3-1532, 2-D

GENERAL ELECTRIC
COMPANY

[redacted]

HW-33313
Page 1

DECLASSIFIED

NOT UCNI

This document consists of
20 pages [redacted]

IMPROVEMENT OF THE HYDROGEN FLUORIDE GAS DELIVERY SYSTEM IN THE 234-5 BUILDING

Classification Cancelled and Changed To
DECLASSIFIED
By Authority of CG-PR-2
D. S. Lewis 7-24-92
By J H Wells 11-21-94
Verified By J E Savely 11-22-94
NOT UCNI

Prepared by

E. O. Swain

October 6, 1954

Process Engineering Sub-Section
Design Section
Engineering Department
General Electric Company - Hanford Atomic Products Operation

This document has been reviewed and approved by the appropriate members of the Process Engineering Sub-Section as a basis for the preparation of a project proposal and detailed designs for improving the hydrogen fluoride gas delivery system in the 234-5 Building.

BEST AVAILABLE COPY

Head, Separations Design & Development

Manager, Process Engineering

[redacted]

[redacted]

THIS DOCUMENT IS PUBLICLY
AVAILABLE

[redacted]

DECLASSIFIED

12, 2-D

GENERAL ELECTRIC
COMPANY

[redacted]

DECLASSIFIED

DISTRIBUTION

1. RH Beaton
2. RS Bell
3. HJ Bellarts
4. JL Boyd
5. HA Carlberg
6. VR Cooper
7. WJ Dowis
8. ME Forsman
9. OH Greager
10. DR Gustavson
11. HE Hanthorn
12. WM Harty
13. TW Hauff
14. OF Hill
15. FT Keenan
16. CE Kent
17. CA Lyneis
18. WK MacCready
19. JE Maider
20. JS McMahon
21. WN Mobley
22. EG Pierick
23. CA Rohrmann
24. HP Shaw
25. OV Smiset
26. AE Smith
27. JM Smith
28. RE Smith
29. EO Swain
30. WK Woods
31. 300 Area File Copy
32. Yellow File
33. Extra
34. Extra
35. Extra

DECLASSIFIED

[redacted]

IMPROVEMENT OF THE HYDROGEN FLUORIDE GAS DELIVERY SYSTEM IN THE 234-5 BUILDING

INTRODUCTION

The dry chemistry operation of the RM Line in the 234-5 Building consists basically of receiving plutonium oxalate in small platinum lined "filter boats", placing these boats inside electrically heated resistance type furnaces, and converting the oxalate powder into plutonium tetrafluoride by drawing a mixture of hydrogen fluoride gas and oxygen through the product. The process requires that the gas be supplied at a rate of 500 grams per hour per furnace. If this amount of gas cannot be supplied continuously to all furnaces simultaneous and under sufficient and steady pressures, the result will be incomplete or inadequate fluorination of the product. This necessitates recycling of the product through the furnaces with a resulting decrease in the overall capacity of process equipment equal to the amount of recycled material. The dry chemistry equipment is referred to as "Task II" and presently consists of six operating furnaces with the seventh furnace currently being installed under Project CG-551.

The hydrogen fluoride gas delivery system does not, at all times, deliver sufficie gas to the furnaces. It is estimated that about six (6) psig is the minimum requi ment for good gas control. The existing system will not maintain this pressure under certain conditions of high flow rate and low liquid level in the HF bottles. The solution of this problem was made part of the Chemical Processing and Reductio Design and Development Program and has been included as part of Design Order 100991, Task II - Dry Chemistry Equipment - 234-5 Building.

PURPOSE

The primary purpose of this report is to set forth the reasons why the Task II hydrogen fluoride gas delivery system is unable to supply sufficient quantities of the gas to the process at all times and to make recommendations on how to correct this problem. However, it is also a secondary purpose of this report to make general recommendations on all phases of the gas delivery system where it is felt that an improvement can be made in either the design or operation of the system.

