in the column is 1.0 N Na OH-- 0.1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. About 700 ml of solution is contained in the scrubbing system. The incoming gas sample from the test section passes up through the packed column, where the iodine present is removed. The gases are cooled in the condenser and excess moisture is removed before they pass through the gas meter. After the meter records the volume, the gas is returned to the test system on the exhaust side. Figure 15A is a flow sheet of the analytical scrubber system.

Downstream of the filter holder, an exhaust scrubber removes all remaining iodine (Figure 6A). It is packed with 1-1/2" Intalox saddles on a base of 8 cubic feet of 2" Intalox saddles. It contains 100 gallons of the scrubbing solution used in the analytical scrubbers. The solution is recirculated continuously by a centrifugal pump at a rate of 40 to 60 gpm.

The gas stream from the top of the exhaust scrubber is drawn off through a duct by a suction fan and exhausted up the stack. This fan is the source of power for the entire system, and its location results in the entire testing apparatus operating at a slight negative pressure.

A holdup tank with a capacity of approximately 60 gallons is provided for use when the exhaust scrubber is drained or when an excess of scrubbing solution occurs in the exhaust scrubber. The scrubbing solution is mixed in a stainless steel 55-gallon drum and pumped through a centrifugal pump to the large scrubber. The drum is equipped with a portable propeller mixer. The system is controlled from an instrument panel containing the following instruments:

- (a) a temperature-humidity recorder controller.
- (b) a manometer to measure the volume of the gas stream.
- (c) a manometer to measure  $\Delta P$  at the entrance of the test section (difference between chamber and atmospheric pressure).

The injection system controls the iodine flow rate and therefore the concentration of iodine in the air stream. The major components are two stainless steel tanks, two diaphragm pulse pumps with a common motor, and tubing and valve connections allowing either pump to service either tank. One tank contains denatured ethyl alcohol; the other contains an iodine-alcohol mixture. The proper rate of iodine injection, at a constant flow rate necessary for smooth burner operation, is accomplished by the blending of the flows from the tanks. Each pump can be set to draw any amount between 0% and 100% of its capacity. The diaphragm pumps operate by pumping into a surge tank against a back pressure to remove pulsations in the line to the burner nozzle. Figure 14A is a flow sheet of the iodine injection system.

The injection system delivers an iodine-alcohol mixture of a predetermined concentration at a flow rate of about 1200 ml per hour to an atomizing nozzle. The iodine-alcohol stream is atomized by contact with a high-velocity air stream. The atomized stream passes into a stainless steel cylinder containing a platinum screen. Combustion of ethyl alcohol and vaporization of iodine take place from the screen. The burner is lit manually and the combustion then becomes self-sustaining. A secondary air stream carries combustion products into the main duct. The system is insulated from the demister through the test chamber, so that heat losses will be minimized and condensation prevented.

A baffle, about 4' downstream of the introduction of the iodine vapor, provides for mixing. Tests performed prior to initiation of the program indicated mixing was adequate. Approximately 20' of straight duct follows. About 4' from the downstream end of the duct, a pitot tube measures the velocity of the gas stream. Wet- and dry-bulb temperatures are taken just beyond this. The dry bulb controls the heating of the incoming air and the wet bulb controls the relative humidity by regulating the steam injection. During the loading tests, an orifice was installed to replace the pitot tube, and a moisture-proof fan was placed inside the duct to maintain a sufficient flow across the psychrometer tube.

The filter-holding section, illustrated in Figures 3A, 4A, and 5A, is made of type 304 stainless steel. It is 4' 9" square and 10' long. A transition section on each end is 8' 4-1/2" long. The test section provides space for three banks of filters, with centers about 4' apart. The outside banks are about 1' from the ends of the test section. A door is located on each side of the test section at each filter bank. The filters are held in place by spring action. This is illustrated in Figures 7A, 8A, 9A, 10A. Drain valves are installed in the floor of the filter holder. Connections

- (d) a manometer to measure  $\Delta P$  across the entire system.
- (e) a manometer to measure  $\Delta P$  across the exhaust scrubber bed.
- (f) a manometer to measure the amount of scrubbing solution in the exhaust scrubber.
- (g) a manometer to measure the flow rate through the centrifugal pump.
- (h) draft gauges to measure the  $\Delta P$  across each set of filters.

In the control system interlocks were present so that

- (a) The main fan to move air through the system would not operate unless the recirculating pump on the exhaust scrubber were operating.
- (b) Iodine-alcohol solution would be blocked from the burner if the main fan lost power.
- (c) Steam would be blocked from the steam injector system used for humidifying if the main fan failed.

#### IV. TEST PROGRAM

##### A. CONDITIONS OF TESTING AND HALOGEN COLLECTORS TESTED

The halogen collectors tested were: a silver-plated copper-ribbon bed 2" deep, an activated-charcoal bed 1/2" deep; an activated-charcoal bed 1" deep; a molecular-sieve bed 1" deep; and a sodium thiosulfate bed 1/2" deep.

Each filter was to be tested at eight sets of conditions. These are summarized in Table I. The temperatures were 70 and 160° F; the relative humidities were 70 and 95%, and the iodine concentrations were 0.1 ppm, 1.0 ppm, and 10.0 ppm. After efficiency tests on the silver-plated copper-ribbon bed were completed, the iodine concentrations were changed to 0.01 ppm, 0.1 ppm, and 1.0 ppm. The air flow rate was 4000 cfm.

A minimum of three tests were performed on each filter at each set of operating conditions so that the efficiency of those conditions could be determined. The tests on any one filter were discontinued if the efficiency on two consecutive tests fell below 50%.

When the halogen-collector efficiency tests were completed, tests were performed to determine the loading capacities and breakthrough points. These tests were performed on the silver-plated copper-ribbon bed at 70° F and 95% relative humidity, and on the 1/2" activated-charcoal filter at 70° F and 95% relative humidity and at 160° F and 95% relative humidity. The iodine injection rate was 10 ppm for the silver-plated copper bed and 40 ppm for the activated charcoal. The air flow rate was 1000 cfm.

##### B. GENERAL PROCEDURES FOR OPERATION OF MAIN TEST SYSTEM, INCLUDING STARTUP AND SHUTDOWN OPERATIONS

###### 1. Startup

1. The centrifugal pump in the exhaust scrubber system is drained when not in use. The drain must be closed and the intake line to the pump from the exhaust scrubber opened.

2. The temperature-humidity controller is turned on.

TABLE I

TEST OPERATING CONDITIONS

Iodine	I <sub>2</sub> Soln Conc (gI <sub>2</sub> / 1 alc)	I <sub>2</sub> Flow Rate (gnI <sub>2</sub> / hr)	Temp. (° F)	Rel Hum (%)	Pump and Tank Arrangements with Micrometer Settings	Order In Which Tests Are To Be Run
0.01	0.38994	0.0798	70	70	PITI (1.96) - P2T2 (4.34)	I
0.01	0.38994	0.0794	70	95	PITI (1.96) - P2T2 (4.34)	II
0.01	0.38994	0.0489	160	93-95	PITI (1.20) - P2T2 (4.67)	III
0.01	0.38994	0.4890	160	93-95	P2T1 (5.45) - None	IV
0.1	6.5526	0.798	70	70	P1T1 (1.185) - P2T2 (4.70)	V
0.1	6.5526	0.794	70	95	P1T1 (1.175) - P2T2 (4.70)	VI
1.0	6.5526	7.940	70	95	P2T1 (5.34) - None	VII
1.0	6.5526	4.890	160	93-95	P2T1 (3.28) - P1T2 (4.42)	VIII

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3. The blower starts to draw air into the system. In cold weather, the dampers in the heating section are adjusted to preheat the incoming air. The preheating steam coil is turned on in this case.

4. Steam is introduced into the humidifying grid and the system is slowly brought up to operating conditions by means of the temperature-humidity controller.

## 2. Operation

5. During operation, the system is maintained at operating conditions. The level in the exhaust scrubber and the volume of gas flow should be checked periodically and adjustments made when necessary.

## 3. Shutdown

6. The temperature-humidity controller is turned off.

7. The steam line, blower, and centrifugal pump are shut off.

8. The pump is drained and flushed with water.

## C. EXHAUST SCRUBBER OPERATIONS

The solution used in the exhaust scrubber is made up in a 25-gallon stainless steel drum equipped with a mixer. The proportions of the solution are: NaOH--33.3 pounds; Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> -- 13.2 pounds; water--25 gallons. This solution is pumped into the scrubber and water is added to bring the volume up to 100 gallons. Since a certain amount of liquid is held by the packing, the actual amount in the scrubber is somewhat greater than 100 gallons. During testing, water must be added at intervals to maintain the proper level in the scrubber. Once a week, a 50-ml sample of the scrubber solution was taken. This was titrated with 0.1N iodine to an end point. The iodine capacity of the scrubbing solution was determined from this titration. When the solution was close to losing its ability to hold iodine, it was concentrated and transferred to the holdup tank for later disposal. A new batch was prepared and the cycle repeated. A bubbler containing a caustic-thiosulfate solution was employed in the duct just after the scrubber, to keep a constant check on the effluent from the system.

D. PROCEDURE FOR EFFICIENCY TESTS

1. Prepare the analytical scrubbers for testing.
2. Take the initial and background count rates for each scrubber. Record the initial levels of the scrubbing solution in the glass sumps. Record the initial gas-meter readings.
3. During the recording of the initial count rates, light the injector burner.
4. When the count rates have been recorded and the burner has been operating for 15 minutes or longer, the test may be started. Begin the test by opening the gas cocks on the analytical scrubbers.
5. During the test:
  - a. Maintain liquid levels in the analytical scrubbers.
  - b. Take the initial and final temperatures and pressures of the gas flowing through the analytical scrubbers.
  - c. Maintain a pressure of approximately 10 psig in the injection system.
  - d. Check the control panel to ensure that all pieces of equipment are functioning normally.
  - e. Maintain the level in the exhaust scrubber at 100 gallons.
6. At the conclusion of the test:
  - a. Shut off the gas cocks on the analytical scrubber systems.
  - b. Shut off the burner and the injection-system pumps.
7. Take the final background count rates for each analytical scrubber. Record the final level of the scrubbing solution in the glass sumps. Record the final gas-meter readings.
8. Begin preparations for the next test.

### E. PROCEDURE FOR LOADING TESTS

The loading tests are conducted with one halogen collector mounted in the center section. The space for the other three filters is blocked off. Iodine is injected continuously at a high rate, generally 40 ppm. Since only the center section is in use, two sets of analytical scrubbers can be used. One set is on stream testing, while the other is being prepared. This allows a continuous check to be run on the efficiency. At high rates of iodine injection, the two sets of scrubbers should be switched every hour. The operation of the test and the preparation of the scrubbers are generally the same as described under Section D and H. Loading of the filter is continued until a breakthrough has been confirmed or a sufficiently large amount of material has been adsorbed.

A 48-hour continuous run of air at 70°F and 70% relative humidity follows the loading of the filter. The count rate of the gas stream behind the halogen collector is recorded so that the amount of desorption of iodine may be determined. When a breakthrough is achieved on the original testing, the desorption test is followed by additional iodine injection at the original rate. Testing is discontinued when the efficiency either fails to improve or shows another breakthrough.

### F. PREPARATION OF IODINE-ALCOHOL SOLUTION

On the basis of the iodine concentration required and the solubility of iodine in alcohol, two basic iodine-alcohol solutions were used during the tests: 0.38994 gram of iodine per liter of alcohol, and 6.5526 grams of iodine per liter of alcohol. By the proper blending of these with 95% ethyl alcohol, all the required iodine concentrations were achieved. Table I indicates which of the two basic solutions was used.

The first step in preparing the iodine-alcohol solution is to estimate the number of hours that the burner will be required to operate. This depends upon the number of tests required and the duration of the tests. A safety margin consisting of a one-hour startup, a one-hour shutdown, and a one-hour interval between tests was added.

The flow from the iodine-alcohol tank is composed of  $x$  grams of iodine per hour and  $y$  ml of alcohol per hour. The specific amounts of each of these under each set of operating conditions are summarized in Table II. When the required number of burner operating hours is determined, the proper amount of solution can be prepared.

TABLE II

COMPONENTS OF IODINE-ALCOHOL SOLUTIONS

<u>Order in Which Tests Are to be Run</u>	<u>g I<sub>2</sub> / hr</u>	<u>ml alc / hr</u>
1	0.0798	204
2	0.0794	204
3	0.0489	124.5
4	0.489	1245
5	0.798	123.2
6	0.794	121.3
7	7.94	1213
8	4.89	740

The prepared solution is transferred to the mixing tank and placed in the hood for the addition of I-131. Enough I-131 is added to provide 1.5 millicuries per burner operating hour.

The solution is given a final mixing in the tank and then transferred to the injection system through a closed system by gravity flow. The lines of the injection system are cleared of air bubbles and the system is then ready for operation.

#### G. OPERATION OF INJECTION SYSTEM

The pump-tank combinations and pump micrometer settings were determined in advance for each set of operating conditions. These are summarized in Table I. The pump micrometers are set, the proper pump-tank combinations are established, and primary and secondary air flows are turned on. The pumps are turned on to build up a pressure of about 10 psig in the system. This pressure is regulated by a needle valve. The benzo-matic torch is then lit and the access hole to the nozzle opened. The pumps are turned on and the solenoid valve is opened simultaneously. The atomized spray from the nozzle is ignited and the access hole closed. The burner should operate without further attention for the duration of the test. However, an occasional adjustment of the needle valve may be necessary, to maintain the pressure in the system at about 10 psig.

#### H. PROCEDURE FOR ANALYTICAL SCRUBBERS

##### 1. Basic Procedure

1. Fill the scrubbers with a 500-ml scrubbing solution made up as 1.0 N NaOH and 0.1N sodium thiosulfate.
2. Start the recirculating pump and run it for 5 minutes to obtain a solution with a uniform activity.
3. Read and record the count rate and background for each scrubber. Be sure that the count rate has become constant before taking a reading.
4. Record the gas-meter readings.
5. As count rates are obtained for each scrubber, shut off the recirculating pump. After the scrubber column has drained for 8-10 minutes, record the volume of the solution as shown by the graduated flask.

6. Turn on the recirculating pumps and begin the test by opening the gas cocks on the gas meters.

7. After 10 minutes, record the temperature and pressure of the gas through each meter.

8. During the test, periodically check the liquid levels in the scrubbers. Maintain a near-initial level by adding water while the system is in operation.

9. Before the test ends, record the temperature and pressure of the gas stream through each meter.

10. Turn off the gas cocks on each scrubber and record the gas-meter readings. This concludes the test.

11. Measure and record the final count rate and the final background for each scrubber. As each scrubber reading is completed, shut off the recirculating pump and allow the column to drain for 8-10 minutes. Read and record the volume in the graduated glass flask.

12. Drain the scrubbers completely.

13. Add 500 ml of water and recirculate it for 5-10 minutes. Drain and repeat twice.

14. Add 500 ml of fresh scrubbing solution, recirculate it for 5-10 minutes, and drain.

15. Add 500 ml of fresh scrubbing solution. The system is now ready for the next test.

## 2. Abbreviated Method

When concluding one test, do not drain and rinse the analytical scrubbers (Steps 12-15). Record the initial count rates, the background count rates, and the gas-meter reading immediately after finishing the final count rates, etc., from the preceding test. Open the gas cocks and begin the next test.

This cannot be done when the system is operating at 160°F, because the liquid level in the scrubbers builds up and the scrubbers must be drained after each test.

V. TEST RESULTS

The results of the efficiency tests are given in Table III. They are listed in the order in which they were run. The first filter tested was the silver-plated copper-ribbon bed. At the halfway mark in the testing, a breakthrough was determined on these filters. A second set was installed, but the efficiencies were poor. It is believed that the adhesive used in manufacturing migrated onto the mesh during degreasing, and that the effective surface was thus reduced. The degreasing was carried out with trichloroethylene being used on both sets of filters; no migration of adhesive was observed on the first set. The tests were discontinued without completion of the series. The 1/2" and then the 1" activated-charcoal filters were tested without appreciable difficulty. As predicted, the molecular-sieve halogen collectors showed a greater affinity for water than for iodine, and the tests were discontinued. The sodium thiosulfate halogen collectors did not attain high efficiencies at any time. They were taken out of service after only a few hours of testing. Upon examination, they were discovered to have lost about 40%, by volume, of the original bed. Some of this lost thiosulfate was discovered to have recrystallized on the floor of the test section downstream of the halogen-collector bank. It is assumed that the lost material was partially carried out of the bed either dissolved or entrained in droplets of water. The remainder of the loss is probably accounted for by the sagging and settling of the filter bed as it gained weight by absorbing water.

Between the efficiency tests and the loading tests, the scrubber factors were redetermined, as discussed in Section I, "Significance and Determination of Scrubber Factors," in Chapter II. Table III contains the results re-evaluated with the revised scrubber factors.

Loading tests were performed on two filters after suitable modification of the metering and air-flow control equipment. A silver-plated copper-ribbon filter from the first series of efficiency tests was reused. An alcohol rinse did not remove any of the adsorbed alcohol, and testing was discontinued after three hours when the efficiency continued to drop. The 1/2" activated-charcoal filter was tested at 70° and 160°F and 95% relative humidity. No breakthrough was achieved although over 1500 grams of iodine were adsorbed in the first test and 1100 grams in the second. Because of the performance of the 1/2" filter, no loading tests were run on the 1" activated charcoal. No loading tests were conducted on either the molecular-sieve or the sodium thiosulfate halogen collectors, because they had previously proved to be unable to operate successfully at the required conditions.

TABLE III

## RESULTS OF TESTS OF HALOGEN COLLECTOR EFFICIENCY

Test*	Operating Conditions			Pressure Drop (Inches of $H_2O$ )						Iodine Concentration				Iodine Removal Eff (%)				Cumulative Iodine Loading (Grams)										
	Air Flow (ft <sup>3</sup> /min)	Temp (°F)	Rel Hum (%)	Prefilter			Absolute		Halogen		PPM After				PPM After				Halogen		Iodine Removal Eff (%)		Prefilter		Absolute		Halogen	
				Initial	Final	Initial	Filter	Collector	Initial	Final	Inlet	Prefilter	Filter	Collector	Absolute	Halogen	Collector	Halogen	Prefilter	Absolute	Collector	Initial	Final	Initial	Final	Initial	Final	
SC-1-1	4000	70	70	0.07	0.08	0.62	0.64	0.37	0.42	0.1	0.0943	0.0330	0.00213	5.67	65.01	93.55	0.0203	0.1108	0.2202	1.1989	0.1109	0.6038						
SC-1-2		70	70	0.08	0.08	0.64	0.65	0.42	0.43	0.1	0.0722	0.00320	0.00160	27.80	95.57	49.87	0.2217	0.6653	1.4742	2.5755	0.6102	0.6357						
SC-1-3		70	70	0.075	0.08	0.68	0.70	0.42	0.46	0.1	0.0589	0.00505	(9)	41.12	91.42	99.95+	0.8851	1.5414	2.8633	3.7224	0.6627	0.7434						
SC-1-4		70	70	0.075	0.075	0.69	0.64	0.43	0.43	0.1	0.0963	0.0261	0.000948	3.73	72.89	96.37	1.5494	1.6044	3.8736	4.9095	0.7976	1.1690						
SC-2-1		70	70	0.07	0.075	0.62	0.62	0.43	0.425	1.0	--	--	--	(1)	41.89	96.62	1.6044	1.6044	6.3469	13.0323	3.0956	12.0563						
SC-2-2		70	70	0.07	0.08	0.60	0.64	0.42	0.44	1.0	--	--	--	(1)	48.45	92.04	1.6044	1.6044	14.5014	22.2338	13.4951	21.0679						
SC-2-3		70	70	0.075	0.075	0.68	0.68	0.46	0.46	1.0	--	--	--	(1)	51.03	83.09	1.6044	1.6044	25.0028	29.0749	23.2759	26.5230						
SC-2-4		70	70	0.08	0.08	0.72	0.72	0.42	0.42	1.0	--	--	--	(1)	60.25	81.02	1.6044	1.6044	32.5364	42.1518	28.3735	33.5138						
SC-2-5		70	70	0.08	0.08	0.72	0.72	0.44	0.42	1.0	--	--	--	(1)	51.80	70.80	1.6044	1.6044	49.3844	57.6503	38.2798	43.7267						
SC-3-1		70	95	0.07	0.07	0.67	0.67	0.38	0.38	1.0	0.974	0.309	0.0881	2.62	68.31	71.45	1.8285	2.2435	63.3459	73.9190	45.6176	49.1193						
SC-3-2		70	95	0.07	0.07	0.67	0.67	0.385	0.38	1.0	0.944	0.265	0.0714	5.56	71.89	73.11	2.9850	3.8456	82.9754	93.4873	51.7081	54.7130						
SC-3-3A	(6)	70	95	0.075	(7)	0.67	(7)	0.39	(7)	1.0	0.938	0.314	0.0986	6.21	66.57	68.58	--	4.2987	--	98.0481	--	56.2839						
SC-3-3B		70	95	0.075	0.075	0.67	0.67	0.39	0.385	1.0	0.997	0.353	0.124	0.33	64.64	64.05	4.3445	4.3969	106.9988	117.2283	59.4604	63.0906						
SC-4-1		70	95	0.075	0.075	0.67	0.68	0.385	0.385	10.0	9.06	5.33	2.14	9.38	41.24	59.88	7.7469	15.1915	130.5798	160.2497	74.4845	99.8042						
SC-4-2		70	95	0.075	0.075	0.68	0.68	0.39	0.39	10.0	8.13	6.93	2.76	18.72	14.71	60.14	18.1628	33.0195	162.1481	171.6399	106.4249	139.5286						
SC-4-3	(4)	70	95	0.07	(7)	0.67	(7)	0.38	(7)	10.0	8.95	6.65	3.51	10.53	25.66	47.23	34.9412	39.7037	175.8323	186.2221	145.2657	159.4836						
SC-4-4		70	95	0.075	(7)	0.70	(7)	0.42	(7)	10.0	8.65	8.13	4.68	13.48	6.10	42.42	41.3086	52.0082	186.8502	191.0378	163.5877	190.9485						
SC-5-1		70	95	0.085	0.10	0.71	0.80	0.37	0.395	0.1	0.0588	0.0103	0.00534	41.18	82.55	47.96	0.1144	0.7683	0.1349	0.9061	0.0137	0.0919						
SC-5-2		70	95	0.10	0.10	0.80	0.80	0.39	0.39	0.1	0.0716	0.00665	0.00428	28.42	90.72	35.68	0.8224	1.2737	1.0298	2.0610	0.0964	0.1341						
SC-5-3		70	95	0.09	0.09	0.80	0.80	0.39	0.39	0.1	0.0547	0.0131	0.00573	45.31	76.09	56.23	1.3564	2.0758	2.1370	2.7979	0.1475	0.2643						
SC-5-4		70	95	0.09	0.10	0.80	0.81	0.39	0.39	0.1	0.0543	0.00843	0.00724	45.67	84.50	14.05	2.3115	3.0366	3.0348	3.7639	0.2704	0.2892						
SC-6-1	(5)	160	95	0.07	0.07	0.68	0.68	0.27	0.27	0.1	0.0422	--	--	57.76	(2)	10.46	3.1722	3.4546	3.7639	3.7639	0.2996	0.3212						
SC-6-2	(5)	160	95	0.07	0.07	0.64	0.68	0.27	0.27	0.1	0.0552	0.0520	0.0313	44.78	5.80	39.82	3.5378	3.7568	3.7698	3.7855	0.3597	0.4610						
SC-6-3	(5)	160	95	0.07	0.07	0.68	0.68	0.27	0.275	0.1	0.0192	0.00573	0.00451	80.76	70.22	21.25	3.8555	4.2504	3.8020	3.8681	0.4625	0.4685						
SC-6-4		160	95	0.07	0.07	0.68	0.68	0.27	0.27	0.1	0.0116	0.00339	0.00278	88.40	70.80	18.06	4.3541	5.2186	3.8777	3.9581	0.4692	0.4752						
SC-6-5		160	95	0.07	0.07	0.64	0.68	0.27	0.27	0.1	0.0597	0.00464	0.00424	40.28	92.23	8.77	5.2422	5.6361	3.9904	4.5292	0.475							

TABLE III (Continued)

Test*	Operating Conditions			Pressure Drop (Inches of H <sub>2</sub> O)						Iodine Concentration						Iodine Removal Eff (%)						Cumulative Iodine Loading (Grams)								
	Air Flow (ft <sup>3</sup> /min)	Temp (°F)	Rel Hum (%)	Prefilter			Absolute		Halogen		PPM After Inlet			PPM After Prefilter			Absolute			Halogen			PPM After Collector			Absolute				
				Initial	Final	Filter	Initial	Final	Collector	Initial	Final	Filter	Collector	Prefilter	Absolute	Collector	Prefilter	Absolute	Collector	Prefilter	Absolute	Collector	Initial	Final	Filter	Initial	Final	Collector	Initial	Final
AC-1-1	4000	70	70	0.085	0.08	0.74	0.74	0.19	0.18	0.01	0.00115	0.000579	0.0000764	88.53	49.56	86.80	13.5645	13.7061	27.7621	27.7712	0.0018	0.0098								
AC-1-2		70	70	0.08	0.08	0.74	0.74	0.19	0.19	0.01	0.00113	0.000620	0.0000659	88.75	44.90	99.99	13.7381	13.8801	27.7730	27.7811	0.0120	0.0219								
AC-1-3		70	70	0.08	(7)	0.675	(7)	0.195	(7)	0.01	0.00160	0.000760	0.0000659	83.97	52.62	91.32	13.9043	14.0386	27.7835	27.7970	0.0239	0.0350								
AC-1-4		70	70	0.09	0.09	0.75	0.74	0.19	0.18	0.01	0.00191	0.00107	0.000115	80.93	43.80	89.27	14.0587	14.1882	27.7991	27.8124	0.0374	0.0527								
AC-2-1		70	95	0.085	0.085	0.75	0.75	0.19	0.19	0.01	0.00395	0.00234	0.000566	60.49	40.90	75.75	14.2159	14.3115	27.8198	27.8453	0.0608	0.0887								
AC-2-2		70	95	0.085	0.085	0.75	0.74	0.19	0.19	0.01	0.00257	0.00200	0.000457	74.28	22.43	97.72	14.3191	14.4365	27.8459	27.8550	0.0907	0.1215								
AC-2-3		70	95	0.085	0.085	0.75	0.75	0.19	0.19	0.01	0.00207	0.00101	(9)	79.30	51.36	99.99	14.4591	14.5844	27.8580	27.8748	0.1244	0.1403								
AC-3-1		160	95	0.065	0.065	0.67	0.66	0.135	0.13	0.01	0.00173	0.000503	0.0000312	82.69	70.97	93.81	14.6059	14.6869	27.8780	27.8900	0.1415	0.1461								
AC-3-2		160	95	0.065	0.065	0.68	0.65	0.13	0.135	0.01	0.00166	0.000734	0.0000304	83.36	55.92	95.86	14.7041	14.7858	27.8919	27.9010	0.1475	0.1544								
AC-3-3		160	95	0.07	0.065	0.66	0.66	0.135	0.14	0.01	0.00235	0.000418	0.0000605	76.47	82.23	85.54	14.7993	14.8742	27.9044	27.9234	0.1550	0.1585								
AC-4-1		160	95	0.065	0.065	0.65	0.66	0.135	0.135	0.1	0.0755	0.00729	0.000619	24.52	90.34	91.52	14.9114	15.1512	28.0268	28.6936	0.1686	0.2339								
AC-4-2		160	95	0.065	0.065	0.67	0.67	0.13	0.13	0.1	0.0844	0.00849	0.0000168	15.59	89.94	99.81	15.1764	15.3288	28.8161	29.5586	0.2476	0.3305								
AC-4-3A	(8)	160	95	(7)	(7)	0.76	(7)	0.13	(7)	0.1	0.0739	0.00685	0.000205	26.07	90.74	97.02	—	15.3925	—	29.7226	—	0.3467								
AC-4-3B		160	95	0.06	0.06	0.58	0.58	0.11	0.11	0.1	0.0706	0.00746	0.000281	29.40	89.44	96.24	15.4256	15.7130	29.7936	30.4112	0.3548	0.4250								
AC-5-1		70	70	0.085	0.09	0.72	0.78	0.18	0.20	0.1	0.0719	0.00519	(9)	28.14	92.78	99.99	15.7983	16.2473	30.6134	31.6776	0.4407	0.5236								
AC-5-2		70	70	0.09	0.09	0.77	0.76	0.20	0.20	0.1	0.0673	0.00541	0.000396	32.69	91.57	92.69	16.3255	16.8472	31.8258	32.8139	0.5356	0.6156								
AC-5-3		70	70	0.09	0.09	0.77	0.76	0.20	0.20	0.1	—	—	—	(1)	90.94	94.00	16.8472	16.8472	32.9082	34.3596	0.6244	0.7604								
AC-6-1		70	95	0.09	0.09	0.78	0.78	0.20	0.21	0.1	0.0636	0.0276	0.000212	36.38	56.60	99.24	16.9136	17.4912	34.4254	34.9972	0.8105	1.2457								
AC-6-2	(10)	70	95	0.09	0.095	0.79	0.79	0.21	0.21	0.1	0.0492	0.0107	0.000743	50.83	78.23	93.06	17.6042	18.4114	35.0827	35.6935	1.2679	1.4261								
AC-6-3		70	95	0.09	0.09	0.84	0.84	0.21	0.21	0.1	0.0832	0.0127	0.000787	16.85	82.72	93.82	18.4381	18.7056	35.8054	36.9240	1.4450	1.6344								
AC-6-4		70	95	0.09	0.09	0.80	0.80	0.21	0.215	0.1	0.0943	0.0421	0.00129	5.66	55.43	96.93	18.7065	18.7963	36.9323	37.7627	1.6409	2.2882								
AC-7-1		70	95	0.09	0.09	0.79	0.79	0.21	0.21	1.0	—	—	—	(1)	45.05	98.98	18.7963	18.7963	40.3736	43.9501	5.4410	9.7599								
AC-7-2		70	95	0.08	0.08	0.79	0.79	0.21	0.21	1.0	—	—	—	(1)	41.93	96.08	18.7963	18.7963	44.1165	47.4451	9.9814	14.4120								
AC-7-3		70	95	0.11	0.11	1.35	1.10	0.21	0.21	1.0</																				

TABLE III (Continued)

Test*	Operating Conditions			Pressure Drop (Inches of H <sub>2</sub> O)						Iodine Concentration				Iodine Removal Eff (%)			Cumulative Iodine Loading (Grams)					
	Air Flow (ft <sup>3</sup> /min)	Temp (°F)	Rel Hum (%)	Prefilter		Absolute Filter		Halogen Collector		PPM Inlet	PPM Prefilter	PPM Absolute Filter	PPM Halogen Collector	Prefilter	Absolute Collector	Halogen Collector	Prefilter		Absolute Filter		Halogen Collector	
				Initial	Final	Initial	Final	Initial	Final								Initial	Final	Initial	Final	Initial	Final
C-1-1	4000	70	70	0.09	0.09	0.76	0.76	0.16	0.16	0.01	0.000818	0.000698	0.000136	91.83	14.71	80.45	28.0501	28.1970	64.4408	64.4427	0.0018	0.0108
C-1-2		70	70	0.09	0.09	0.76	0.76	0.17	0.17	0.01	0.00145	0.000747	0.00000783	85.49	48.57	98.96	28.2312	28.3680	64.4455	64.4568	0.0138	0.0256
C-1-3		70	70	0.08	0.08	0.70	0.70	0.15	0.14	0.01	0.00245	0.00112	0.0000662	75.52	54.37	94.08	28.3759	28.4967	64.4582	64.4795	0.0267	0.0435
C-1-4		70	70	0.08	0.08	0.70	0.71	0.14	0.15	0.01	0.00189	0.00109	(9)	81.13	42.23	99.99+	28.5019	28.6317	64.4800	64.4928	0.0442	0.0616
C-2-1		70	95	0.085	0.085	0.76	0.76	0.16	0.16	0.01	0.00228	0.00110	0.0000008	77.16	51.86	99.93	28.6530	28.7749	64.4961	64.5148	0.0646	0.0820
C-2-2		70	95	0.085	0.085	0.76	0.76	0.16	0.16	0.01	0.00310	0.00139	(9)	69.03	55.07	99.99+	28.7787	28.8878	64.5157	64.5427	0.0828	0.1047
C-2-3		70	95	0.08	0.09	0.74	0.77	0.15	0.165	0.01	0.00307	0.00129	0.0000668	69.31	58.09	94.81	28.8960	29.0055	64.5448	64.5720	0.1061	0.1254
C-3-1		160	95	0.07	0.07	0.68	0.68	0.12	0.12	0.01	0.000857	0.000555	0.0000132	91.43	35.24	97.62	29.0355	29.1251	64.5740	64.5770	0.1272	0.1325
C-3-2		160	95	0.07	0.07	0.68	0.68	0.12	0.12	0.01	0.00115	0.000832	0.000172	88.51	27.65	79.28	29.1316	29.2183	64.5772	64.5804	0.1330	0.1394
C-3-3		160	95	0.06	0.06	0.62	0.64	0.10	0.10	0.01	0.00177	0.000427	(9)	82.26	75.93	99.99+	29.2256	29.3062	64.5816	64.5948	0.1398	0.1440
C-3-4		160	95	0.06	0.06	0.62	0.62	0.10	0.10	0.01	0.00136	0.000864	0.000209	86.43	36.40	75.83	29.3117	29.3964	64.5951	64.6000	0.1444	0.1508
C-3-5		160	95	0.06	0.06	0.64	0.68	0.10	0.12	0.01	0.00110	0.000331	0.0000736	89.03	69.80	77.81	29.4038	29.4911	64.6006	64.6081	0.1510	0.1536
C-3-6	(13)	160	95	0.06	0.06	0.64	0.62	0.10	0.10	0.01	0.00631	0.000267	(9)	36.87	95.77	99.99+	32.6726	32.7087	65.7039	65.7632	0.3460	0.3486
C-4-1		160	95	0.07	0.07	0.68	0.68	0.12	0.12	0.1	0.0407	0.00785	0.00118	59.33	80.71	84.93	29.5549	30.1351	64.6434	64.9645	0.1608	0.2260
C-4-2	(12)	160	95	0.06	0.06	0.64	0.64	0.10	0.10	0.1	0.0477	0.00471	(9)	52.34	90.12	99.99+	30.1658	30.6776	64.9897	65.4098	0.2288	0.2748
C-4-3A	(11)	160	95	0.06	(7)	0.64	(7)	0.10	(7)	0.1	0.0231	0.00365	(9)	76.91	84.18	99.99+	--	30.8056	--	65.4573	--	0.2837
C-4-3B		160	95	0.06	0.06	0.62	0.62	0.10	0.10	0.1	0.0195	0.00510	(9)	80.51	73.82	99.99+	30.9365	31.7238	65.4700	65.6107	0.2882	0.3381
C-4-4	(13)	160	95	0.06	0.06	0.64	0.64	0.10	0.09	0.1	0.00921	0.000737	(9)	90.80	92.00	99.99+	31.7815	32.6695	65.6161	65.6989	0.3386	0.3458
C-5-1		70	70	0.09	0.09	0.78	0.78	0.17	0.17	0.1	0.0111	0.00560	(9)	88.89	49.62	99.99+	32.8860	34.3046	65.7742	65.8622	0.3598	0.4492
C-5-2		70	70	0.09	0.09	0.76	0.76	0.16	0.16	0.1	0.0407	0.00584	0.000324	59.29	85.65	94.46	34.4465	35.3928	65.9457	66.5022	0.4624	0.5505
C-5-3		70	70	0.09	0.09	0.74	0.74	0.16	0.16	0.1	--	--	--	(1)	81.73	99.99+	35.3928	35.3298	66.6196	67.9240	0.5767	0.8683
C-6-1		70	95	0.09	0.09	0.74	0.76	0.16	0.17	0.1	--	--	--	(1)	67.83	97.06	35.3928	35.3928	68.0209	69.0980	0.9129	1.4089
C-6-2		70	95	0.09	0.09	0.77	0.77	0.17	0.17	0.1	0.0741	0.0236	0.00170	25.86	68.15	92.82	35.4031	35.8137	69.1181	69.9204	1.4176	1.7657
C-6-3		70	95	0.095	0.095	0.78	0.78	0.17	0.17	0.1	0.0679	0.0132	0.00141	32.11	80.63	89.27	35.8647	36.3744	70.0073	70.8767	1.7843	1.9708
C-7-1	(14)	70	95	0.09	0.09	0.78	0.78	0.17	0.17	1.0	--	--	--	(1)	74.93	96.15	36.3744	36.3744	71.7691	77.7183	2.2579	4.1721
C-7-2		70	95	0.09	0.09	0.77	0.77	0.17	0.17	1.0	--	--	--	(1)	70.97	95.50	36.3744	36.3744	78.2818	82.9586	4.3922	6.2195
C-7-3		70	95	0.09	0.09	0.77	0.77	0.17	0.17	1.0	--	--	--	(1)	65.54	94.46	36.3744	36.3744	83.1147	88.3178	6.2971	8.8821
C-8-1		160	95	0.07	0.07	0.66	0.66	0.11	0.11	1.0	--	--	--	(1)	83.52	99.99+	36.3744	36.3744	88.9304	93.0145	9.0030	9.8089
C-8-2		160	95	0.07	0.07	0.66	0.66	0.11	0.11	1.0	--	--	--	(1)	7							

TABLE III (Continued)

Test*	Operating Conditions			Pressure Drop (Inches of H <sub>2</sub> O)						Iodine Concentration						Iodine Removal Eff (%)						Cumulative Iodine Loading (Grams)							
	Air Flow (ft <sup>3</sup> /min)	Temp (°F)	Rel Hum (%)	Prefilter		Absolute Filter		Halogen Collector		PPM Inlet	PPM Prefilter	PPM Absolute Filter	PPM Halogen Collector	Halogen Eff (%)	Prefilter	Absolute Filter	Halogen Eff (%)	Prefilter	Absolute Filter	Halogen Eff (%)	Prefilter		Absolute Filter		Halogen Collector				
				Initial	Final	Initial	Final	Initial	Final												Initial	Final	Initial	Final	Initial	Final			
MS-3-1	4000 (15)	160	95	0.06	0.06	0.61	0.63	0.14	0.14	0.01	0.00489	0.00167	0.000133	51.15	65.91	92.00	36.3824	36.4325	101.7209	101.7525	0.0024	0.0174							
MS-3-2	(15)	160	95	0.06	0.06	0.61	0.64	0.14	0.14	0.01	0.00263	0.00137	0.000373	73.71	47.84	72.82	36.4368	36.5091	101.7532	101.7656	0.0180	0.0278							
MS-3-3	(15)	160	95	0.07	0.07	0.64	0.64	0.14	0.14	0.01	0.000801	0.0000909	0.0000909	91	92.00	88.65	99.99	36.5159	36.6060	101.7661	101.7731	0.0279	0.0287						
MS-3-4	(15)	160	95	0.07	0.07	0.64	0.64	0.14	0.14	0.01	0.00194	0.000621	0.0000792	80.63	67.95	87.25	36.6080	36.6838	101.7734	101.7858	0.0288	0.0339							
MS-1-1	(15)	70	70	0.09	0.09	0.77	0.77	0.23	0.22	0.01	0.00235	0.000614	--	76.55	73.82	(3)	36.6930	36.8155	101.7879	101.8156	0.0339	0.0339							
MS-1-2	70	70	0.09	0.09	0.76	0.76	0.18	0.18	0.01	0.00143	0.000755	0.000495	85.70	47.21	34.40	36.8244	36.9615	101.8163	101.8271	0.0342	0.0383								
MS-3-5	160	95	0.06	0.06	0.64	0.64	0.11	0.11	0.01	0.000748	0.000268	0.000133	92.53	64.21	50.48	36.9674	37.0581	101.8274	101.8321	0.0384	0.0397								
MS-3-6	160	95	0.06	0.06	0.64	0.64	0.11	0.11	0.01	0.00192	0.000630	0.000423	80.80	67.19	32.88	37.0648	37.1440	101.8332	101.8458	0.0399	0.0419								
MS-3-7	160	95	0.06	0.06	0.64	0.64	0.11	0.11	0.01	0.00369	0.00143	0.00101	63.06	61.25	29.74	37.1493	37.2111	101.8477	101.8699	0.0423	0.0464								
T-1-1	70	70	0.09	0.09	0.66	0.76	0.19	0.20	0.01	0.000836	--	--	91.65	(2)	74.57	37.2206	37.3673	101.8699	101.8699	0.0006	0.0106								
T-1-2	70	70	0.09	0.09	0.76	0.76	0.19	0.19	0.01	0.000508	--	--	94.92	(2)	83.79	37.3840	37.5359	101.8699	101.8699	0.0114	0.0182								
T-1-3	70	70	0.09	0.09	0.76	0.76	0.19	0.19	0.01	0.000601	0.000232	0.000163	94.00	61.48	29.69	37.5397	37.6901	101.8700	101.8760	0.0182	0.0193								
T-1-4	70	70	0.09	0.09	0.76	0.76	0.20	0.20	0.01	0.00103	0.000581	0.000445	89.69	43.65	23.51	37.6951	37.8386	101.8763	101.8835	0.0194	0.0216								
T-2-1	70	95	0.09	0.09	0.78	0.78	0.19	0.135	0.01	0.000725	0.000346	0.000202	92.75	52.30	41.78	37.8481	37.9947	101.8839	101.8899	0.0217	0.0240								

## NOTES

- Indications are that desorption occurred in the prefilter.
- Indications are that desorption occurred in the absolute filter.
- Indications are that desorption occurred in the Halogen collector.
- Test SC-4-3 was only 34 min long.
- Test terminated after 1 hr because of liquid buildup in analytical scrubbers.
- Test terminated just after start because of burner trouble.
- Not recorded.
- Test SC-4-3A terminated after burner was on for 1/2 hr.
- Too small for measurement.
- Unable to determine halogen-collector efficiency because of difficulty with No. 4 analytical scrubber. Filter loading was determined by use of average efficiency for these operating conditions.
- Test C-4-3A was only 17 min long.
- Difficulty with No. 1 analytical scrubber. The prefilter efficiency was estimated from the average of other tests at these operating conditions.
- Test C-3-6 was run after test C-4-4.
- Difficulty developed with No. 2 analytical scrubber. Efficiencies for the prefilters and absolute filters were estimated from the average of other tests at these operating conditions.
- Air admitted in the halogen-collector section of the filter holder.

## TEST

SC Silver-plated copper  
 AC Activated charcoal 1/2  
 C Activated charcoal 2"  
 MS Molecular sieve 1"  
 T Sodium thiosulfate

## VI. ANALYSIS OF RESULTS

As illustrated in Table III, the activated-charcoal halogen collectors were the only ones tested that continually showed what we believe to be an acceptable iodine removal efficiency. They performed creditably at each set of operating conditions. Loading tests performed on the 1/2" activated-charcoal bed were discontinued without evidence of a breakthrough, despite a total iodine loading of 1535 grams in the first test and 1107 grams in the second. This is illustrated by Figures 1 and 3. Desorption was observed over a period of 45 hours following the first loading test. The total amount of iodine leakage over this period was 6.85 grams. The rate of iodine leakage as a function of time is shown in Figure 2. Iodine leakage over a period of several hours following the second loading test was negligible. Because of these results, no loading tests were performed on the 1" activated-charcoal filters.

On the basis of the iodine-removal-efficiency tests, there is little to choose from between the 1/2" and 1" activated-charcoal beds. The 1" bed achieved an average efficiency about 1.5% higher than that of the 1/2" bed and appeared to offer a more consistent performance.