SUMMARY AND CONCLUSIONS

The HF gas delivery is accomplished by connecting four anhydrous hydrofluoric acid cylinders to a common header, directly distilling off the gas, and routing it through a steam heated line to the process area. The cylinders are enclosed in a small shed behind the 234-5 Building. The enclosure is electrically heated to maintain a constant temperature of 50°C. (122°F.) to assist in the direct distillation of the HF gas. In the process area each furnace is supplied HF gas through branch lines which connect to a common header routed along the barrier. Each branch line contains equipment for measuring and controlling the flow of gas to the furnace.

The direct distillation of HF gas from the cylinders is not suitable for supplying the required quantities of gas to the process. The amount of gas that may be supplied and the supply pressure is a function of the rate of heat transfer from the atmosphere of the HF enclosure to the cylinders. As the liquid level in the

SUMMARY AND CONCLUSIONS (Cont'd)

cylinders drop there is a corresponding drop in the rate of heat transfer which results in a drop of pressure and quantity of evaporated HF.

If an adequate supply of HF gas can be assured at high and steady pressure, the existing piping system is satisfactory and can be used as is, however, some simplification and improvement of control instruments in the supply line could be made.

RECOMMENDATIONS

The following recommendations are made as a means of providing an adequate hydrogen fluoride gas delivery system in regards to capacity and maintenance.

1. Install an HF system in which the heat of vaporization of HF is supplied at some point other than through the walls of the storage cylinders. The recommended method of doing this is shown on Engineers Sketch, Flow Diagram, attached as Figure I in the appendix.
2. Do not replace the HF header that runs along the barrier in front of Hood #9 as it is of sufficient size to handle the gas requirements under any reasonable amount of positive pressure in the supply system.
3. Remove the differential pressure controllers in the HF feed lines to the furnaces and modify the motor operated flow control valves.
4. Use monel pipe and fittings throughout the HF system from inside the HF enclosure to the process area.

DISCUSSION

Description of Existing System

The Task II operation of the 234-5 Building is the operation in which plutonium oxalate is converted into plutonium tetrafluoride inside electrically heated furnaces. Moderate quantities of hydrogen fluoride gas are used in this operation. The HF gas is routed to the process area through a steam traced monel line originating in a small HF enclosure in back of the 234-5 Building. The HF enclosure is so piped that two banks of HF cylinders, four cylinders per bank, may be alternately connected to the system. HF is supplied by direct distillation of the gas from the bank of 4 cylinders. The temperature of the enclosure is maintained at 50°C. (122°F.) in order to increase the gas pressure inside the cylinders. The cylinders are constructed of steel and, when full contain 200 lbs. of commercial anhydrous hydrofluoric acid.

The Task II process equipment presently consists of six horizontal furnaces all connecting to a single large hood (Hood #9). A seventh furnace is currently being installed under Project CG-551. HF is supplied to each furnace from a common header that runs along a steel contamination barrier in front of the hood. Each furnace is fed from an individual branch line between the furnace and the header. Each of the branch lines contain a remotely operated shut-off valve, a flow meter, a differential pressure controller, and a motor operated regulating valve.

Description of Existing System (Cont'd)

This system as now constituted does not, in all cases, deliver sufficient gas to the furnaces to permit completely satisfactory service. HF is metered through the flow meters at a desired rate of 500 grams per hour per furnace. At times all furnaces must operate simultaneously. The principle effect of insufficient flow of HF to the furnace is a sharp increase in the number of batches that must be recycled for refluorination. This of course materially affects the total capacity of the Task II equipment.

Hydrogen Fluoride Evaporation

Under the present method of distilling HF directly from the cylinders, all the heat of vaporization must be supplied from the heated atmosphere of the HF enclosure and transferred through the walls of the cylinders into the fluid. The heat of vaporization of anhydrous HF is plotted in Figure III of the appendix and for present operating conditions is about 200 Btu per lb. As the demand for HF may at times reach the rate of 10 lbs. of HF per hour, the heat that must be transferred from the room to the bottles may reach 2,000 Btu/hr.