Completion of the required program with the silver-plated copper-ribbon beds required the use of two sets of filters. Efficiencies were generally good until a cumulative loading of about 12 grams of iodine was achieved. From this point, the efficiencies declined an average of 1.43% per hour at an iodine injection rate of 1.0 ppm and 4.60% per hour at an injection rate of 10.0 ppm. (See Figure 4.) At the halfway mark of the test series, the second set of filters was installed. As a result of the difficulties encountered in the degreasing operation--described in Section V--no valid data were obtained from this set of filters.

The molecular-sieve halogen collectors were tested initially at 160°F and 95% relative humidity. Their greater affinity for water--which was to a degree expected--was quickly determined when the iodine removal efficiency began to fall off after the test system had operated for about 13 hours at these conditions. The burner had operated for about 8 hours and only 0.0339 gram of iodine was being held on the filters. Published data indicate that the iodine adsorbed would be displaced by water as the filter approached its capacity. These filters would not be recommended for use where the bed would be exposed to a substance for which its affinity would be greater than that for iodine. Water vapor is the most abundant of these substances.

The performance of the sodium thiosulfate was poor. After several hours of testing, the bed broke down completely. It appears that as the bed picked up water from the gas stream, it began to sag because of the additional weight. In addition, an undetermined part of the bed was carried off in the gas stream and recrystallized on the base of the test section behind the halogen-collector filter bank. These filters could not be recommended for use under any of the specified test conditions.

A test was conducted on one of the first set of silver-plated copper ribbon beds to determine whether iodine was adsorbed on the surface of the bed. Chemical analysis of an alcohol rinse solution gave no indication of iodine. When testing was resumed on this filter, the efficiency had not improved. It is believed that iodine is held on these filters by chemical combination with the filter media.

It was observed in Table III that where a breakthrough had occurred, an increase in the relative humidity was accompanied by a temporary increase in iodine removal efficiency.

The tests on the 1/2" activated-charcoal bed showed that efficiency increased slightly as the iodine concentration increased. The 1" charcoal bed shows the same characteristics, with a smaller differential between concentrations. This may be due in part to the increased partial pressure of iodine at the higher concentrations.

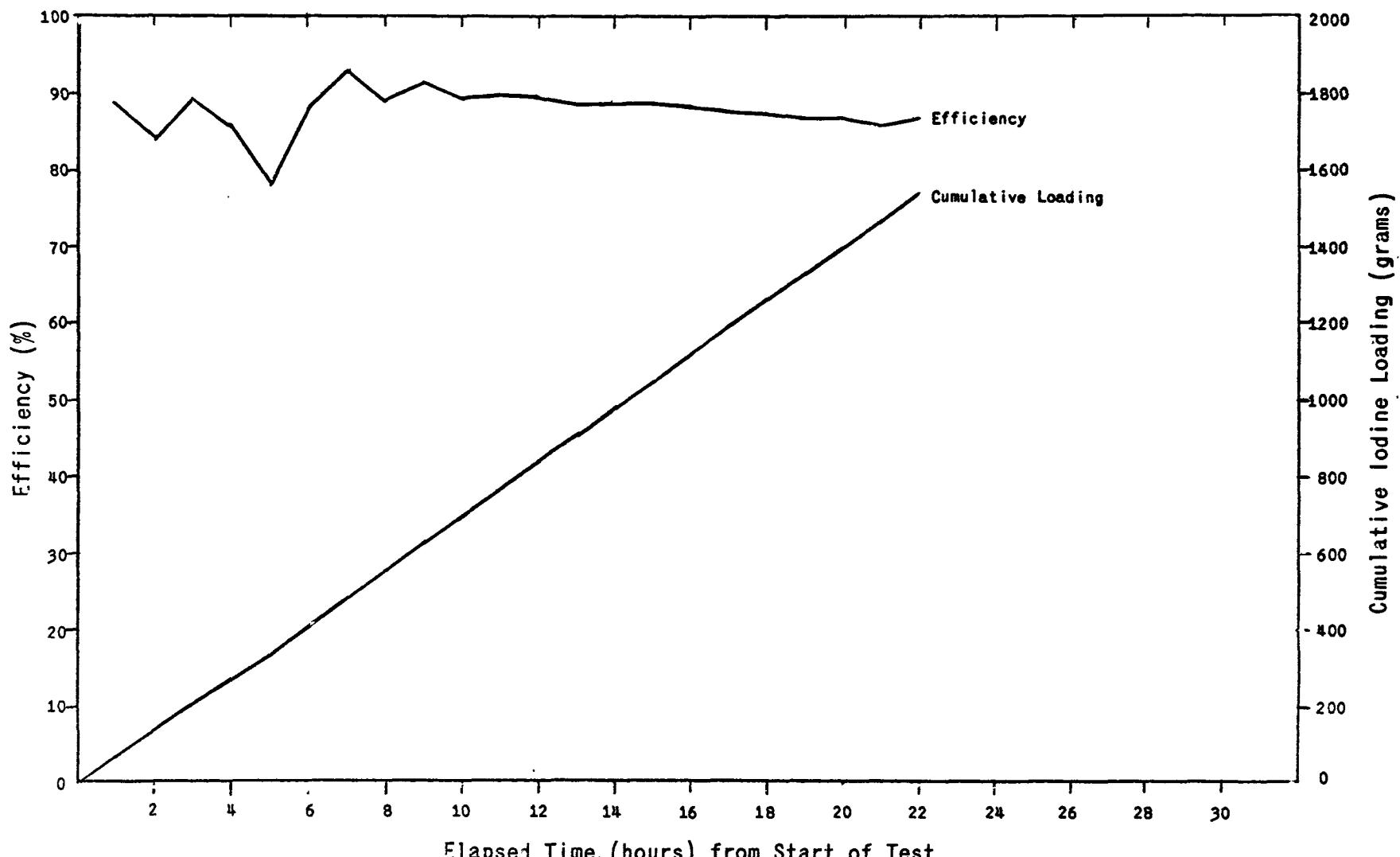


Figure 1

EFFICIENCY AND CUMULATIVE LOADING OF HALF-INCH ACTIVATED-CHARCOAL BED  
AT 70°F AND 95% RELATIVE HUMIDITY

HW-65587  
ALI-54

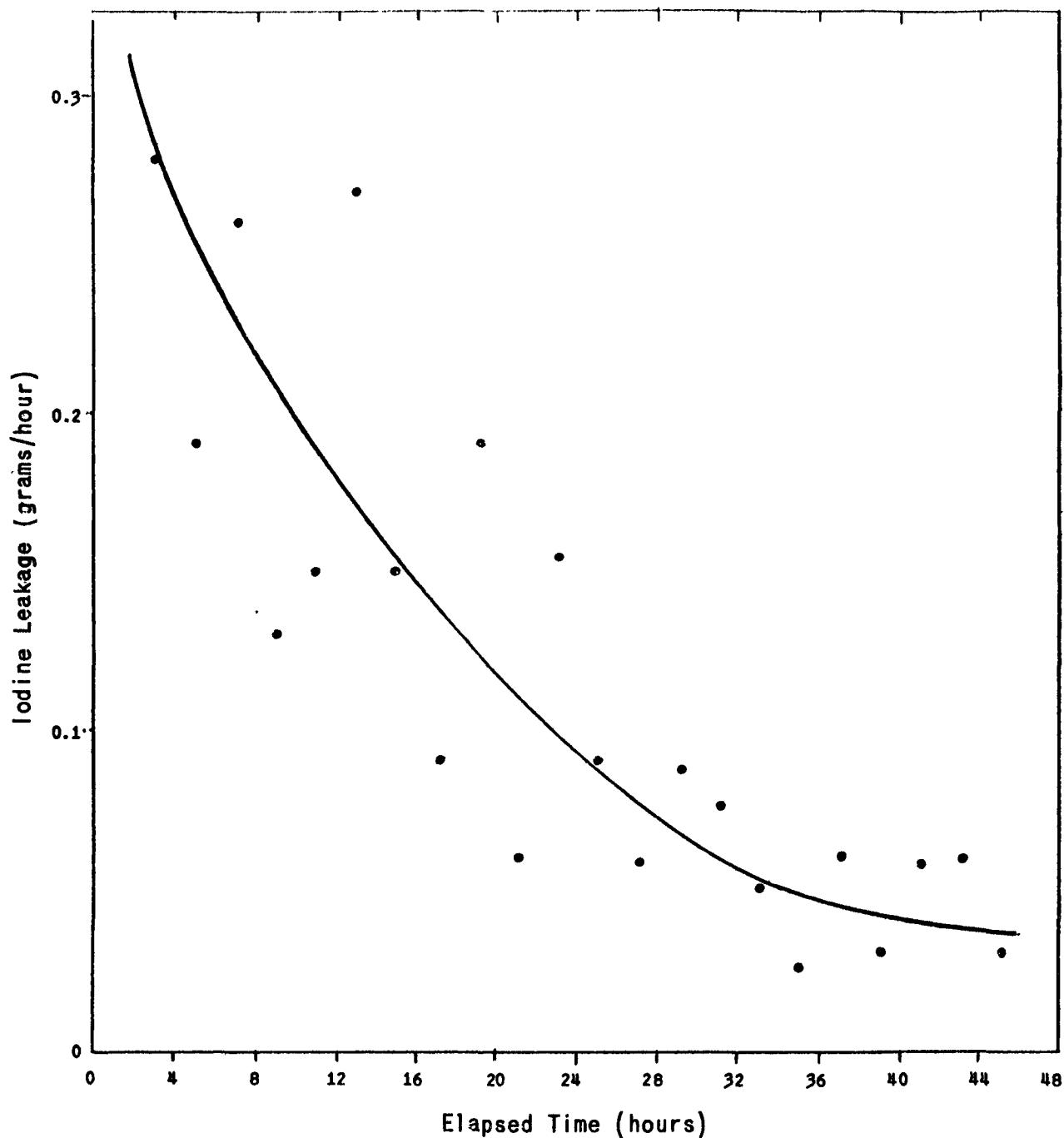


Figure 2  
DESORPTION RATE OF IODINE FROM HALF-INCH ACTIVATED-CHARCOAL  
BED LOADED AT 70°F AND 95% RELATIVE HUMIDITY

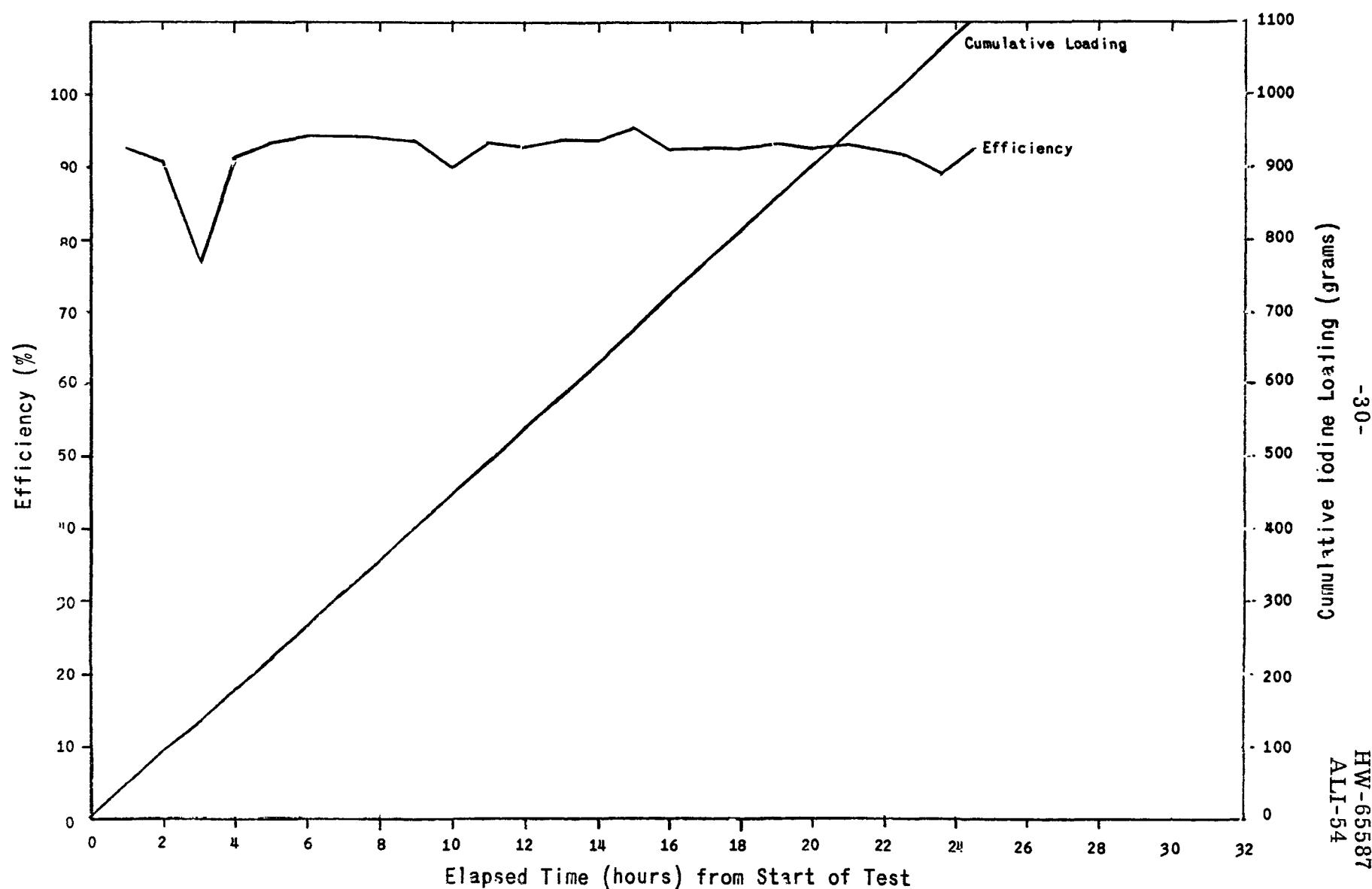


Figure 3

EFFICIENCY AND CUMULATIVE LOADING OF HALF-INCH ACTIVATED-CHARCOAL BED  
AT 160 F AND 95% RELATIVE HUMIDITY

HW-6587  
ALL-54

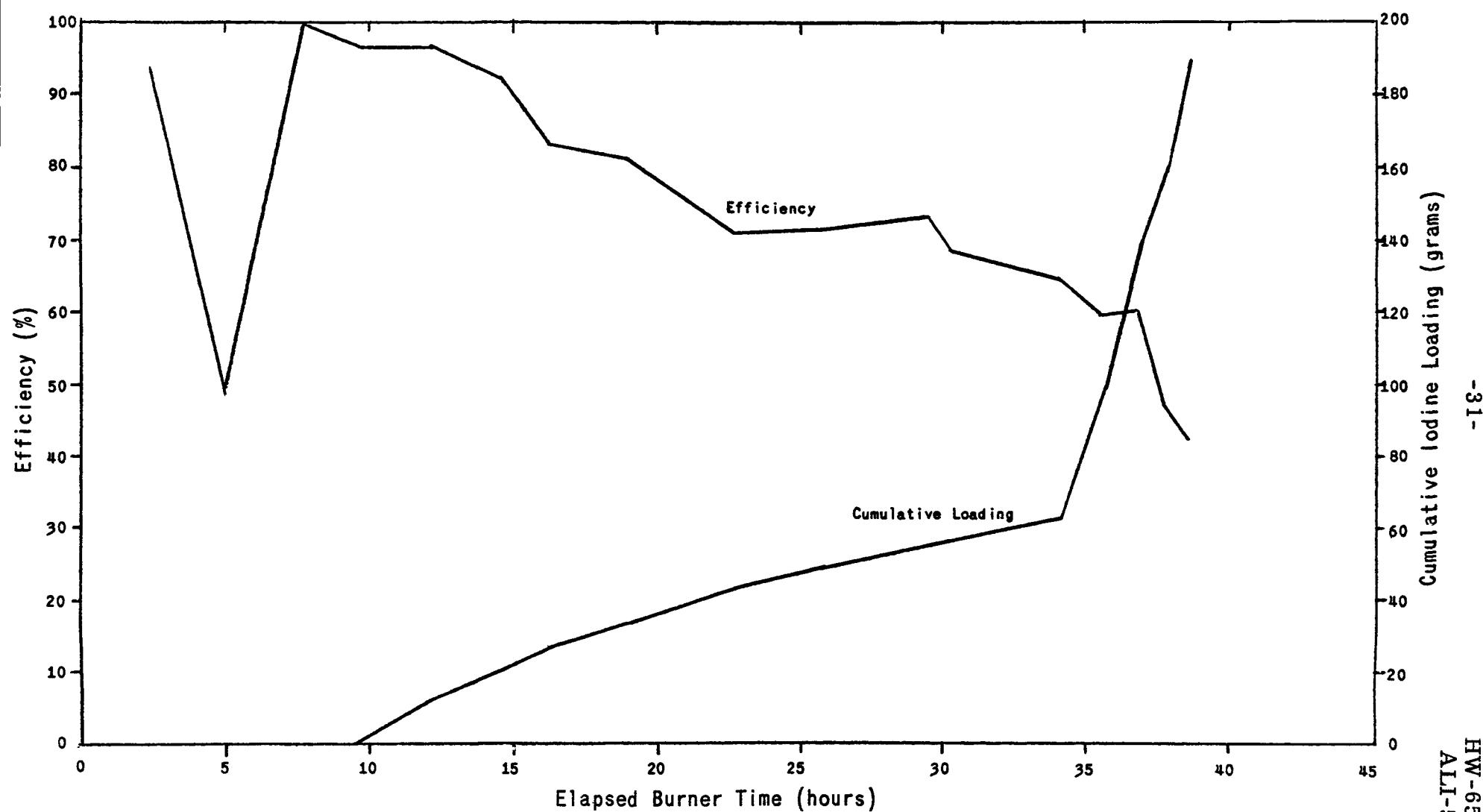


Figure 4

EFFICIENCY AND CUMULATIVE LOADING OF TWO-INCH SILVER-PLATED COPPER-RIBBON BED

HW-65587  
ALI-54

-31-

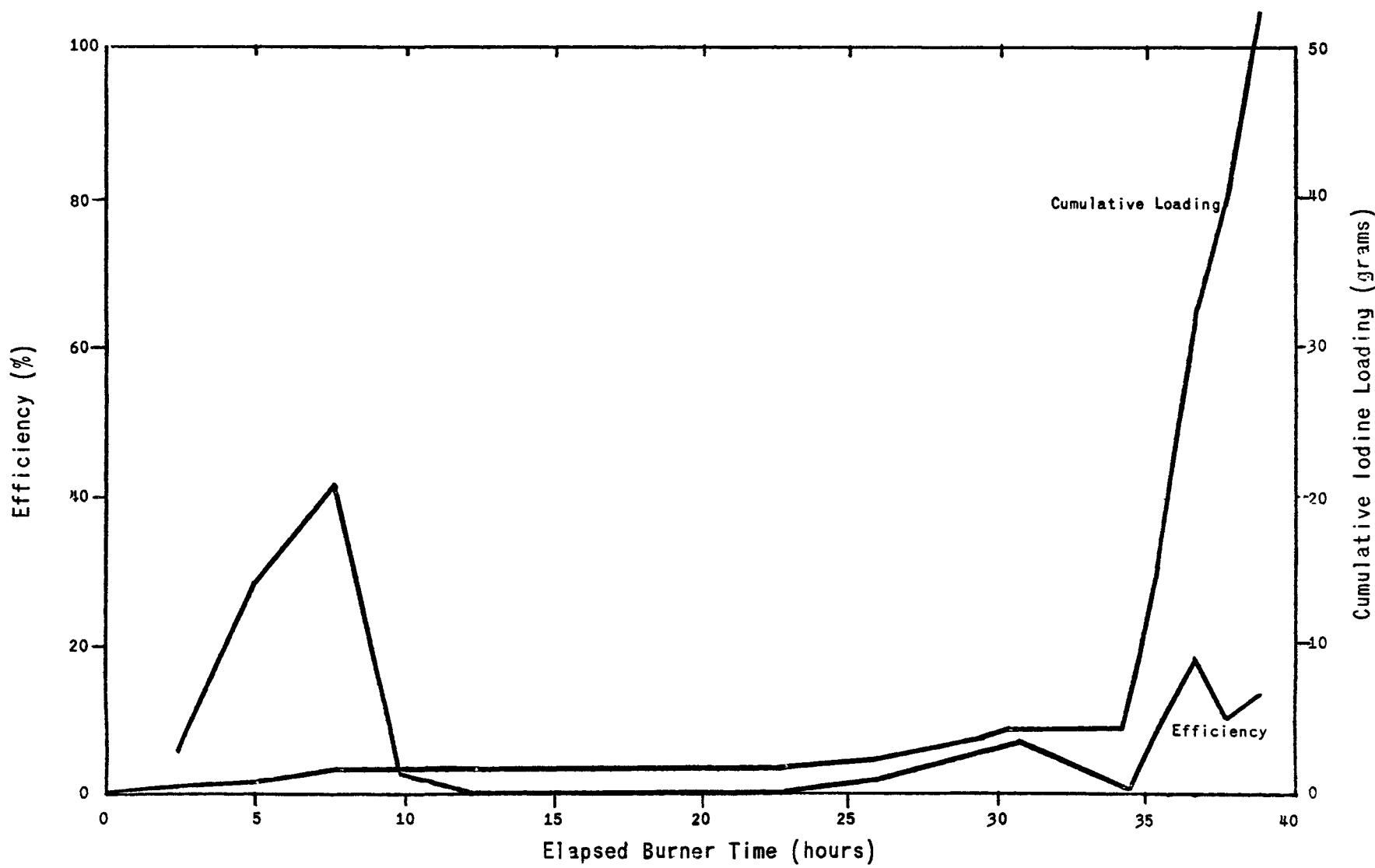


Figure 5

EFFICIENCY AND CUMULATIVE LOADING OF PREFILTERS--FIRST SET

HW-6587  
ALL-54

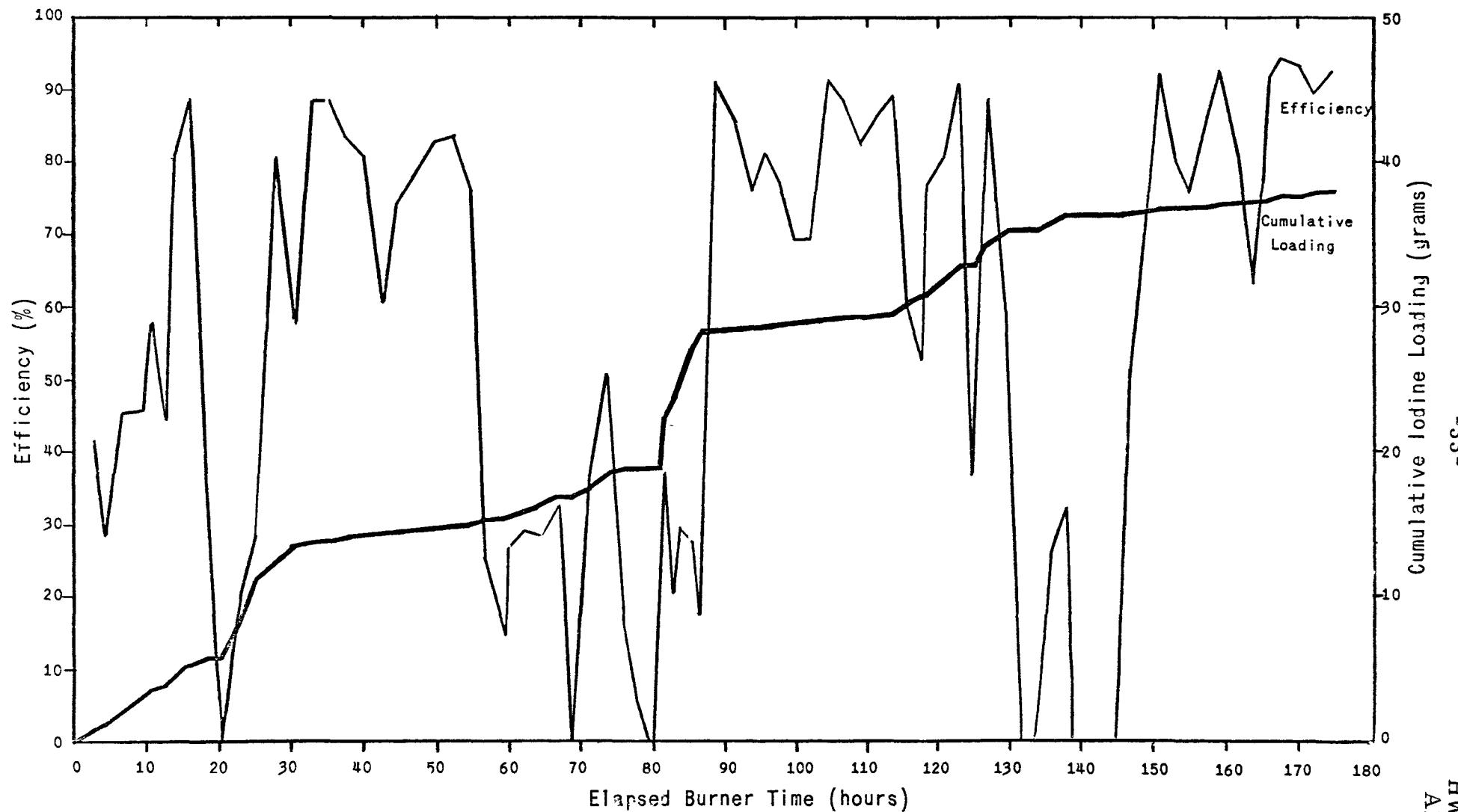


Figure 6  
EFFICIENCY AND CUMULATIVE LOADING OF PREFILTERS--SECOND SET

ALI-54  
HW-65587

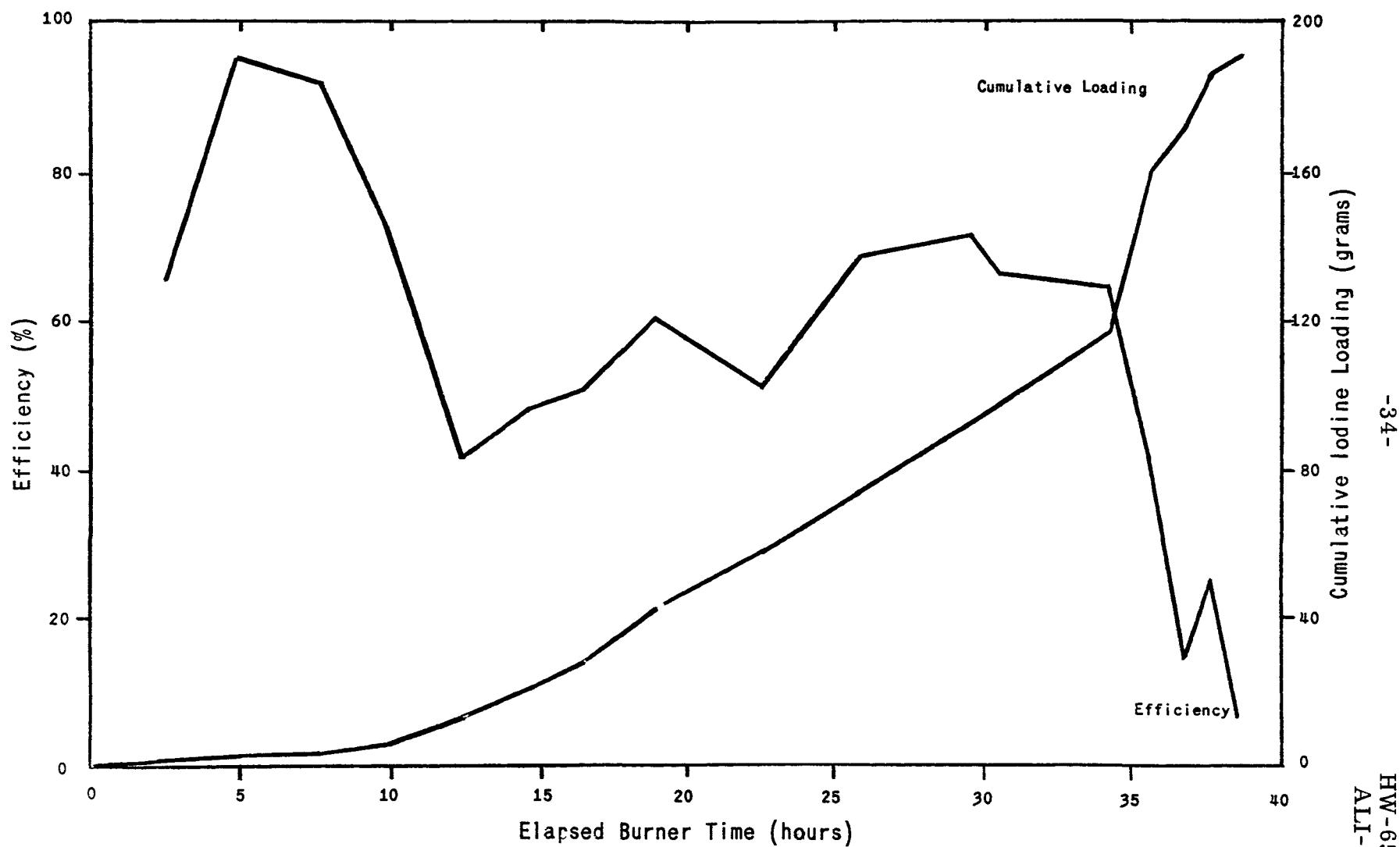


Figure 7  
EFFICIENCY AND CUMULATIVE LOADING OF ABSOLUTE FILTERS--FIRST SET

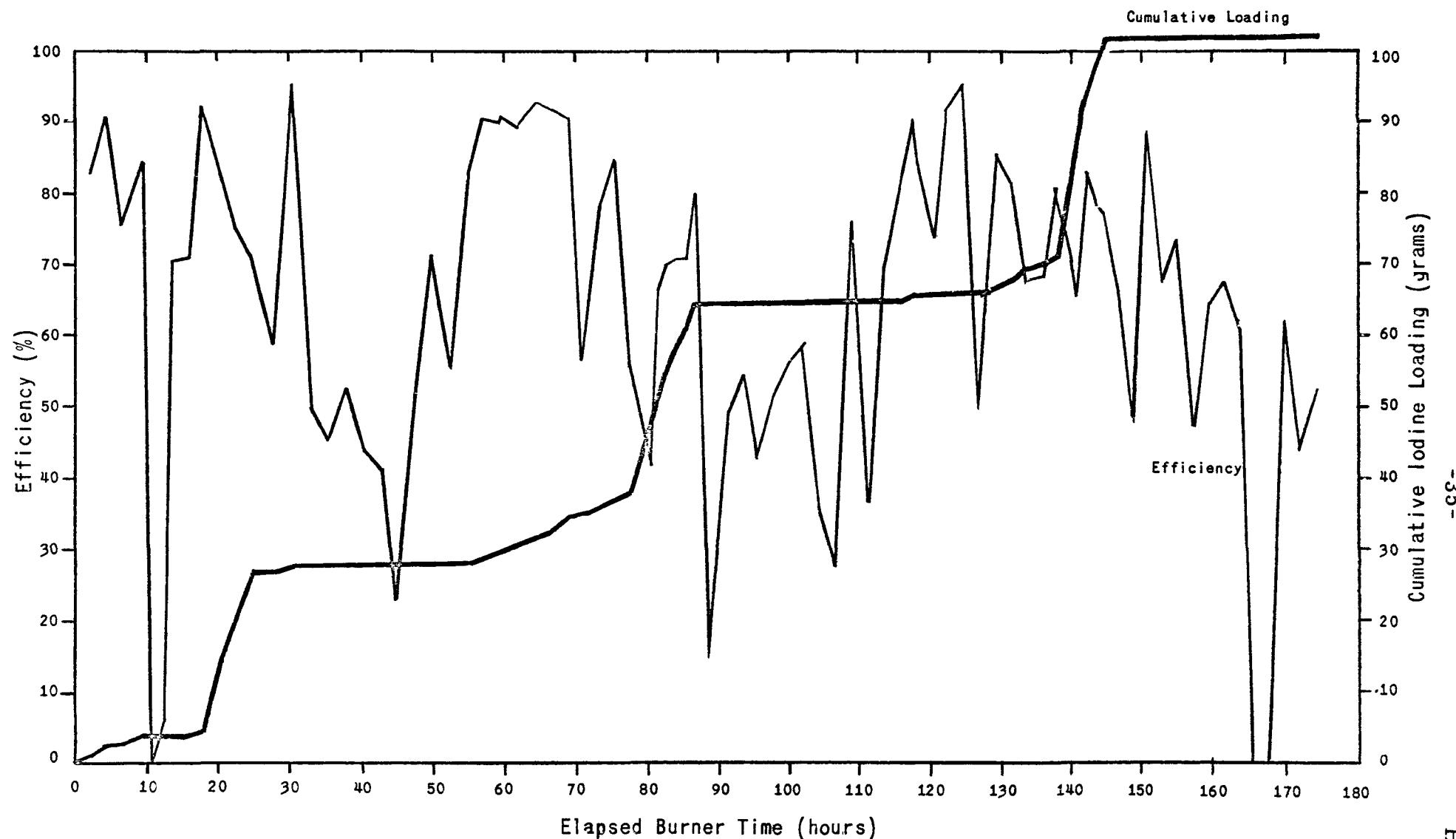


Figure 8

EFFICIENCY AND CUMULATIVE LOADING OF ABSOLUTE FILTERS--SECOND SET

HW-65587  
ALI-54

## VII. TEST PROBLEMS

In the performance of this test program, only two problems that arose necessitated our deviation from the program specifications. Both of these slight modifications were endorsed by E. L. Etheridge of General Electric.

In the iodine removal-efficiency test runs it was not possible to determine the "leakage" rates of the components, because of the variation in the history of the I-131 used for tracing. Since the amount of I-131 had to be held more or less constant in the injected iodine for analytical accuracy, the ratio and disposition of I-131 and I-127 in the filter became extremely complex because of the short half-life of I-131 and the variations in the iodine injection rate. If desorption or "leakage" occurred, it was impossible to determine the 131/127 ratio of the desorbed iodine and therefore the amount of iodine desorbed.

Because of this problem the loading tests were run in such a manner that the history of the iodine injected was the same as that present on the filter. That is, the ratio of the I-131 to I-127 in the feed solution was maintained the same as that of the already absorbed iodine. In this way it was possible to determine the amount of iodine desorbed during the leakage-rate test.

The other problem encountered was control of relative humidity at 100%. At this humidity, wet- and dry-bulb temperatures are the same; if excess moisture is added there is no rapid means of sensing it, for the wet-bulb temperature will remain the same as that of the dry bulb. Since the absolute filters are sensitive to free moisture, it was decided to run with a 1°F difference between wet- and dry-bulb temperatures, thus enabling satisfactory control of test conditions. This one-degree difference resulted in a relative humidity closer to 95%.

VIII. FUTURE DEVELOPMENT WORK RECOMMENDED

From the results obtained we believe that future work on halogen collectors for large air volumes should be focused on the improvement and durability of charcoal filters. It is quite possible that new absorbing materials will be developed, but we believe that in the field of the dry-type collectors, charcoal will be difficult to beat. We believe that future development work should be of the following nature:

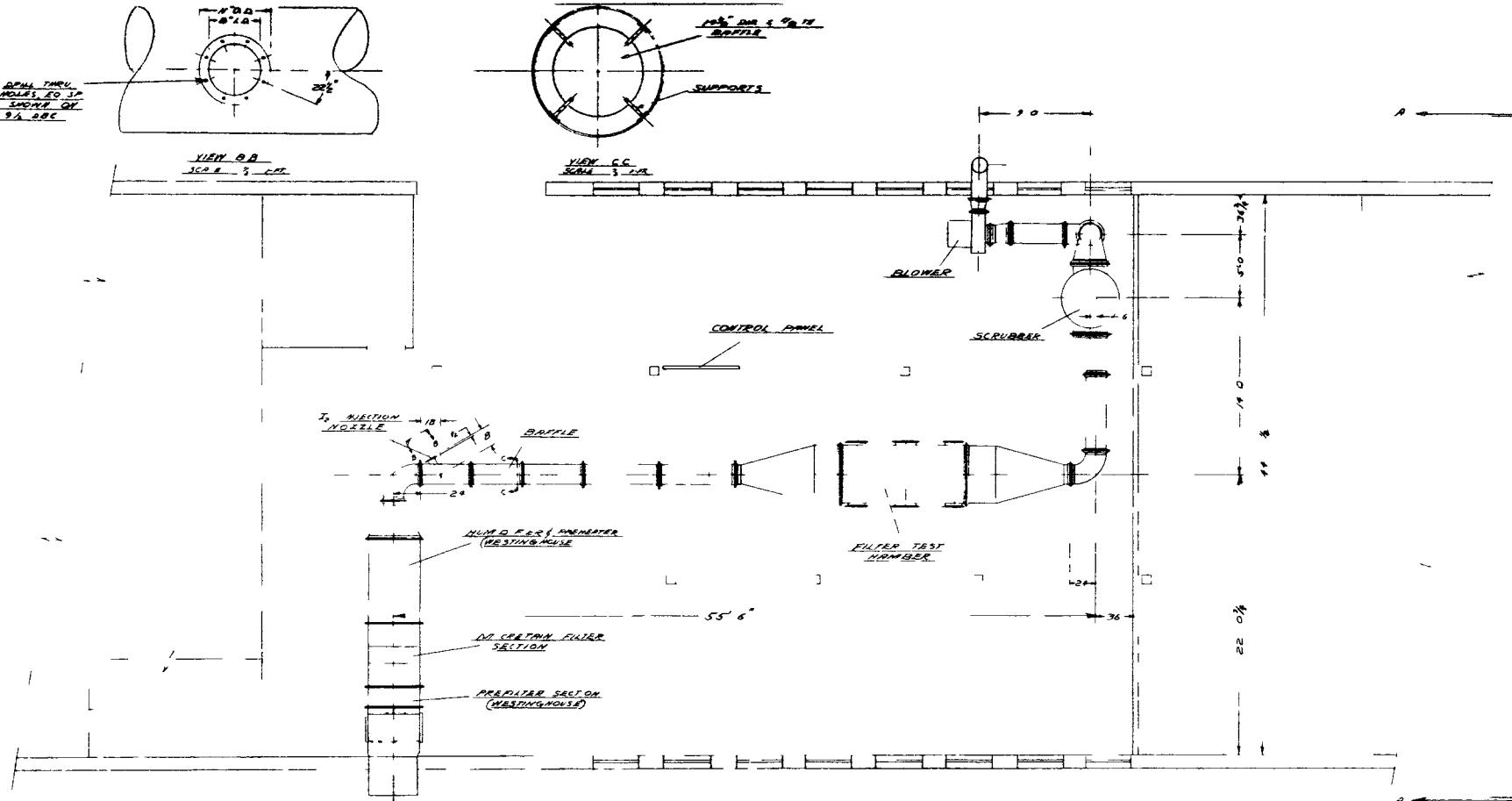
1. A program to develop for the charcoal-type absorbers a fireproofing agent that would not affect their absorbing capacity. (We have done considerable work of this nature on other types of material and have had quite satisfactory results.)
2. A program to check periodically the effects of "aging" of the charcoal in actual use, to determine the effect of time on collection efficiency and loading capacity.

-38-

HW-65587  
ALI-54

APPENDIX A

FIGURES 1A TO 19A



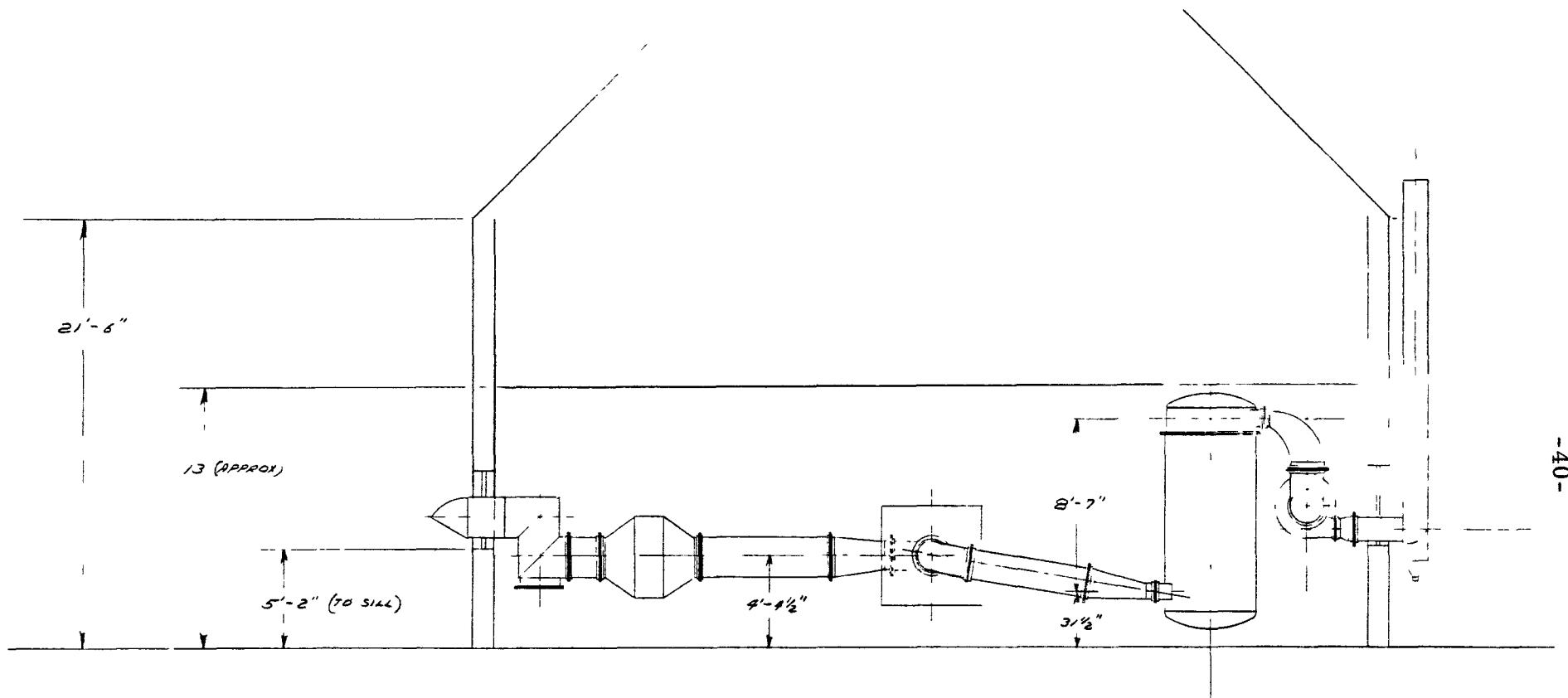
REFERENCE DRAWINGS  
VIEW A-A D 62249 2

OPERATING CONDITIONS  
TEMP = 160°F  
VACUUM = 10 OF INCHES (0.26 MMHG)

MATERIAL  
FROM TWO FEET UPSTREAM OF  
THE INLET ON NOZZLE TO THE ENTRANCE  
OF THE SCRUBBER SHELL BE ONE IN  
STAINLESS STEEL SHEET 304 THE REST  
OF THE MATERIAL SHALL BE GALVANIZED  
OR A THICKNESS TO SATISFY OPERATING  
CONDITIONS