Calculations show that, when the liquid level inside the cylinders drops and thus decreases the heat transfer surface, a point is soon reached that sufficient heat will not transfer into the cylinder to maintain adequate operating pressures.

The following sample calculation is given to illustrate how this heat transfer rate can affect the available pressure:

Problem: Calculate pressure in HF cylinder when using HF at rate of ten lbs/hr. with HF enclosure temperature of 120°F. and bottles only 50% full.

$$Q = UA\,(t_1 - t_2)$$

where Q = 200 Btu/lb. x 10 lb/hr. = 2000 Btu/hr.

U = estimated under these conditions to be 2 Btu/hr/sq. ft./degree F.

A = .5 x 51.2 = 25.6 ft^2 on basis of four cylindrical bottles $40\frac{1}{2}$" high x $14\frac{1}{2}$" O.D.

t_1 = 120°F. = Temperature of HF enclosure

t_2 = temperature in bottle °F.

then $2000 = 2 \times 25.6 \times (120 - t_2)$

$$t_2 = 120 - \frac{2000}{2 \times 25.6} = 120 - 39 = 81^\circ F.$$

With a bottle temperature of 81°F. the vapor pressure inside the bottle may be read from the vapor pressure curve, Figure II in the appendix, as 4.4 psig.

[redacted]

Hydrogen Fluoride Evaporation (Cont'd)

With the same flow rate, this pressure will become even smaller as the liquid level in the cylinders lowers. The flow rate of HF is plotted against the cylinder pressure in Figure IV. Figure V shows the amount of flow that can be attained at different liquid levels in the cylinders under various pressure conditions. These two graphs show clearly why the available pressure drops rapidly as the level in the cylinder decreases. These two graphs also indicate that a rather large percentage of the liquid HF will have to be thrown away if adequate pressure is to be maintained.

It is felt the only reliable and the most practical system that would be capable under all conditions to maintain adequate pressures and permit use of all the HF in the cylinder is to provide a means whereby the heat of vaporization can be supplied in a manner other than through the walls of the gas cylinders. The recommended method of doing this is shown on Figure I, Engineers Sketch, Flow Diagram, Proposed HF Evaporization System. A copy of this sketch is in the appendix.

The liquid HF flows from the inverted HF bottles, through a control valve into the monel evaporator vessel. Liquid level in the evaporator is held constant by means of a liquid level controller and alarm controlling the flow control valve in the feed line. A separate high liquid level alarm is provided as an added safety feature. The HF is heated and evaporated by warm water flowing in the heater coils. The warm water is obtained by mixing cold water and steam and is kept at constant temperature, not to exceed 130°F., by controlling the quantity of steam with a temperature controller and flow control valve. The evaporator is maintained at a constant pressure by means of a pressure controller controlling the quantity of warm water supplied to the coils. Although there are several satisfactory methods of adapting this scheme to the present installation, the following is probably the most satisfactory method:

1. Modify the existing HF enclosure manifold and cylinder racks to provide means for inverting and securely fastening the cylinders before connecting them to the manifold.
2. Install evaporator and controls in a separate enclosure constructed behind the 234-5 Building adjacent to the existing enclosure.
3. Use the existing 1¼" monel line to transfer the gas from the evaporator to the process area.

The estimated cost for revising the HF gas delivery system in accordance with this scheme is $12,000, including approximately $1,400 for contingency. A copy of this cost estimate is included in the appendix.

HF Piping and Control System

Each of the HF branch lines that connect the furnace to the header along the barrier is equipped with valves and instruments for measuring and controlling the flow of HF to the furnace. A fluorothene, air operated, diaphragm valve is used for a shut-off valve. A motor operated valve is used to set the desired amount of flow to the furnace. A differential

[redacted]

HF Piping and Control System (Cont'd)

pressure regulator is installed in this line to maintain this flow regardless of pressure fluctuations on either side of the motor operated valve. Maintenance work on this section of the HF line is difficult because of the limited amount of working space between the barrier and Hood #9. Any simplification in the make-up of the line will result in a reduction in the amount and cost of the required maintenance.