Figure 1A  
HALOGEN COLLECTOR TEST PROGRAM, EQUIPMENT LAYOUT

HW-65587  
ALI-54

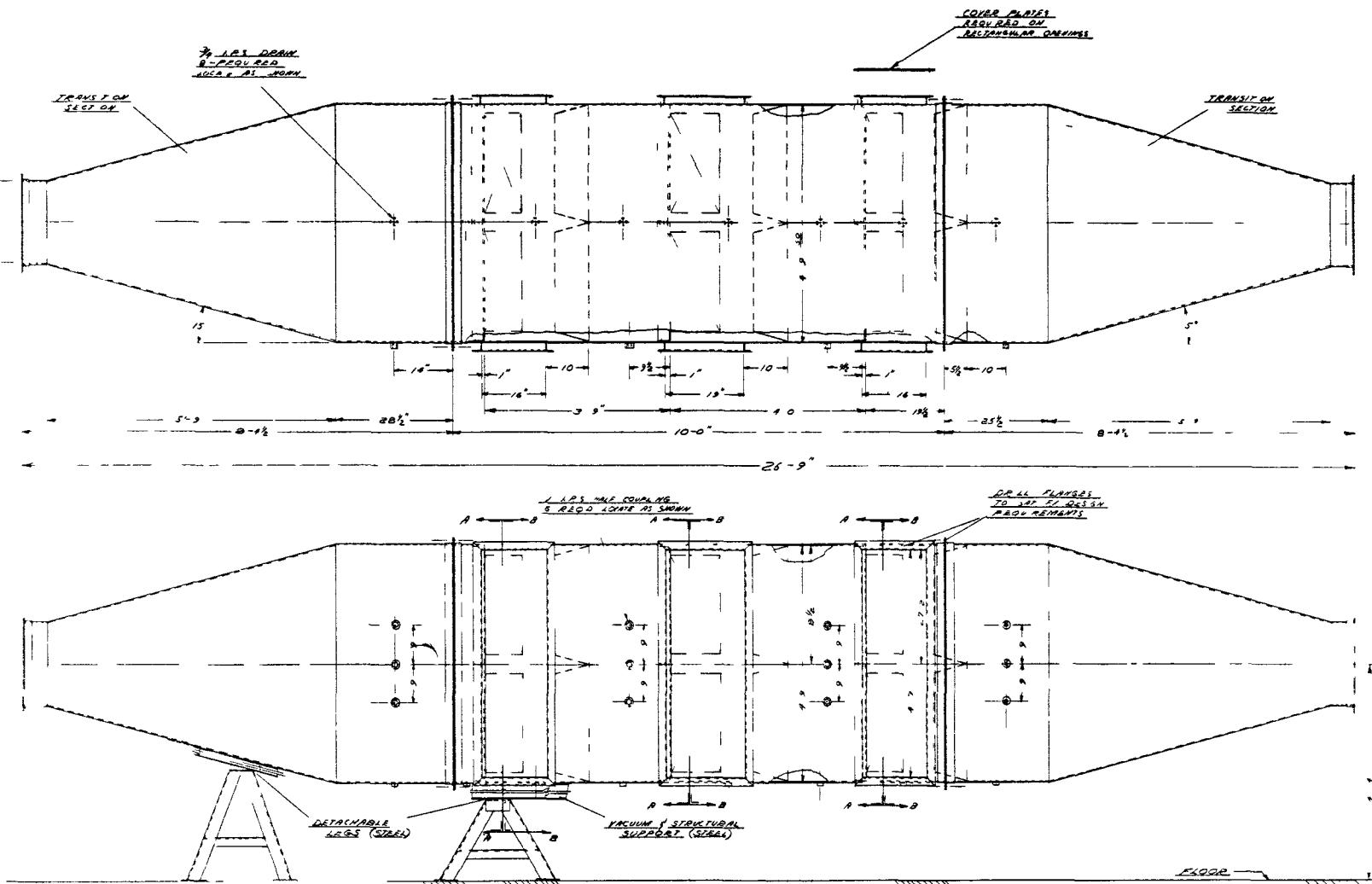


-40-

HW-65587  
ALL-54

REFERENCE DWG  
EQUIPMENT LAYOUT PLAN D-62249-1

Figure 2A  
VIEW "A - A," EQUIPMENT LAYOUT



COM MATERIALS OF CONSTRUCTION

ALL MATERIAL WILL BE TYPE 304 STAINLESS STEEL WITH  
EXCEPTION OF SUPPORTING LEGS, NUTS & BOLTS USED  
IN FLANGE. NO OUTSIDE SUPPORTS NEEDED TO MEET THE  
VACUUM REQUIREMENTS.

DESIGN SPECIFICATIONS

SHELL THICKNESS	0.62
DOOR TEMP	160°F
OPR VACUUM	10" OF WATER (0.26 ATA)

REFERENCE DRAWINGS

SECTION P.P. / DETAIL	D 52212 3
SECTION B.B.	D 52219 4

Figure 3A  
FILTER TEST CHAMBER

HW-65587  
ALI-54

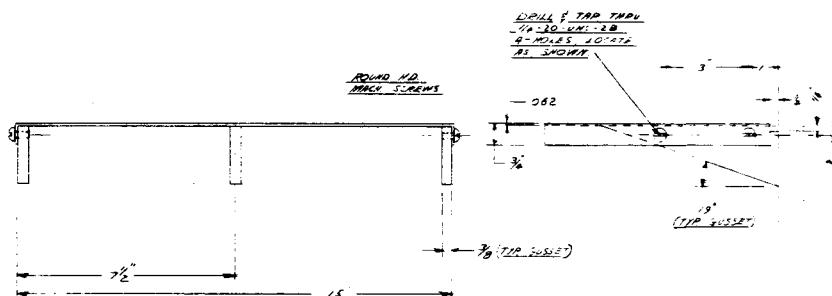
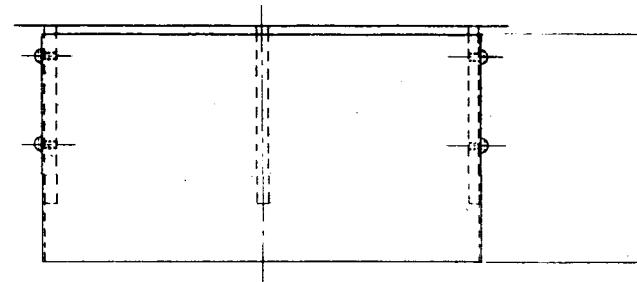
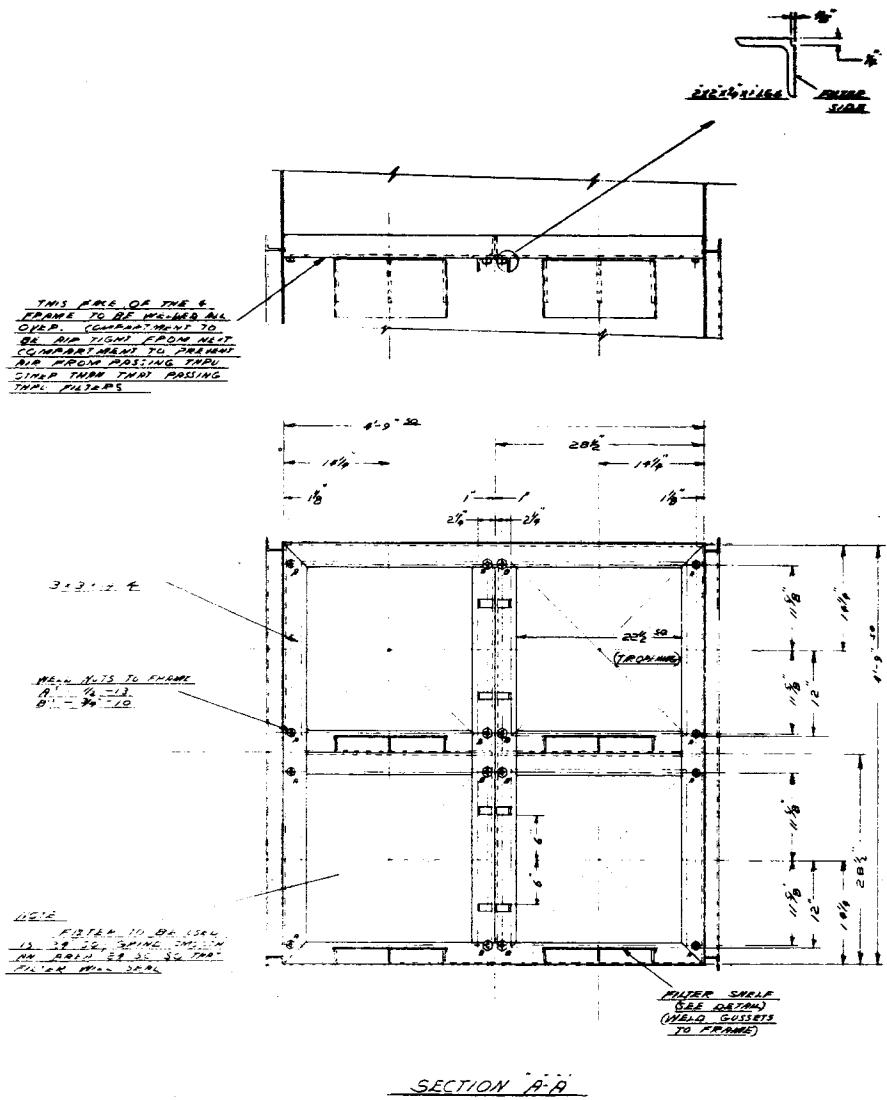


Figure 4A

## SECTION "A - A" AND DETAILS, FILTER TEST CHAMBER

FACE WALLS IN SLOPES 15°  
AS PER ASSY TO SHELL 1 IN 5

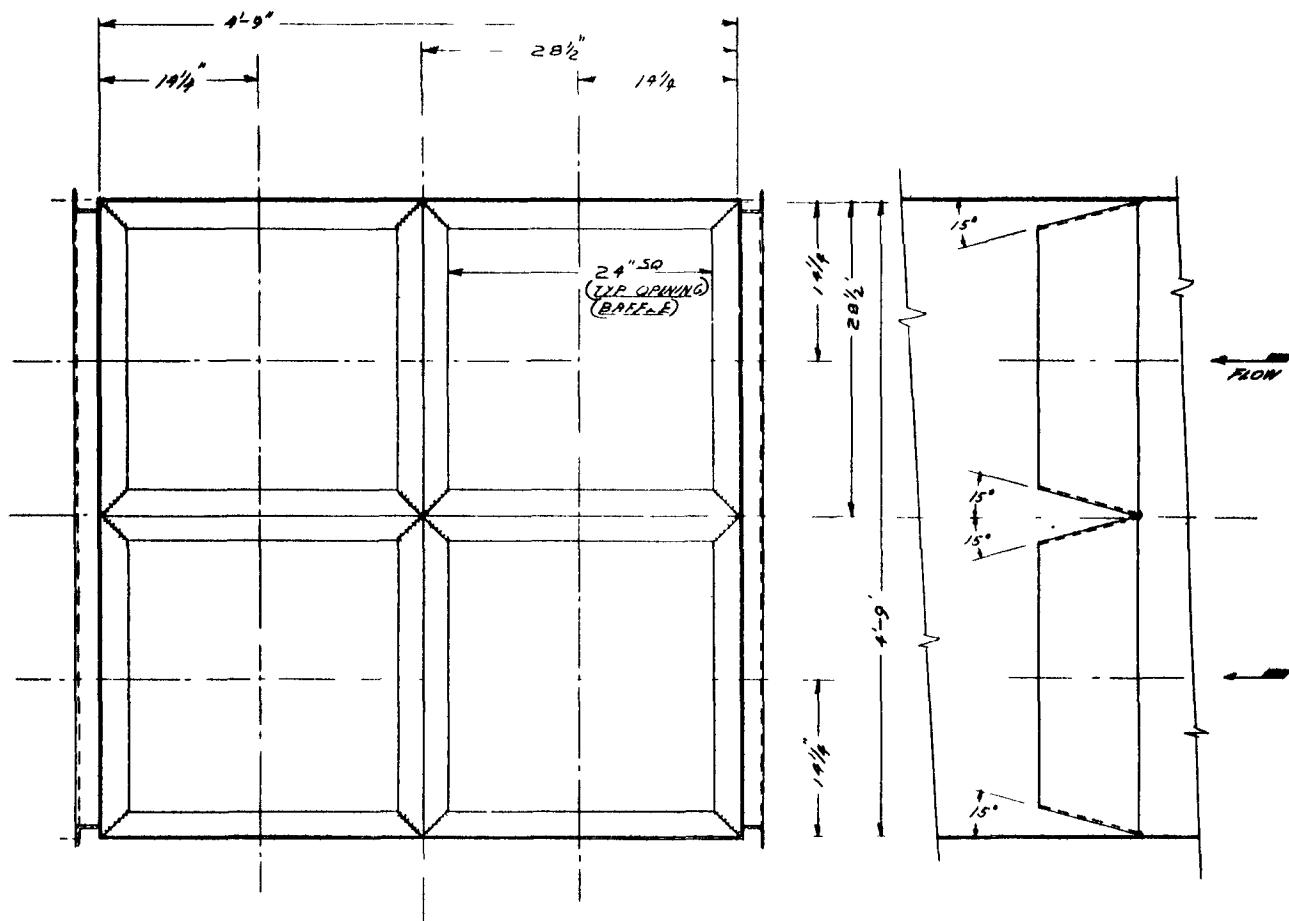
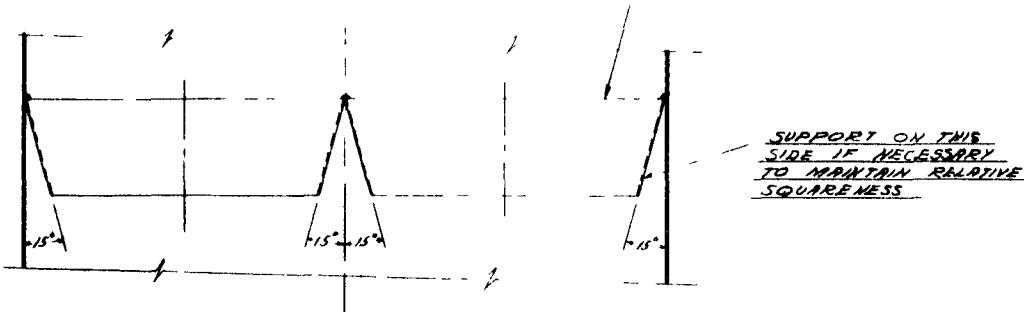
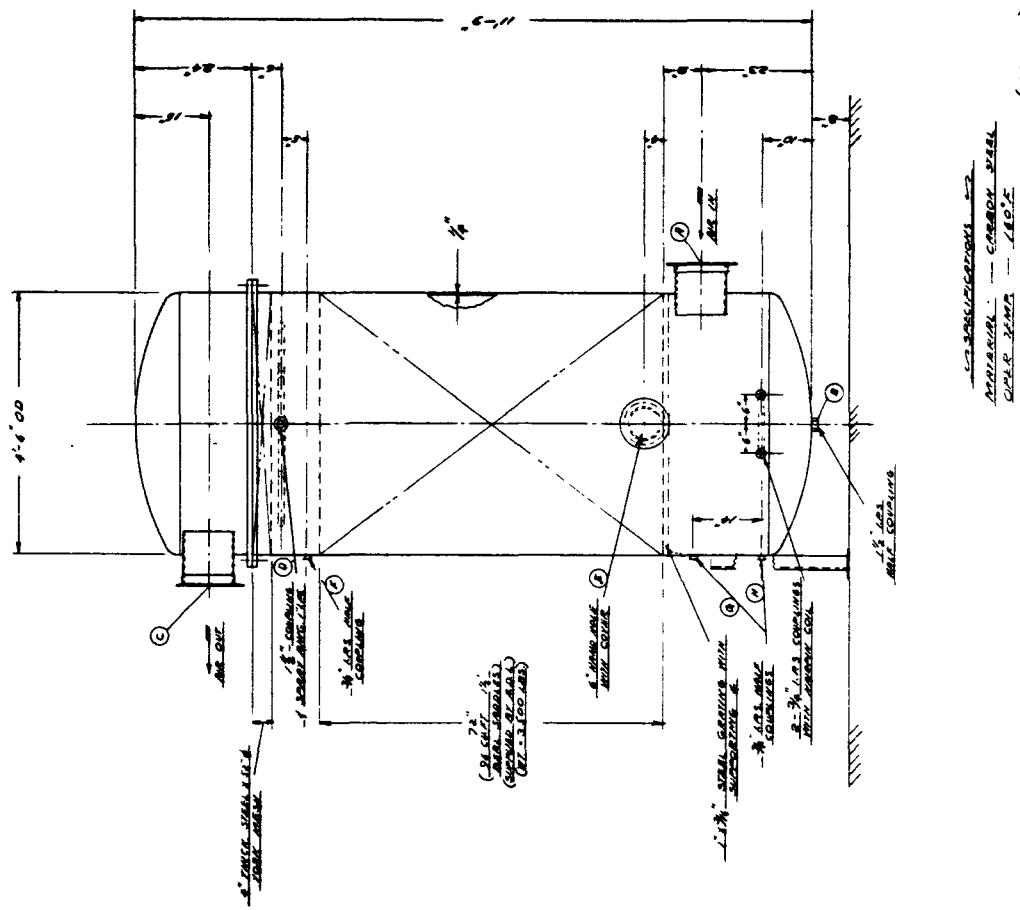
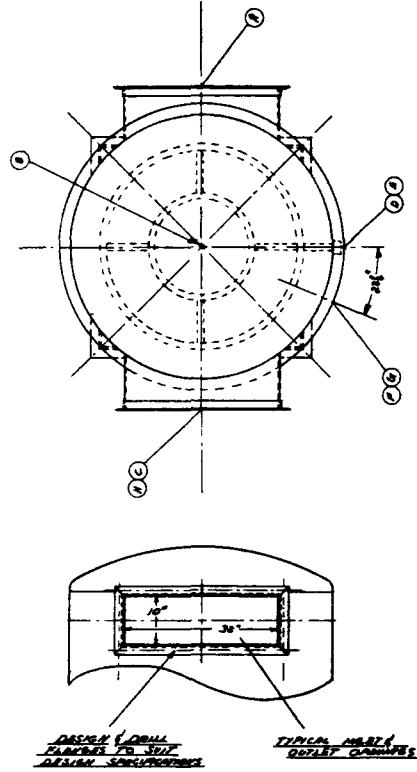


Figure 5A

SECTION "B - B" FILTER TEST CHAMBER



**Figure 6A**

HW-65587  
ALI-54

ITEM NO.	QTY	DESCRIPTION	REFERENCE
1	1	COMPRESSION BRACKET	D-6224P-13
2	1	EXTENSION ROD 3/8"	D-6224P-14 REF-8
3	2	ANT. 3/8" 10	
4	2	ANT. 45° 10	
5	2	PLAIN WASHER 1/4"	
6	1	EXTENSION ROD 1/4" (REF-13)	D-6224P-13
7	1	COMPRESSION SPRING (REF-14)	D-6224P-14 REF-15
8			

ITEM NO.	QTY	DESCRIPTION	REFERENCE
1	1	COMPRESSION BRACKET	D-6224P-13
2	1	EXTENSION ROD 3/8"	D-6224P-14 REF-8
3	2	ANT. 3/8" 10	
4	2	ANT. 45° 10	
5	2	PLAIN WASHER 1/4"	
6	1	EXTENSION ROD 1/4" (REF-13)	D-6224P-13
7	1	EXTENSION SPRING (REF-14)	D-6224P-14 REF-15
8	1	EXTENSION ROD 1/4"	D-6224P-14 REF-15
9	1	EXTENSION ROD 1/4"	D-6224P-14 REF-15
10	1	EXTENSION ROD 3/8"	D-6224P-14 REF-15
11			

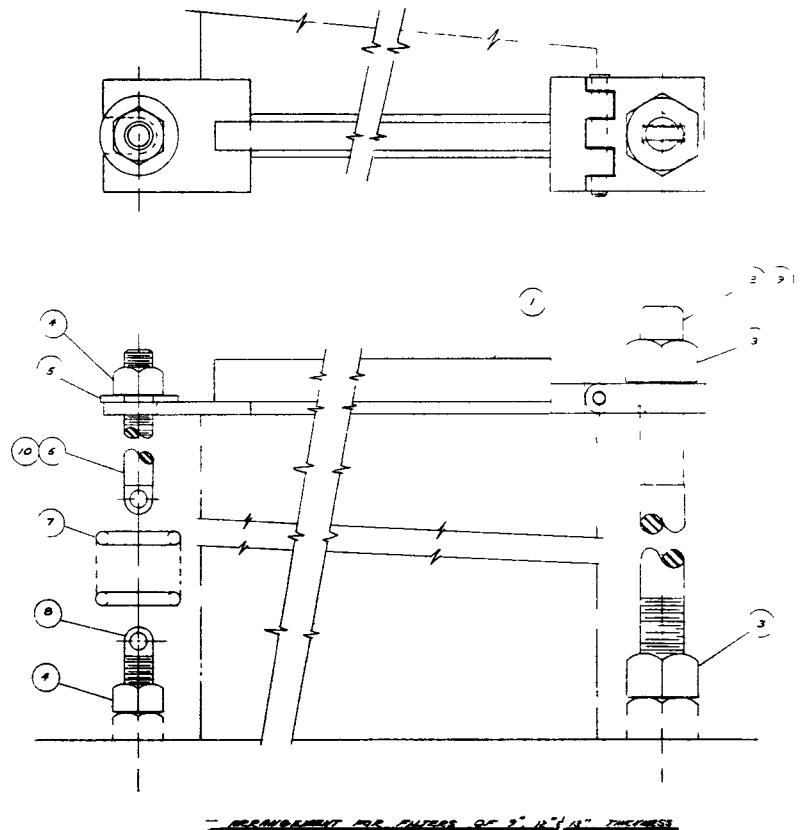
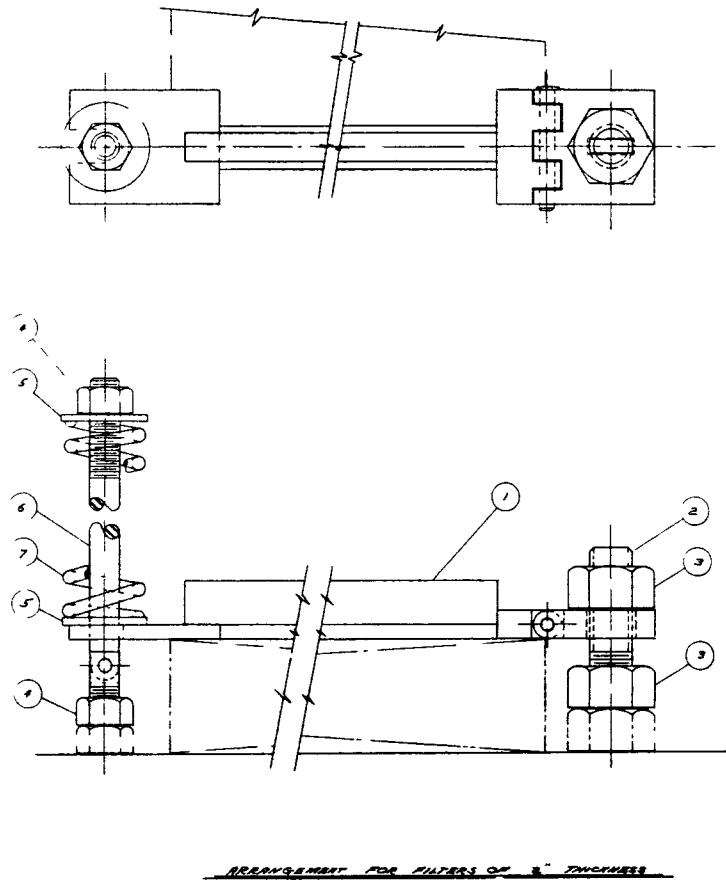