There have been several changes in valving and piping of this line since it was originally installed. Mathematical analysis confirms operating experience in that it appears good flow control with the present system would be impossible. The differential pressure regulators are designed to maintain a 3 psi differential pressure across the motor operated control valve. This flow control valve is a ¼" AVECO Series #1090 valve constructed of monel and provided with a standard AVECO characterized needle. Manufacturers flow data on this valve indicate that, to maintain a flow of 500 grams per hour at a 3 psi differential pressure, the valve must operate at considerably less than 10% opening. The differential pressure regulator is a Conoflow Corporation, Model H-11-T. It is probable that under normal conditions this regulator is operating so near the fully closed position that it is considerably beyond its designed operating range.

In order to correct these conditions and at the same time simplify this section of the HF line the following method of control is recommended:

1. Remove the differential pressure controllers from the HF line.

2. Provide a gas delivery system and instrumentation as outlined previously plus an accurate pressure regulating valve in the HF header to maintain a constant header pressure under all operating conditions.

3. Provide the motor operated valves with new special designed plugs to replace the standard characterized needles.

With a constant header pressure and a properly designed motor control valve, the relatively small variations in the furnace pressures, which are in the order of a few inches of water, will not materially affect the flow of gases into the furnace.

In regards to the special designed plugs to replace the existing teflon needles, attention is directed to Document HW-25955. The work performed by the Separations Technology Unit, Technical Section, in development of valve stem cones for this same AVECO valve is described in this document. The development of valve plugs as recommended herein could be done reliably only by repeating this type of work with the newly defined flow conditions. It is suggested, however, that better flow control and valve characteristics can be obtained if the valve stem plugs are designed to be cylindrical to give a close fit inside the cylindrical bore of the AVECO valve. Control is attained by maching a small "V" shaped groove longitudinally along the surface of the cylindrical plug.

Because of the lack of sufficient data and the many variables involved, all instrumentation should be designed flexible enough so that optimum operating pressures may be determined under actual operating conditions.

HF Piping and Control System (Cont'd)

Also present plans for improvements and modifications to the basic fluorination equipment dictate that this flexibility be provided. It is believed the following recommendations will provide the necessary flexibility.

1. The gas generator should be provided with instrumentation so that the operating pressure may be readily varied between 5 and 20 psig.

2. The pressure regulating valve in the HF header has a regulated pressure output range of from 2 to 15 psig.

There has been much discussion regarding the size of the HF header running along the barrier in front of Hood #9. This header is constructed of $\frac{1}{2}$" monel pipe. Each of the branch lines are 3/8" monel tubes. The question has thus arisen whether this header is of sufficient size to adequately supply each of these branch lines. Calculations show that the pressure drop through this line is very small for even the maximum flow rates. For example, the pressure drop through 50 feet of this line with HF at $140^{\circ}F$. and 12 psig flowing at the rate of 10 lbs. per hour is only .0535 psi or 1.49 inches of water. This calculation is included in the appendix. With the same mass flow rate except with a pressure of 2 psig this pressure drop approximately doubles.

In either case the amount of pressure drop is very small and can be neglected provided the total pressure in the header can be maintained at any level in excess of 2 psig. However, under present conditions of operation with several furnaces connected to this header and each furnace held at least to 10" H_2O Vac. it is possible for the header pressure to decrease to zero lbs. gage or actually have a negative gage pressure. Under such circumstances the very small pressure drops can have an appreciable effect. As the flow rate into each furnace is inversely proportional to the square root of the length of line, the first furnaces will then "rob" the other furnaces and equal flows can not be maintained. This leads to the conclusion that the refluorination rate will increase as length of the HF line increases. Operating data confirm this conclusion to be generally true.