Figure 7A  
FILTER COMPRESSION BRACKETS, ASSEMBLY

HW-65587  
ALI-54

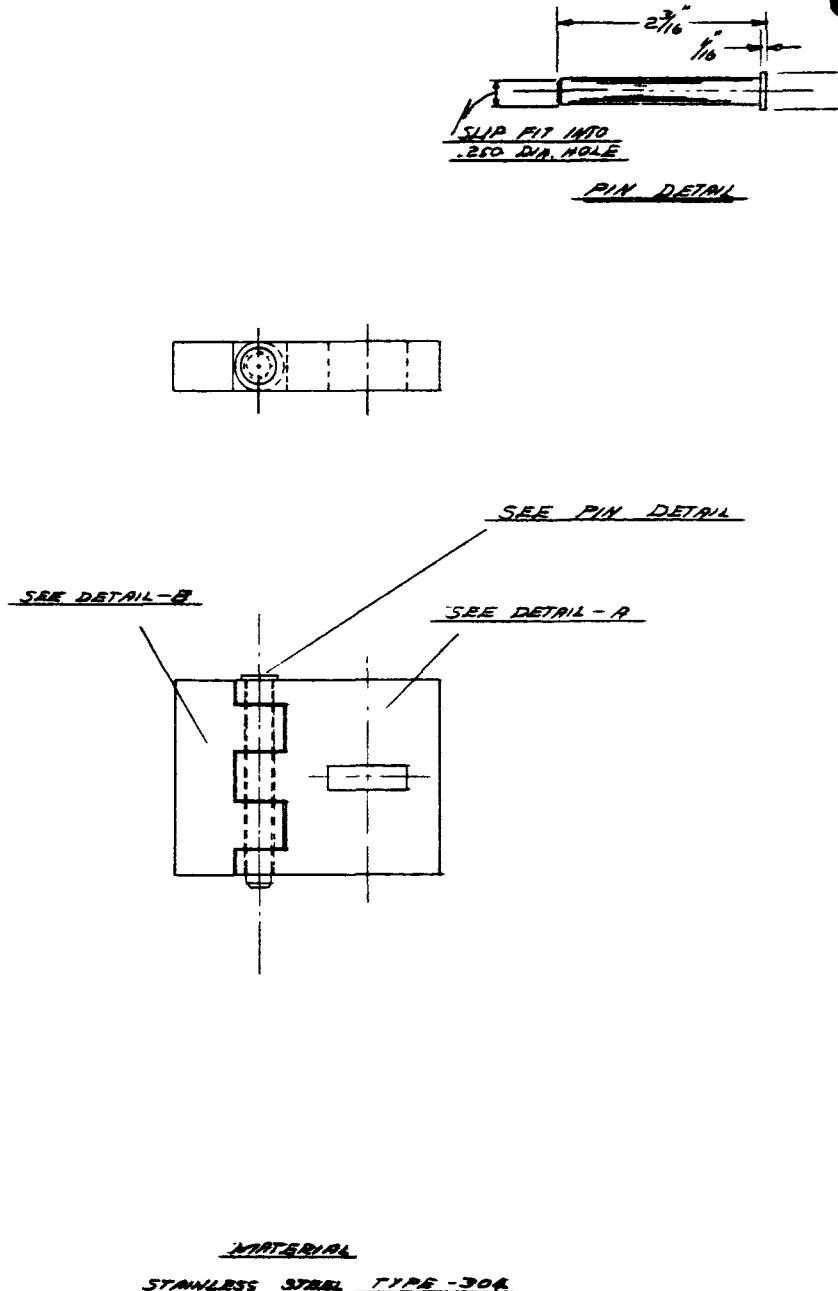
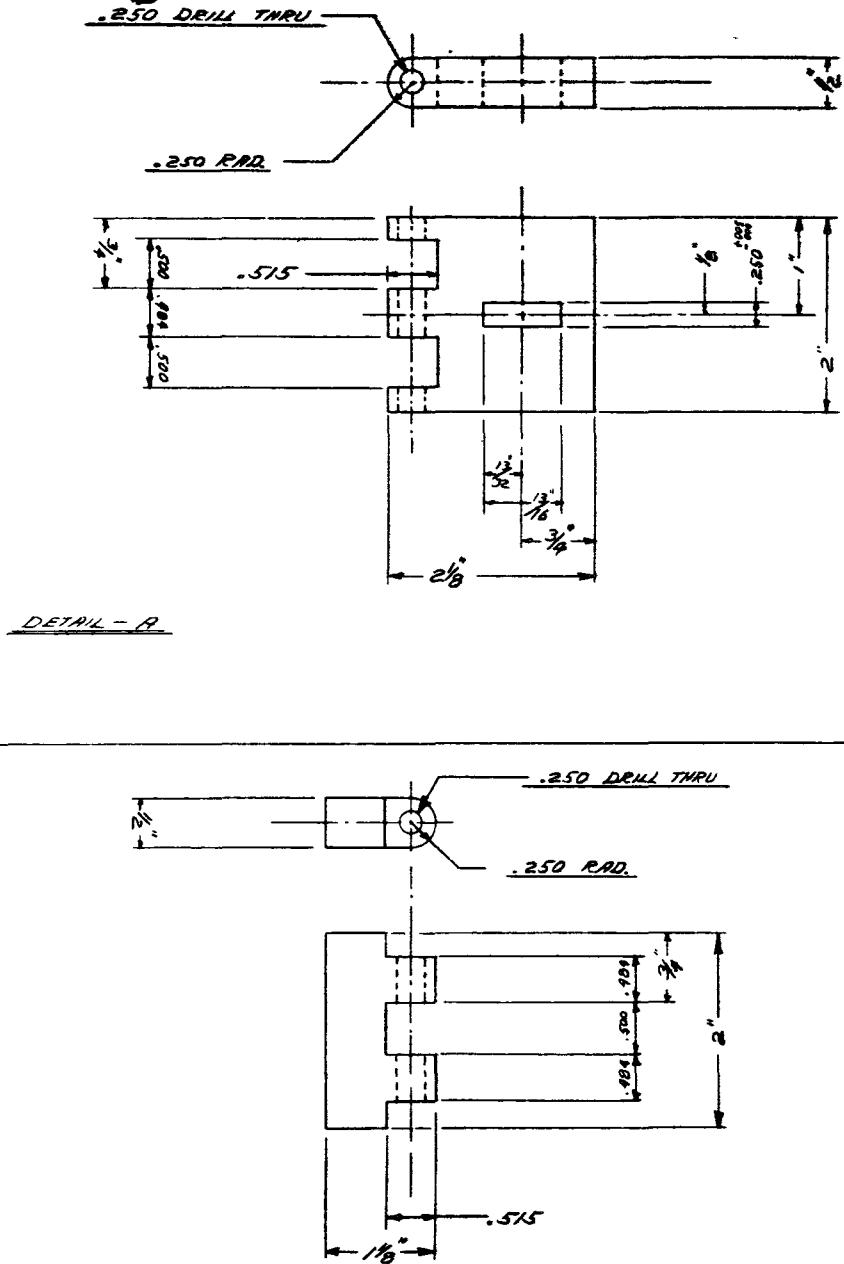
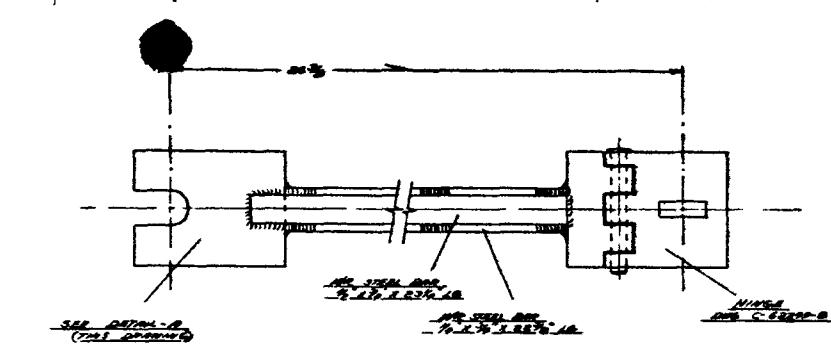
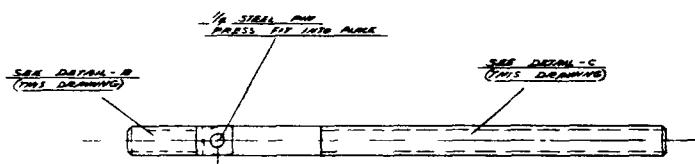
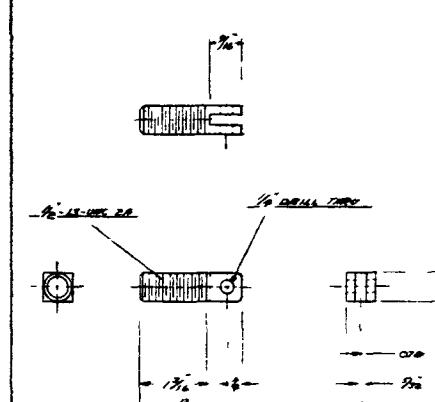
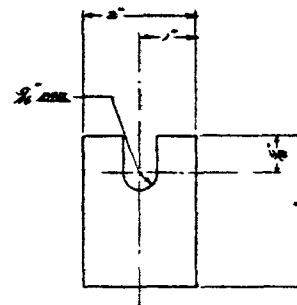