The installation of an HF evaporator and the attainment of higher pressures will eliminate any need for replacing this header with a larger line. These calculations and conclusions are made, of course, neglecting any possibility of plugging in the header. In the event of any future evidence of plugging, it may then be decided to replace the header with a larger diameter pipe. No justification for this, however, is indicated at this time.

Materials of Construction

Carbon steel is the most widely used construction material for equipment in which anhydrous hydrofluoric acid is conveyed or processed. It has been widely used in the 234-5 Building HF system with some degree of success but the present trend is toward replacement of steel with monel. Recently the steel transfer line between the HF enclosure and the process area was replaced with a monel line. It is recommended that this trend toward monel continue, if not be actually increased. Below is a list of reasons which indicates that all steel components in the system should eventually be replaced with monel.

[redacted]

DECLASSIFIED

Materials of Construction (Cont'd)

1. The corrosion rates of steel are measurably higher at room temperature than monel and show a significant increase with an increase in temperature. Steel is not considered a satisfactory material for service above a temperature of 150°F. Many parts of the Task II HF supply system exceed this temperature. The corrosion rate of monel also increases with temperature but not nearly so rapidly as steel.

2. The HF corrosion process, in most cases, seems to be accompanied by the evolution of hydrogen, while the fluorine combines with the metal to form metallic fluorides. The resulting fire and explosion hazards created by the presence of hydrogen gas make it imperative in the Task II process that hydrogen formation be minimized. Minimum corrosion rates thus will minimize hydrogen formation, and the use of monel will minimize these corrosion rates. It should be pointed out that hydrogen gas may be formed in spite of passivation of the steel.

3. By necessity, all welded construction on HF piping is used insofar as practical. When welding steel pipe, great care must be used to eliminate slag since slag pockets are attacked very rapidly by the HF acid. Monel piping with inert-gas welding will minimize this potential danger spot.

4. When using steel pipe and fittings in an HF system, great care must be exercised to insure use of satisfactory materials. For example, lap and butt welded steel pipes are subject to attack at the seams, thus necessitating the use of only seamless steel pipe. Also materials that are brittle or notch-sensitive must be avoided. This includes free-machining steel, high phosphorous steel and other materials of similar properties. On a very small HF system of the type used in the 234-5 Building, the savings would be too small to justify setting up a reliable system that will positively eliminate the use of any of these unsatisfactory materials in the HF lines.

Edwin O. Swain
Separations Design & Development
Design Section
ENGINEERING DEPARTMENT

EO Swain/baj

DECLASSIFIED

[redacted]

[redacted]

APPENDIX

SAMPLE CALCULATION OF DETERMINATION OF PRESSURE DROP IN HF PIPING

Problem: Determine the pressure drop in 50 feet of ½" schedule 40 pipe with HF gas at 140°F. and a pressure of 12 psig flowing at the rate of 10#/hr.

Formula: This problem may be solved by use of the general flow equation shown below and developed in most elementary texts on Fluid Mechanics.

$$M^2 = \frac{2gA^2 \int_{P_2}^{P_1} \rho \, dP}{\frac{fL}{D} + 2 \ln \frac{P_1}{P_2}}$$

in which M = mass flow rate, lbs/sec.

g = 32.2 ft/sec.^{2}
D = inside diameter of pipe, ft.
A = pipe cross sectional area, sq. ft.
L = pipe length, ft.
f = friction factor
ρ = fluid density, lbs/cu. ft.

Two approximations are made to greatly simplify the above equation. The second term in the denominator is the correction for kinetic energy change and is so small compared with fL/D it will be neglected. The second approximation is in the evaluation of ρ dP. It can be safely assumed in these calculations that:

$$\rho = A + BP \text{ where A \& B are constants}$$

$$\int_{P_2}^{P_1} \rho \, dP = \tfrac{1}{2} (P_1 - P_2) \; (\rho_1 + \rho_2)$$

Applying these approximations and substituting constants to convert the terms of the equations into more useable units, the flow equation can be written as follows:

$$M^2 = \frac{D^5 (P_1 - P_2) \; (\rho_1 + \rho_2)}{87 \, fL}$$

where M = mass flow rate - lbs/sec.

f = friction factor
D = diameter - inches
ρ = density, lbs/cu. ft.
P = pressure, psi.
L = length of line - ft.