Figure 8A

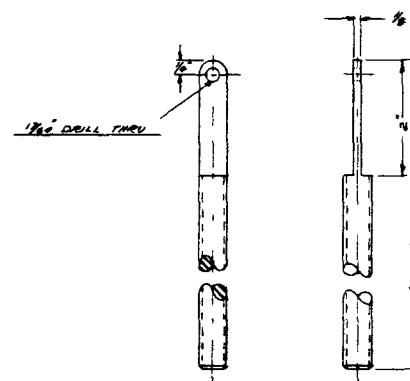


NOTE  
ARC WELD AS SHOWN

COMPRESSION BRACKET ASSEMBLY



EXTENSION ROD ASSEMBLY



COMPRESSION ROD ASSEMBLY  
D-62202 S

**Figure 9A**

**FILTER COMPRESSION BRACKETS, DETAILS AND SUBASSEMBLY**

HW-65587  
ALI-54

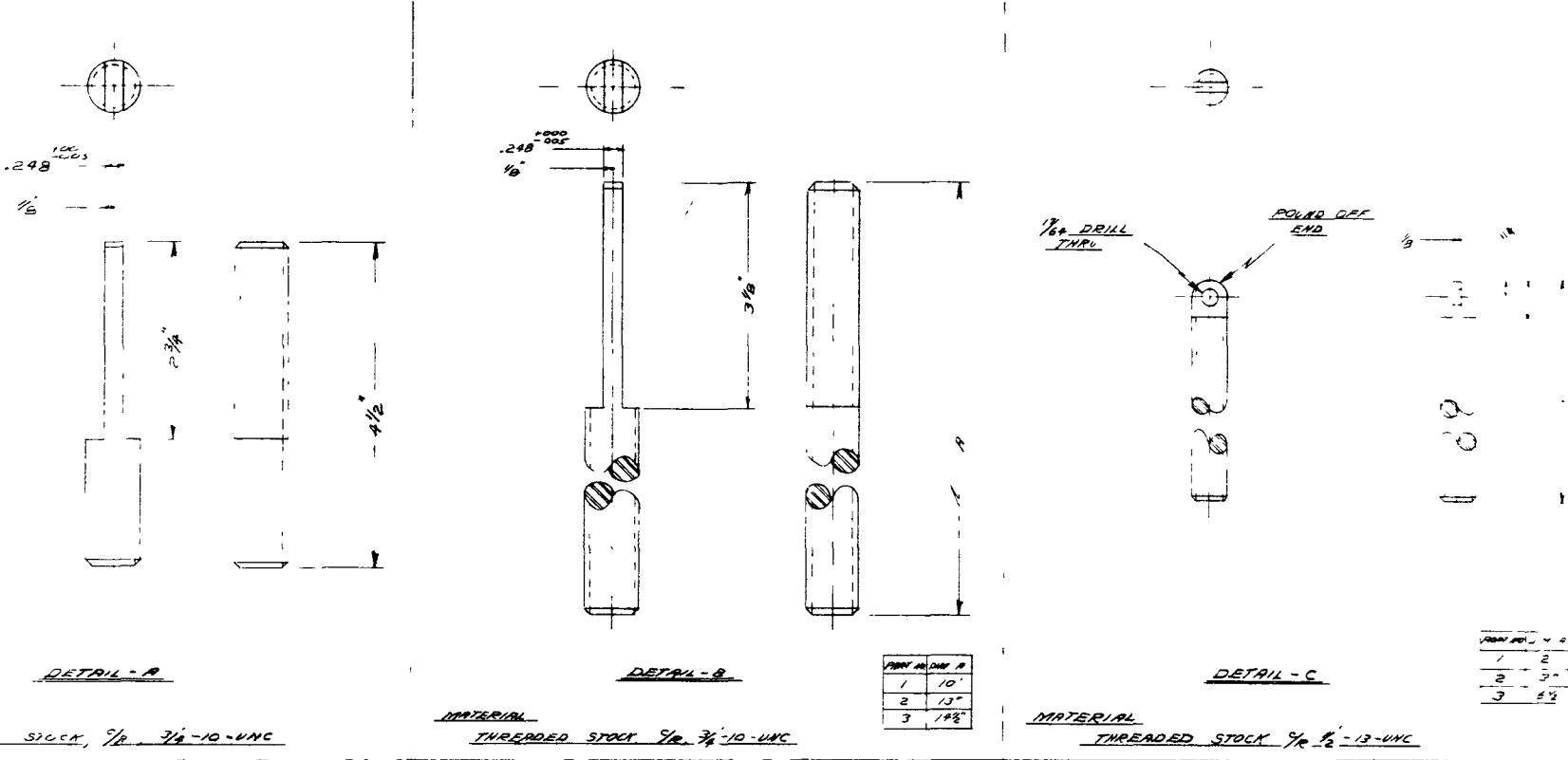


Figure 10A

**FILTER COMPRESSION BRACKETS, DETAILS**

CONFIDENTIAL 32

GENERAL NOTES

1 - FIELD CONSTRUCTION (gas plant)  
2 - GASKET MATERIAL GASKET 10,200

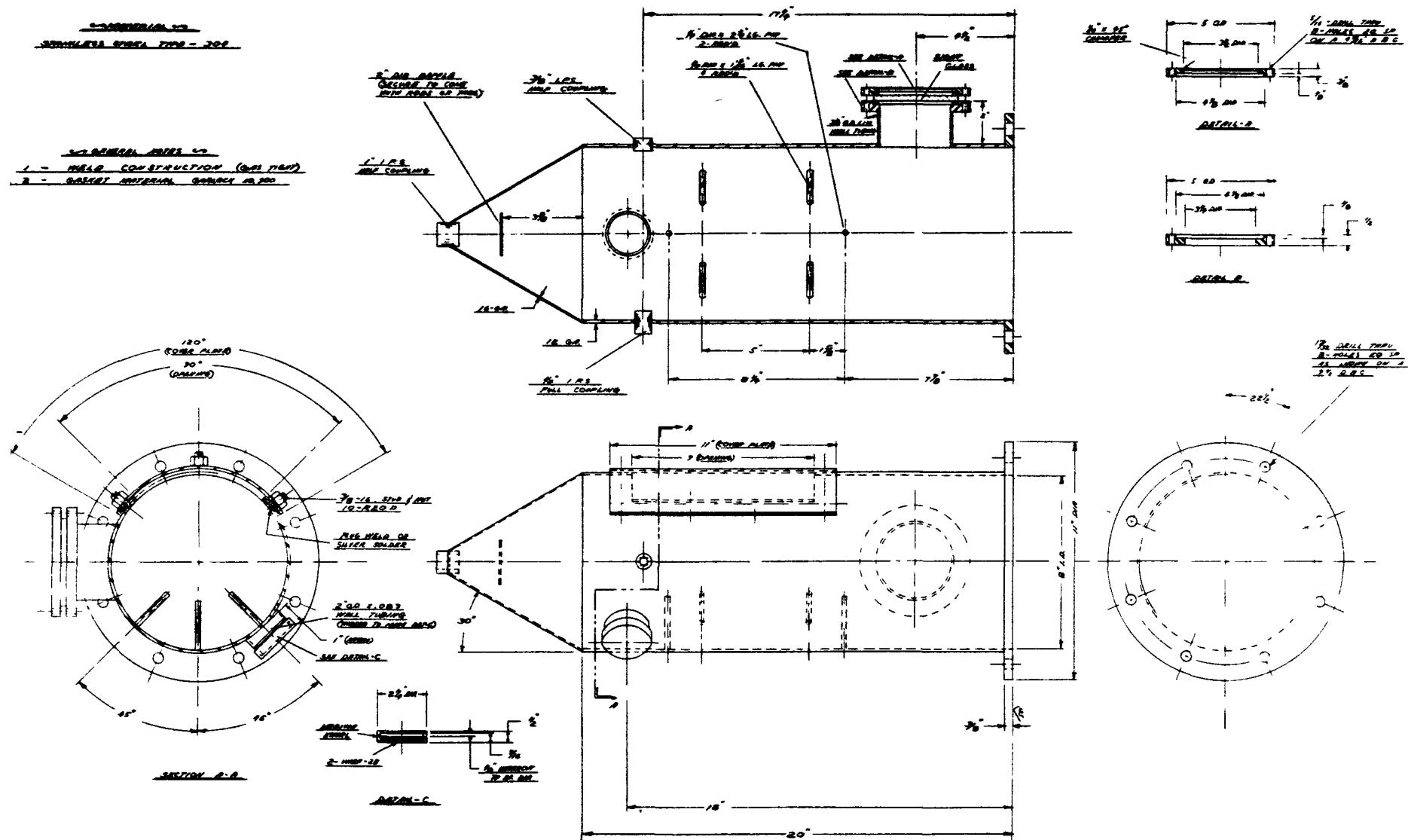


Figure 11A  
**BURNER HOUSING**

HW-65587  
ALI-54

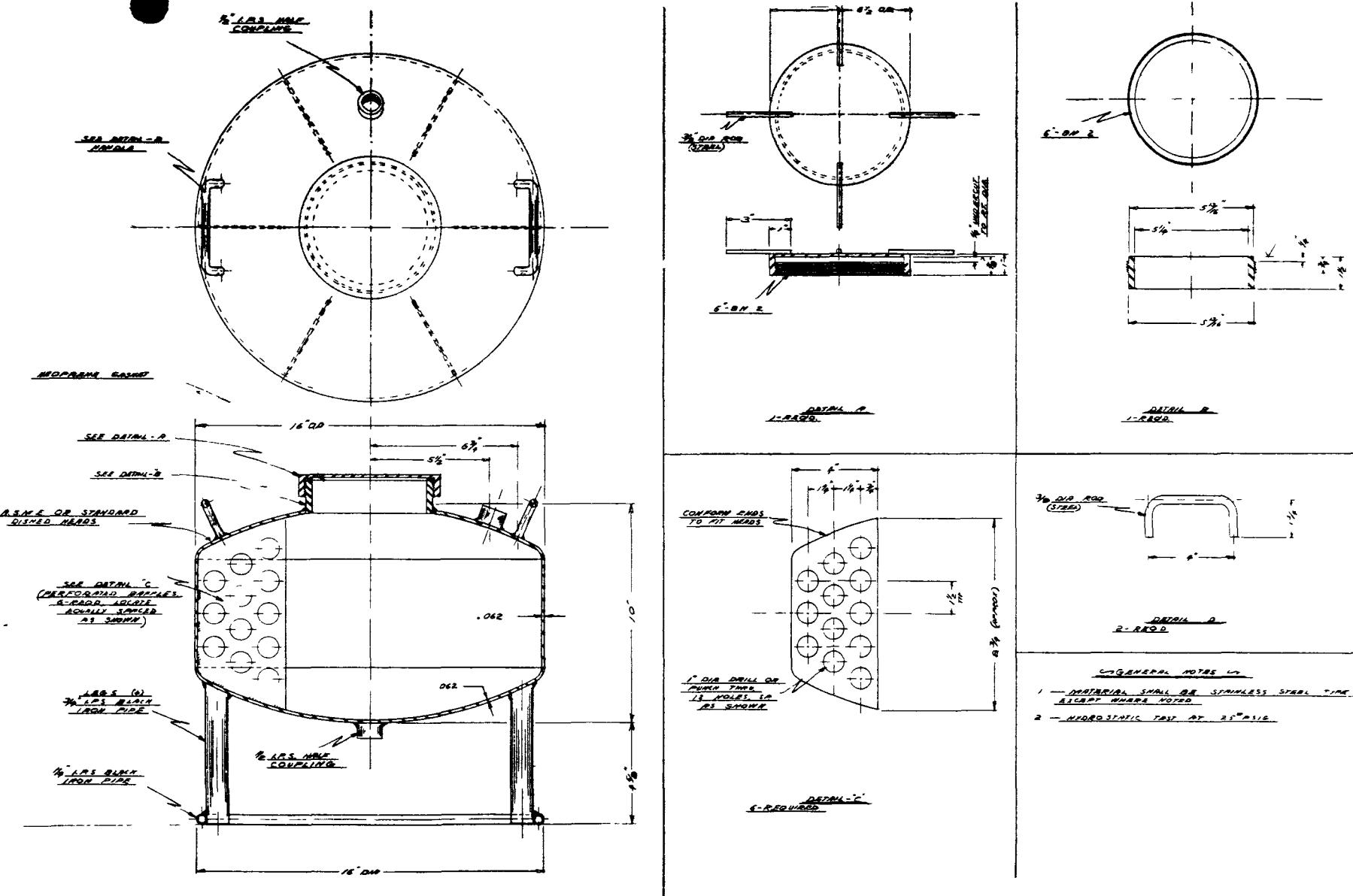


Figure 12A

ALCOHOL - IODINE MIXING TANK

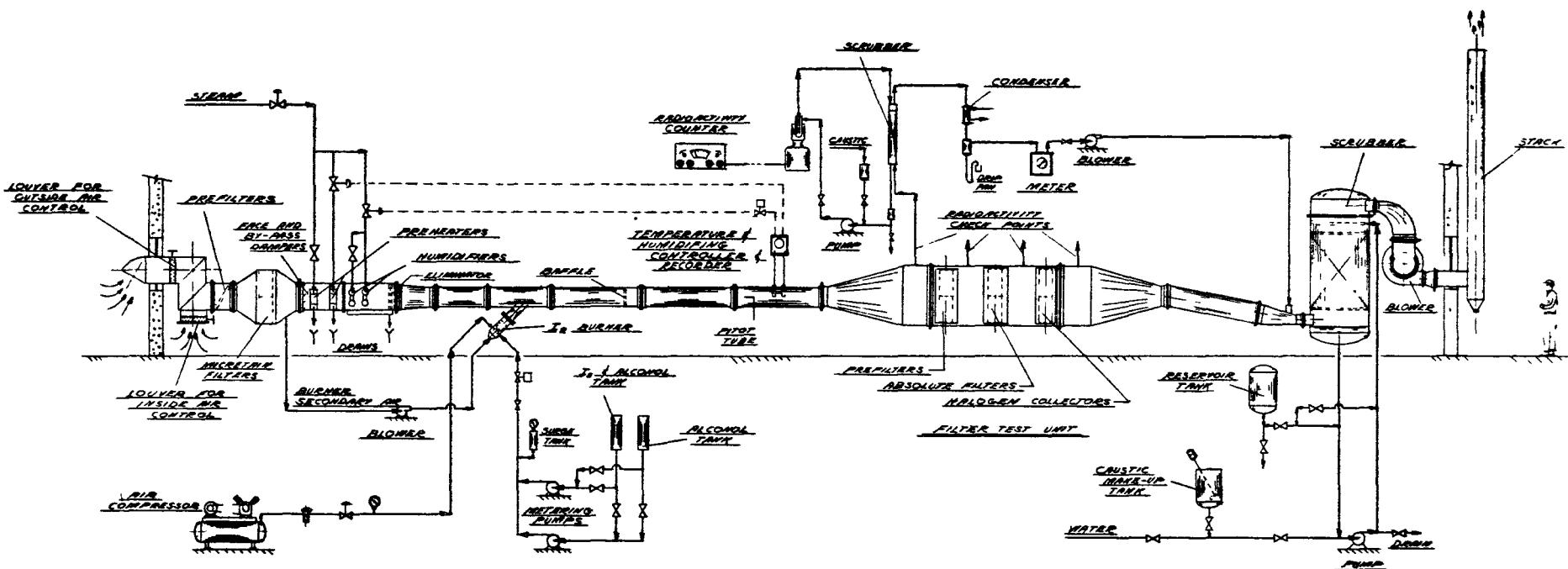


Figure 13A

HW-65587  
ALI-54

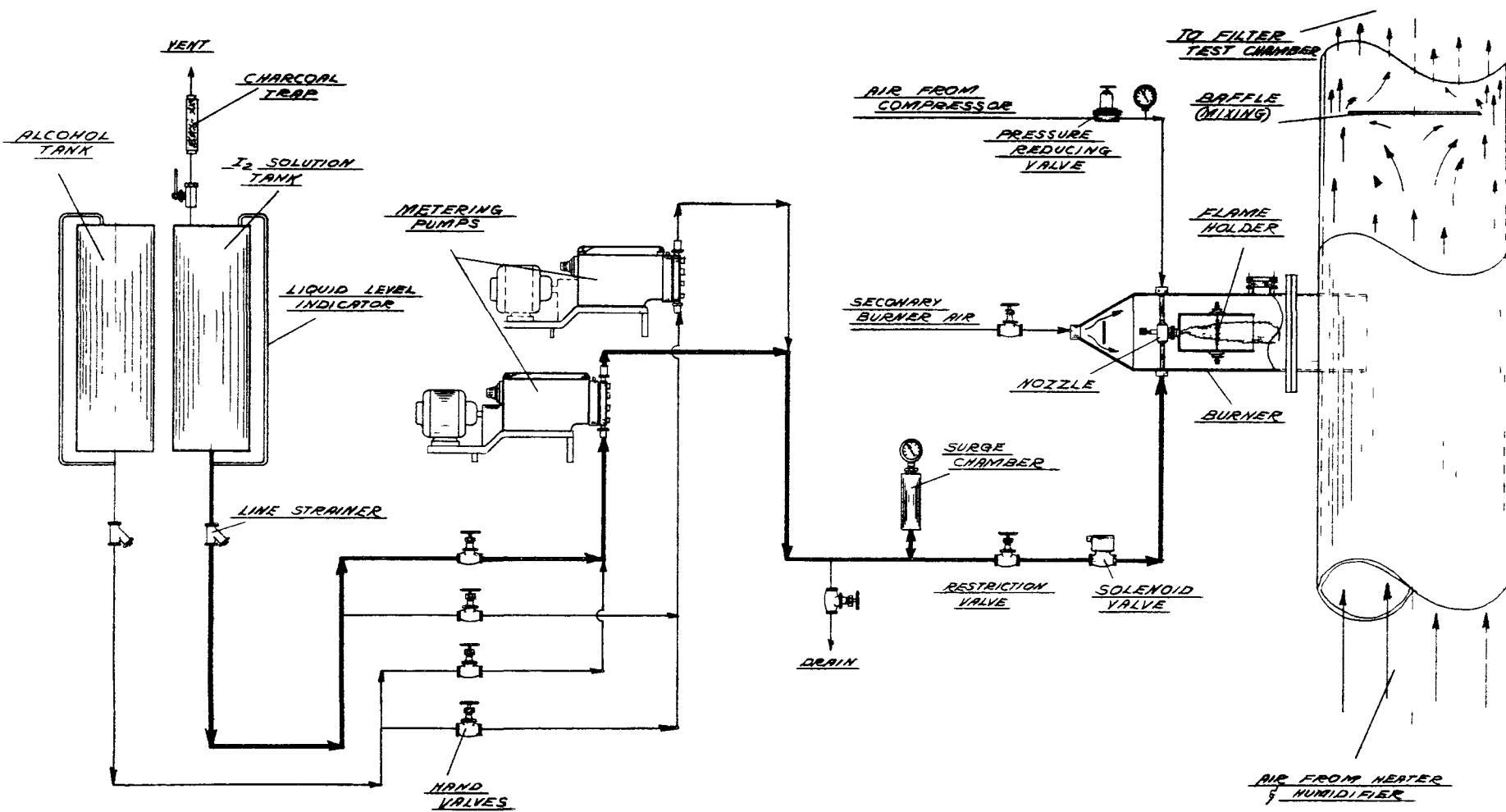


Figure 14A

SCHEMATIC FLOW SHEET, IODINE INJECTION SYSTEM

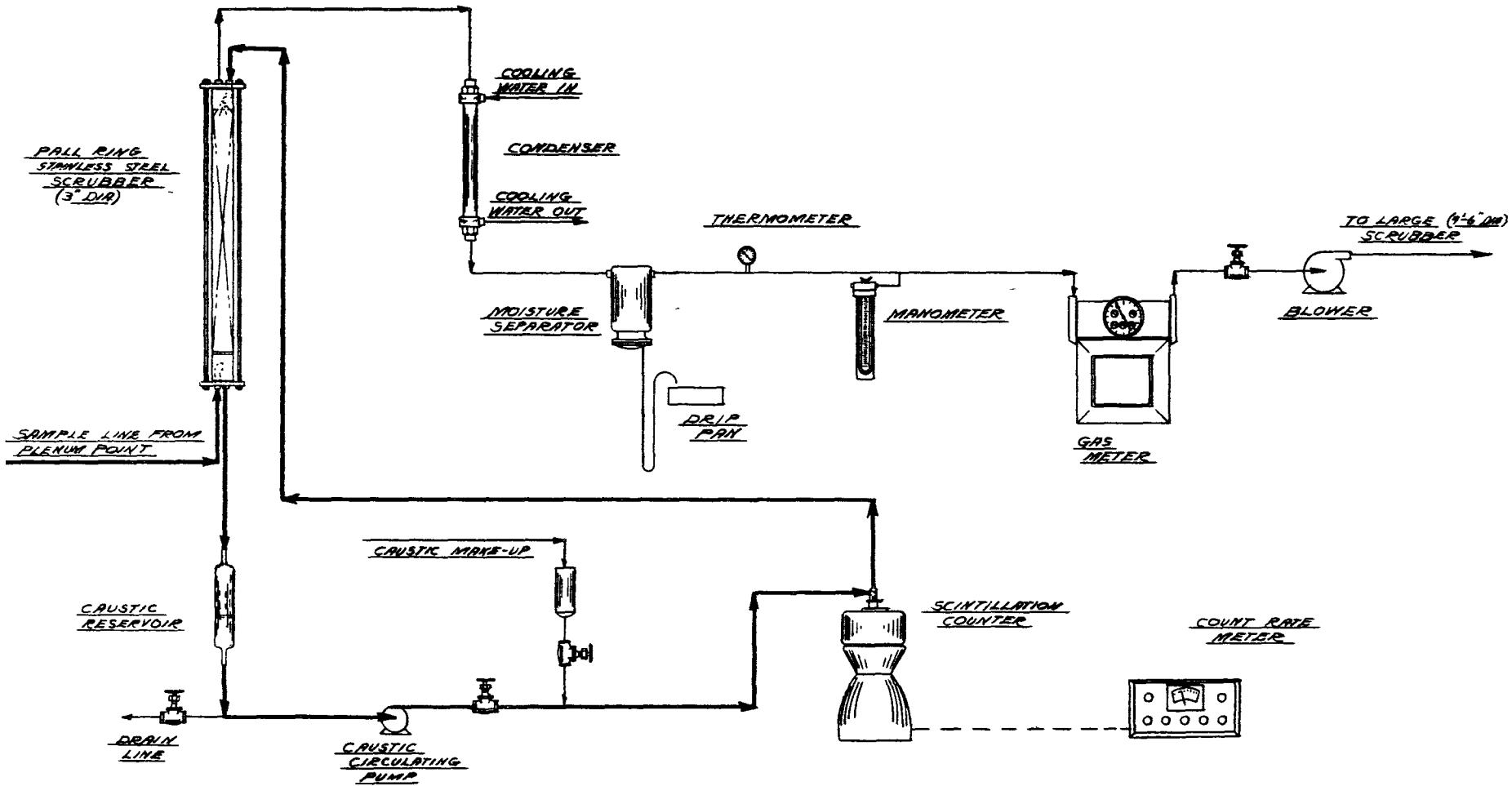
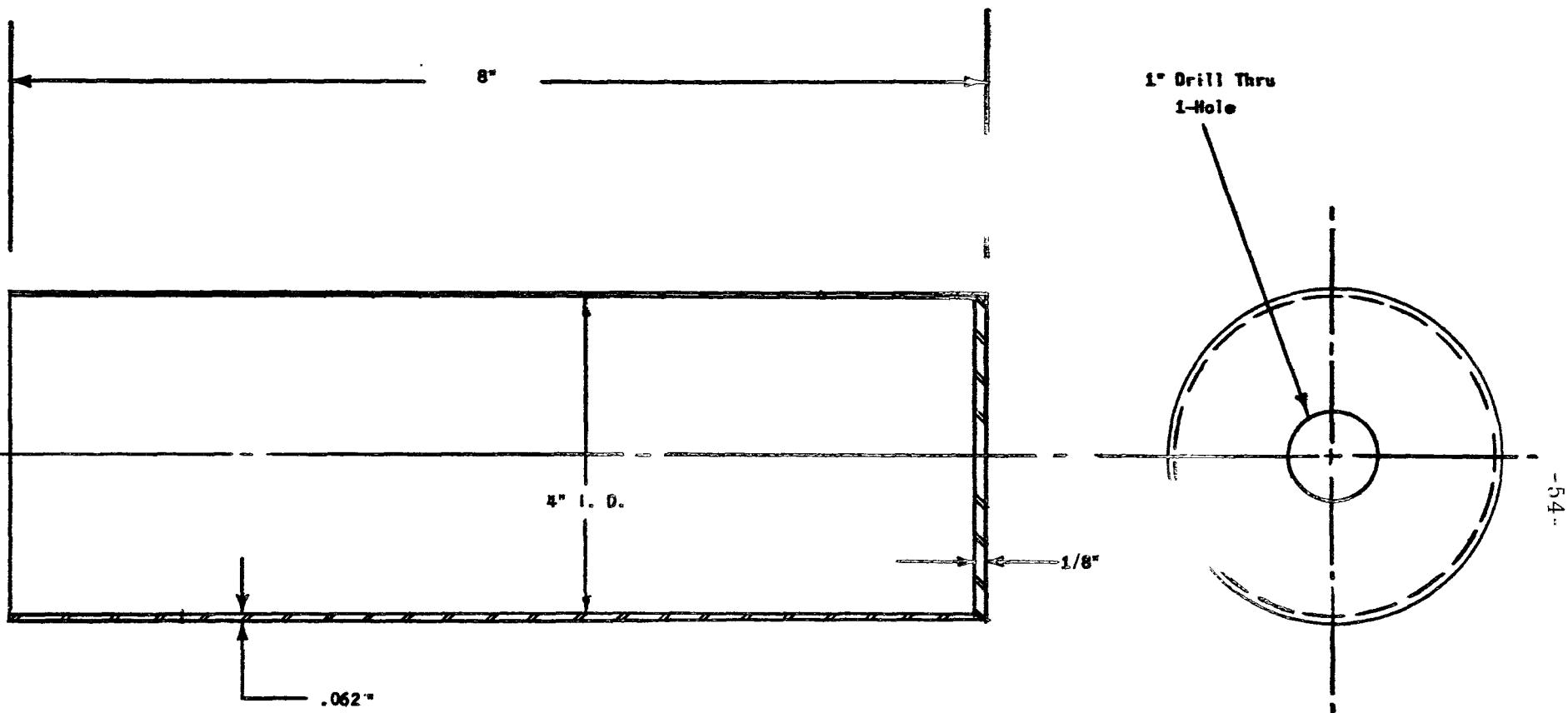


Figure 15A  
SCHEMATIC FLOW SHEET, IODINE DETECTION SYSTEM

HW-655587  
ALI-54



Material: Stainless Steel, Type 317

Quantity: 2

Figure 16A  
BURNER SHIELD

NOTE

1. - Weld Construction.
2. - Longitudinal Seam  
to be Ground Smooth  
Inside and Out.

HW-65587  
ALI-54

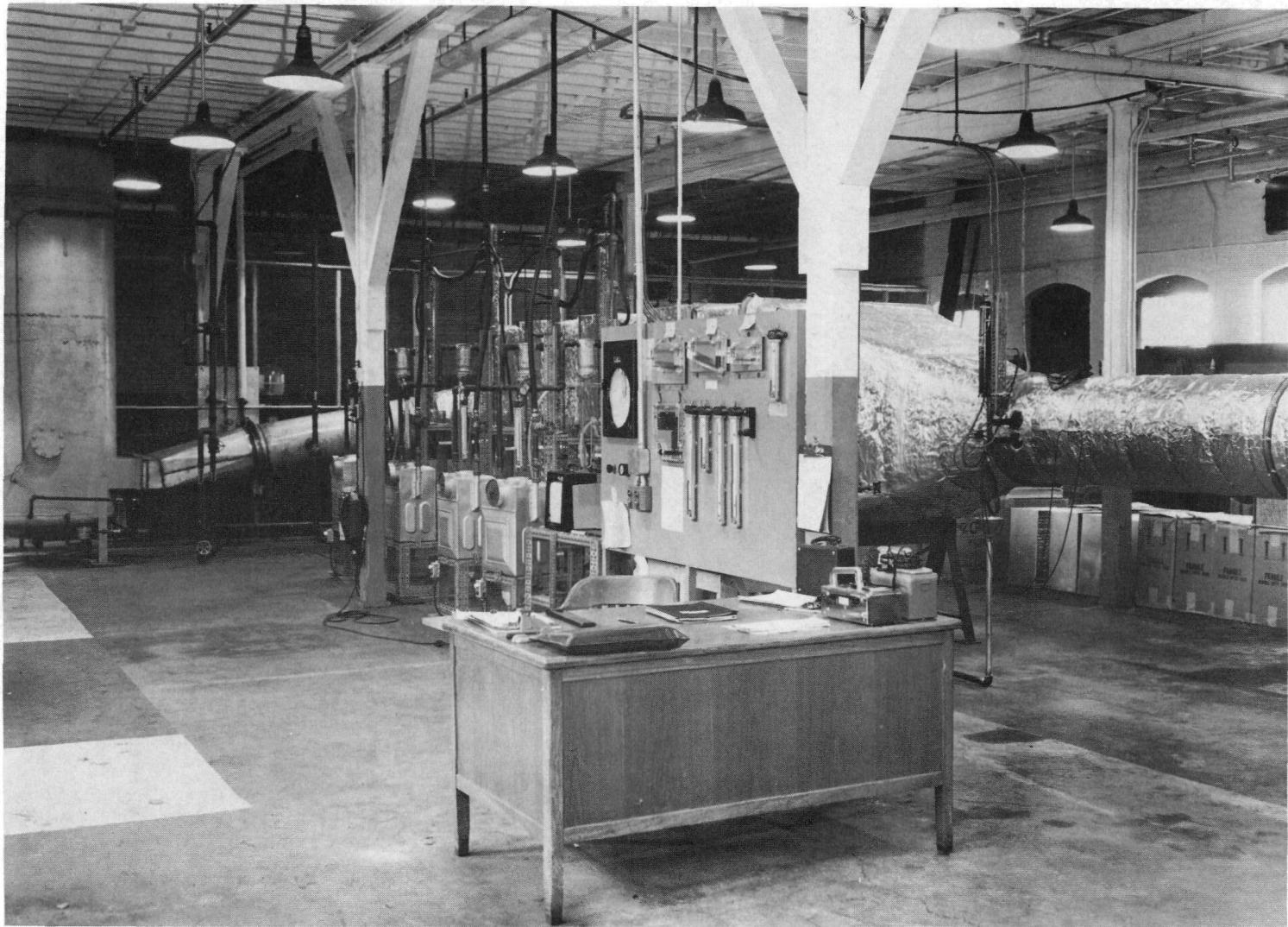


FIGURE 17A  
CONTROL PANEL AND TEST SECTION

-55-

HW-65587  
ALI-54

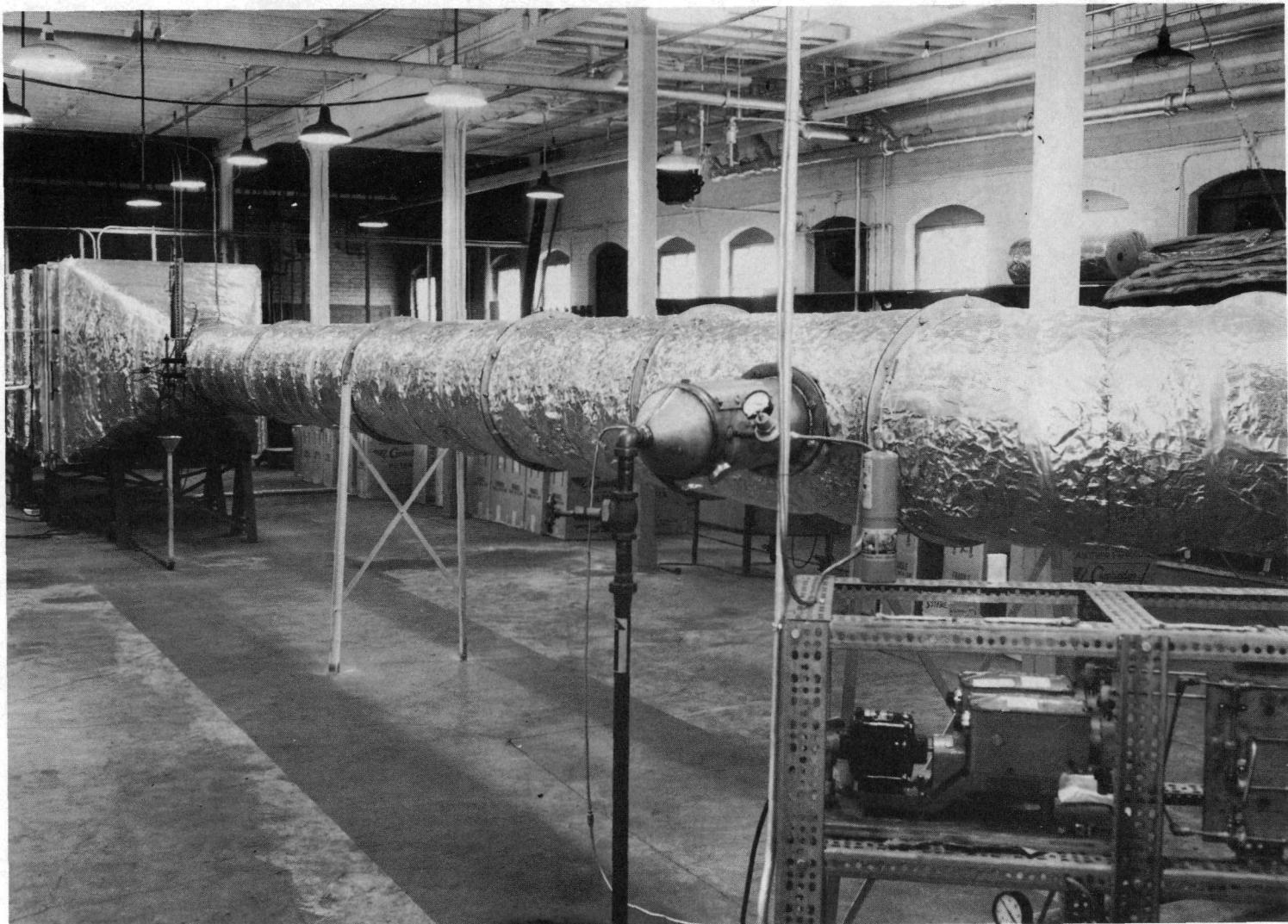


FIGURE 18A  
IODINE INJECTION SYSTEM AND BURNER

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HW-65587  
ALI-54



FIGURE 19A  
ANALYTICAL SCRUBBER SYSTEM

-57-

HW-65587  
ALI-54

APPENDIX B

COUNT-RATE CALCULATIONS

The following calculation was used to determine a relative count rate per cubic foot of air. Filter efficiencies are based on these relative count rates as shown.

TYPICAL ANALYTICAL SCRUBBER DATA

Final Gas -Meter Reading	8744.3 ft <sup>3</sup>
Initial Gas -Meter Reading	8352.3 ft <sup>3</sup>
Temperature Start*	66°F
Temperature Finish*	70°F
Pressure Start*	-4.6" H <sub>2</sub> O
Pressure Finish*	-4.4" H <sub>2</sub> O
Initial Scrubber Solution Level	590 ml
Final Scrubber Solution Level	1425 ml
Initial Count Rate	1716 cpm
Final Count Rate	17055 cpm
Initial Background	1482 cpm
Final Background	1560 cpm

1. The corrected air volume is first calculated by correction to 60°F, atmospheric pressure, and moisture-free air.  
Atmospheric pressure at time of test = 411.4" H<sub>2</sub>O  
 $8744.3 - 8352.3 = 392.0 \text{ ft}^3$  uncorrected air volume  
The corrected air volume is calculated by use of the equation

$$\text{Uncor Air Vol} \times \frac{\text{Atm Press ("H}_2\text{O)} - \text{Avg Press ("H}_2\text{O)} - \text{Vap Press of H}_2\text{O ("H}_2\text{O)}}{\text{Atm Press ("H}_2\text{O)}} \times \frac{520}{\text{Avg Temp (°F)} + 460}$$

\*Readings at entrance to gas meter.

$$\text{Therefore, } 392.0 \times \frac{411.4 - 4.5 - 5.8}{411.4} \times \frac{520}{68 + 460} = 376.5 \text{ ft}^3$$

2. The increase in count rate during the test is determined for a scrubber station as follows:

The initial count rate reading less the background multiplied by a volume change factor gives a corrected initial count rate; therefore,

$$(1716 - 1482) \frac{736}{1571} = 110 \text{ cpm}$$

The volume change factor corrects for the difference in scrubbing-solution volumes before and after a test. These volumes were calculated as

$$\text{Initial } 590 + 146 = 736 \text{ ml}$$

$$\text{Final } 1425 + 146 = 1571 \text{ ml}$$

The 146 ml is the amount of solution held up in the system in pipes, the pump, and the packing. This volume was checked weekly for each scrubber.

The final count rate is then the final count-rate reading less the background:

$$17055 - 1560 = 15495 \text{ cpm}$$

At these low count rates the loss of counts due to coincidence was negligible.

The relative count rate (RCR) is then equal to

$$\frac{\text{Count Rate Increase (cpm)}}{\text{Corrected Air Volume (ft}^3\text{)}} \times \frac{\text{Final Scrubber Solution Volume (ml)}}{\text{Scrubber Factor}*}$$

$$\text{Therefore, } \frac{15386}{376.5} \times \frac{1571}{0.986} = 65100 \text{ cpm}$$

The percentage efficiency of a particular filter is therefore

$$\frac{\text{RCR upstream} - \text{RCR downstream}}{\text{RCR upstream}} \times 100$$

\*See Section I, "Significance and Determination of Scrubber Factors," in Chapter II.

APPENDIX C

PROOF THAT COMBUSTION OF TINCTURE OF IODINE  
RESULTS ONLY IN FREE IODINE

The purpose of this experiment was to prove that the combustion of the tincture of iodine solution results in the liberation of free iodine rather than complex iodine compounds. This was determined by the analytical procedure outlined below.

A. TEST RUN

Starting materials for the combustion test were: 4 gallons (15.15 1) N/10 thiosulfate solution buffered with sodium bicarbonate to a pH of 7.5 - 8. (16 gr). Iodine tincture (20.1136 gr of  $I_2$  in 1200 ml of alcohol).

During the run, 0.69 gallon of scrubbing liquid was lost by evaporation.

B. ANALYTICAL RESULTS

Two samples were received for assaying:

Sample A - original scrubbing liquid

Sample B - concentrated scrubbing liquid containing  
 $Na_2S_2O_3$ ,  $NaHCO_3$ ,  $NaI$ ,  $Na_2S_4O_6$

Presuming that the original scrubbing liquid was exactly N/10, we supposed that we had a fictional  $A_2$  sample on hand to run with an unknown but calculable factor, and by difference we were able to determine the consumed iodine:

$$A = 24.82 \text{ gr thio}/1000 \text{ ml H}_2\text{O} \quad \text{N/10 thio: } F = 1.000$$

Loss of  $H_2O$ /1000 ml during run = 172.3 ml

$$A_2 = 24.82 \text{ gr thio}/827.7 \text{ ml H}_2\text{O} \quad \text{N/10 thio: } f = 1.2082$$

(10,000: 8277 = 1.2082)

20 ml original thio (A) = 20 ml N/10 (F 1.000) thio

20 ml supposed thio (A<sub>2</sub>) = 24.16 ml N/10 (F 1.000) thio  
(20 x 1.2082)

Since titrating A requires 18.2 ml N/10 I<sub>2</sub> (f 1.1), titrating A<sub>2</sub> (24.16 ml) requires 22.0 ml N/10 I<sub>2</sub> (f 1.1).

To titrate solution B (= nonexisting A<sub>2</sub> + unknown amount of I<sub>2</sub>), 19.3 ml N/10 I<sub>2</sub> (f 1.1) was used.

The difference was (22.0 - 19.3) 2.7 ml N/10 I<sub>2</sub>

2.7 ml I<sub>2</sub> (f 1.1) = 2.97 ml N/10 I<sub>2</sub> (f 1.00)

Since 24.2 ml of sample B contained 2.97 ml N/10 (f 1.1) I<sub>2</sub> solution, 1000 ml contained 122.7 ml N/10 (f 1.1) I<sub>2</sub> solution.

1000 ml of N/10 (f = 1) I<sub>2</sub> solution contained 12.692 gr I<sub>2</sub>; thus 122.7 N/10 (f = 1) I<sub>2</sub> solution contained 1.56 gr I<sub>2</sub>.

At the end of the run, 12.541 of scrubbing liquid remained on hand, so this should have contained 19.56 gr I<sub>2</sub>, whereas 20.1136 gr were introduced. The loss during the combustion test was 0.55 gr, or 2.73%.

This loss is explained by evaporation, corrosion (iodine combining with parts of the equipment), and adherence to the tincture container, tubes, etc.

## APPENDIX D

### SPECIFICATION FOR HALOGEN COLLECTOR TEST PROGRAM\*

#### 1. 0 OBJECT

This specification describes the scope of the test program, required test facilities, test procedures, and desired test data for the Halogen Collector Test Program.

#### 2. 0 SCOPE OF THE TEST PROGRAM

The scope of the test program shall be limited to radio-iodine tracer testing, on a pilot plant scale, of laboratory-proven methods of removing radio-iodine from air streams. These methods shall be restricted to "dry" processes. The tests shall determine, for evaluation, the iodine removal efficiencies, loading capacities, and flow engineering data of standard air filter size halogen collector units fabricated to specifications prepared from laboratory data of these processes. The test conditions will also be limited to the range of iodine concentrations, air temperatures, and relative humidities which are anticipated for the proposed application.

The tests shall be conducted at the contractor's site. The contractor shall design, fabricate, and operate the pilot plant facilities according to the test conditions outlined herein. The contractor shall also be responsible for procuring and scheduling the shipments of Iodine-131 to be used in the test program although the General Electric Company will assist in obtaining a priority should the need arise. The General Electric Company will specify the components to be tested and will procure and make available these components.

---

\* This Appendix issued under separate cover at Hanford Atomic Products Operation as HWS-6750.

### 3.0 TEST PROGRAM

#### 3.1 Iodine Removal Efficiency Tests

##### 3.1.1 General

The iodine removal efficiency tests shall consist of injecting trace concentrations of elemental iodine into an air stream which is then passed through the test section which is comprised of air prefilters and absolute filters followed by the halogen collector. Air samples shall be taken and analyzed to determine iodine removal efficiency of the various test components.

##### 3.1.2 Test Facility

The test facility shall be sized to accommodate an air flow of 4,000 SCFM and shall be equipped to provide the following test conditions:

1. Inlet supply air - filtered atmospheric air with maximum dust loading of 0.005 mg per cubic meter.
2. Inlet air temperature<sup>(1)</sup> - 70 and 160 F.
3. Inlet air humidity<sup>(1)</sup> - 70 and 100 per cent relative humidity.
4. Iodine concentration - 0.1, 1.0, and 10 ppm by weight based on STP dry air. The injection rate shall be continuous and shall not vary greater than  $\pm$  10 per cent. The method of generating iodine vapor shall be thoroughly tested to assure complete reduction to the elemental form. Baffles shall be installed downstream of the injection point in the test facility to insure uniform mixing in the air stream prior to entering the test section.

---

(1) It is recognized that the tests will be performed during the summer months. Due to weather conditions it may not be possible to obtain these temperature and humidity conditions without provisions for cooling the air. Cooling equipment will not be provided, but the test conditions will be adjusted to fit the weather conditions by mutual agreement between the General Electric Company and the contractor.

5. Test section - The test section shall contain prefilters, absolute filters, and halogen collectors in series. The components shall be installed in vertical four feet by four feet banks and shall be spaced at approximately three-foot intervals. Provision shall be made for the replacement of the halogen collector units.

#### 3.1.3 Sampling

Continuous air samples shall be drawn from the inlet, exit, and spaces between the prefilter, absolute filters and halogen collector banks. No more than 5 per cent of the total air flow shall be withdrawn for sampling from the inlet and spaces between the components. One continuous sample per test run will be adequate for the samples from the spaces between the components and the exit. However, for the inlet, at least three samples per test run shall be taken for averaging the inlet iodine concentration rate during the run. If it is possible to measure and record the instantaneous inlet iodine concentration rate by means of radiation instrumentation during the test run, one sample at the inlet, per run, shall be sufficient.

#### 3.1.4 Test Procedure

The supply air shall be heated to the test temperature and then humidified to the test relative humidity. After equilibrium has been reached, iodine will be injected in the air stream and the test run started. The length of the test runs for 0.1 and 1.0 ppm tests shall be at least two (2) hours and for the 10 ppm tests one (1) hour. Each test shall be repeated at least three (3) times to verify the efficiency at that particular air condition and iodine concentration. Following completion of the last test run for the particular halogen collector, the air flow shall be continued through the test section for at least 48 hours to determine the iodine "leakage" rate of the air filters and halogen collectors.

3.1.5 Iodine Removal Efficiency Test Runs

The following test runs shall be conducted with the specified halogen collectors in the test section as outlined below:

Halogen Collector	Iodine Concentration ppm	Face Air Velocity fpm	Air Temperature F	Relative Humidity Per cent	Minimum No. of Tests
Silver-Plated Copper Ribbon Bed 2" Deep	0.1	250	70	70	3
			70	100	3
			160	100	3
			70	70	3
			70	100	3
	10.0	10.0	160	100	3
			70	100	3
			160	100	3
			70	100	3
			160	100	3
Activated Charcoal Bed 1/2" Deep	0.1	250	70	70	3
			70	100	3
			160	100	3
			70	70	3
			70	100	3
	10.0	10.0	160	100	3
			70	100	3
			160	100	3
			70	100	3
			160	100	3
Activated Charcoal Bed 1" Deep	0.1	250	70	70	3
			70	100	3
			160	100	3
			70	70	3
			70	100	3
	10.0	10.0	160	100	3
			70	100	3
			160	100	3
			70	100	3
			160	100	3
Molecular Sieve Bed 1" Deep	0.1	250	70	70	3
			70	100	3
			160	100	3
			70	70	3
			70	100	3
	10.0	10.0	160	100	3
			70	100	3
			160	100	3
			70	100	3
			160	100	3

<u>Halogen Collector</u>	<u>Iodine Concentration ppm</u>	<u>Face Air Velocity fpm</u>	<u>Air Temperature F</u>	<u>Relative Humidity Per cent</u>	<u>Minimum No. of Tests</u>
Molecular Sieve	0.1	250	70	70	3
Bed 1" Deep plus			70	100	3
Activated Charcoal			160	100	3
Bed Depth (to be determined from the above tests)	1.0		70	70	3
			70	100	3
			160	100	3
	10.0		70	100	3
			160	100	3

### 3.1.6 Required Test Data

The following data shall be required from each test run:

1. Air flow.
2. Air temperature.
  - a. Inlet.
  - b. Outlet.
3. Air humidity.
  - a. Inlet.
  - b. Outlet.
4. Pressure drop across individual components.
  - a. Initial.
  - b. Final.
5. Iodine concentration.
  - a. Inlet
  - b. Space between prefilter and absolute filter.
  - c. Space between absolute and halogen collector.
  - d. Exit.
6. Iodine removal efficiency for each component.
7. Cumulative iodine loading of the components.
  - a. Initial.
  - b. Final.

8. Iodine "leakage" rates of the components.

3.2 Iodine Loading Tests

3.2.1 General

The loading tests shall be accelerated test runs to determine the iodine loading capacities of the candidate halogen collectors.

3.2.2 Test Facility

The test facility shall be sized for an air flow 1,000 SCFM and shall be equipped to provide the following test conditions:

1. Supply air - filtered atmospheric air with maximum dust loading of 0.005 mg per cubic meter.
2. Air temperature<sup>(1)</sup> - 70 and 160 F.
3. Air humidity<sup>(1)</sup> - 100 per cent relative humidity.
4. Iodine concentration - 90 per cent of saturated vapor concentration based on the test conditions. The injection rate shall be continuous and shall not vary greater than 10 per cent. Baffles shall be installed downstream of the injection point in the test facility to insure uniform mixing in the air stream prior to entering the test section.
5. Test section - The test section shall contain one halogen collector unit in a vertical bank. Provision shall be made for replacement of the halogen collector unit.

3.2.3 Sampling

Air samples shall be drawn from the inlet and exit of the test section. No more than 5 per cent of the total air flow shall be withdrawn for the inlet sample.

---

(1) See Footnote (1), Paragraph 3.1.2.

### 3.2.4 Test Procedure

The supply air shall be heated to the test temperature and humidified to the test relative humidity. After equilibrium has been reached, iodine shall be injected into the air stream and passed through the test section until a breakthrough is noted. A breakthrough shall be defined as when the iodine removal efficiencies of the halogen collectors determined from the above efficiency tests drop off 10 per cent. When the breakthrough has been reached, iodine injection will be discontinued, and the air flow continued for 48 hours to determine the iodine "leakage" rate. The loading test shall then be resumed to ascertain whether additional iodine would be retained.

### 3.2.5 Iodine Loading Test Runs

The following test runs shall be conducted:

Halogen Collector	Iodine Concentration	Face Air Velocity fpm	Air Temperature F	Relative Humidity Per Cent	No. of Tests
Silver-Plated Copper Ribbon Bed - 2" Deep	As noted in Paragraph 3.2.2	250	70 160	100 100	1 1
Molecular Sieve Bed, 1" Deep	As noted in Paragraph 3.2.2	250	70 160	100 100	1 1
Activated Charcoal Bed, 1/2" Deep	As noted in Paragraph 3.2.2	250	70 160	100 100	1 1

### 3.2.6 Required Test Data

The following data shall be required from each test run:

1. Air Flow.
2. Air temperature.
  - a. Inlet.
  - b. Outlet.

### 3.5 Test Components

The properties and engineering data of the components which will be used in the test program are listed below. These components will be supplied by the General Electric Company. Additional components are included to avoid delaying the test program due to the lack of spare test components which may result from some unforeseen circumstances. The test components not used shall be returned to the General Electric Company. The contaminated components shall be disposed of by the contractor.

#### 1. Air Filters

##### a. Prefilters

Frame size	24" x 24" x 8-3/4"
Media	Fiberglas
Rated capacity	1000 cfm
Pressure drop (clean)	0.16 in. w. g.

##### b. Absolute Filter

Frame size	24" x 24" x 11-3/4"
Media	Fiberglas
Rated capacity	1000 cfm
Pressure drop (clean)	0.9 in.

#### 2. Halogen Collectors

##### a. Silver-Plated Copper Ribbon Bed

Frame size	24" x 24" x 2"
Media	Silver-plated copper ribbon
Ribbon size	0.002" x 0.018"
Bed thickness	2 in.
Packing density	27 lb. /cu. ft.
Rated capacity	1000 cfm
Pressure drop (clean)	0.4 in. w. g.

##### b. Activated Charcoal Filters

Frame size	24" x 24" x 8-3/4"
Media	Activated charcoal-coconut shells
Packing density	34 lb. /cu. ft.
Particle size	6 - 10 mesh
Bed thickness	0.5 in.
Rated capacity	1000 cfm
Pressure drop (clean)	0.2 in. w. g.

c. Activated Charcoal Filter

Frame size	24" x 24" x 8"
Media	Activated charcoal-coco-nut shells
Particle size	8-14 mesh
Bed thickness	1.0 in.
Rated capacity	1000 cfm
Pressure drop (clean)	0.25 in. w. g.

d. Molecular Sieve Bed

Frame size	24" x 24" x 8"
Media	Linde Molecular Sieve 13X
Particle size	1/16" pellets
Bed thickness	1 in.
Rated capacity	1000 cfm
Pressure drop	

#### 4.0 PREFERENTIAL ORDER OF TESTING

The preferential order of testing shall be as listed in Paragraphs 3.1.5 and 3.2.5. Following evaluation of the efficiency tests, the loading tests shall be performed.

#### 5.0 REPORTS

The following information and reports shall be required:

1. Complete set of engineering drawings of the test facilities.
2. Test and analytical procedures prior to initiating tests.
3. Bi-weekly progress reports in letter form (5 copies).
4. Final report - Ten (10) copies and one reproducible copy of the final report shall be provided and shall contain the following information:
  - a. Schematic drawings and photographs of the test facilities.
  - b. Test and analytical procedures.
  - c. Test data.
  - d. Unusual test problems and performances.
  - e. Analysis of test results based on performance.
  - f. Recommendations and conclusions.
  - g. Future development work recommended.

## 6.0 DEVELOPMENT HISTORY OF THE "DRY" IODINE REMOVAL PROCESSES

The following is a brief synopsis of the laboratory and plant performances of the "dry" iodine removal processes which may be of interest and benefit in conducting the developmental test program:

### 1. Silver-Plated Copper Ribbon Bed

Preliminary laboratory testing has been performed at the Harvard Air Cleaning Laboratory as part of a development program being conducted at the request of the Division of Reactor Development, U. S. Atomic Energy Commission. Preliminary tests, conducted with stable iodine, have indicated removal efficiencies of 99 per cent at 20°C with somewhat higher efficiencies at 300 C. The tests were conducted at superficial velocities from 60-240 fpm through a packing density of 27 lb. /cu. ft. Pressure drop through the bed at 240 fpm was 0.4 inches of water. Iodine concentrations ranged from 10-30 ppm. Additional testing will be performed using radioactive Iodine-131 tracer techniques.

### 2. Activated Charcoal Bed

Laboratory tests at Oak Ridge have indicated iodine removal efficiencies of 99.9 per cent through a 12-inch bed. The mesh size of the charcoal was 8-14. The pressure drop through the bed at 170 fpm was 2.8 inches of water. Analyses of the bed indicated that more than 90 per cent was "retained" in the first 3/8-inch of the bed depth. The iodine remained "fixed" and did not move through the beds. Plant facility performances have also shown similar results.

### 3. Molecular Sieve Bed

Linde Molecular Sieve Material was also tested under similar laboratory conditions as the activated charcoal bed. The iodine removal efficiency was 99.9 per cent for the bed. However, only 55 per cent of the iodine was retained in the first 3/8-inch of the bed and a slow, steady downstream migration of iodine through the bed was noted.

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