Solving for Density of HF @ 140°F. & 12 psig:

Figure VII shows the variation of molecular weight of the polymerized HF gas at various temperature and pressure conditions. By use of this graph the density of HF gas at 140°F. and 12 psig is calculated as follows:

[redacted]

[redacted]

DECLASSIFIED

From Figure II the Saturation pressure @ 140°F = 38 psig = 52.7 psia.
Line pressure = 12 psig = 28.7 psia.

Line pressure represents $\frac{28.7}{52.7}$ or .545 x saturation pressure.

Extrapolating from Figure VII the molecular weight is read as 25. Figure VI, molecular weight of HF vs. temperature, is shown for information purposes only.

Density may then be calculated as follows:

$$\frac{\rho_{air}}{\rho_{HF}} = \frac{\text{Mol. Wt. of Air}}{\text{Mol. Wt. of HF}}$$

$$\rho_{HF} : \frac{.120 \text{ grams/mole}}{15 \text{ moles/liter} \times .035 \times 454} = .104 \text{ lbs/ft}^3$$

Solving for friction factor:

$$\text{Reynolds No.} = \frac{DV\rho}{\mu}$$

where D = diameter in ft. = .0518 ft.

$$V = \text{velocity - ft/sec.} = \frac{Q}{A} = \frac{.0267}{.00211} = 12.65$$

$$\rho = \text{density} = .104\#/\text{ft.}^3$$

$$\mu = .00000672 \text{ lbs/ft. - sec.}$$

$$N_r = \frac{.0518 \times 12.65 \times .104}{.00000672} = 10150$$

With Reynolds Number = 10.150 then f = .031

Solving for Pressure Drop

$$M^2 = \frac{D^5 (P_1 - P_2) (\rho_1 + \rho_2)}{87 fL}$$

$$\left(\frac{10}{3600}\right)^2 = \frac{(.622)^5 (P_1 - P_2) (.208)}{87 \times .031 \times 50}$$

$$P_1 - P_2 = .0535 \text{ psi.}$$

$$= 1.49 \text{ in. of water}$$

[redacted]

DECLASSIFIED

DECLASSIFIED

[redacted]

GENERAL ELECTRIC
COMPANY

APPENDIX

C O P Y

September 29, 1954

E. O Swain
Separations Design & Development

COST ESTIMATE
MODIFICATION TO HF ENCLOSURE 234-5 BLDG.
PROJECT D.O. 100991

Please refer to your request of September 10, 1954. You ask that we prepare an estimate of cost for revising the HF gas delivery system in the 234-5 Building. Work to include the following:

1. Revise the manifold and bottle racks in the existing HF shack to conform with the arrangement shown on SK-2-943 and piped in accordance with Engineers Sketch Flow Diagram.
2. Construct another enclosure similar to the existing one shown on H-2-16221 for housing a new HF evaporator.
3. Provide an HF evaporator in the new enclosure complete with piping and instrumentation as shown on attached Engineers Sketch. No changes to piping inside the building will be required.

The estimate is based on all work performance by Plant Forces. Drawings SK-2-943 and H-2-16221 along with a sketch and flow diagram which you have furnished were used in preparing the estimate.

Attached is the cost summary totaling $12,000 including approximately $1,400 for contingency.

Returned herewith are the above-mentioned drawings, sketch, etc. furnished by you.

W. F. Garetson, Head
Estimating & Unit Costs Unit
PROJECT AUXILIARIES SUB-SECTION

WFG:dsm

Attachment

cc: JW Brands
File

DECLASSIFIED

DECLASSIFIED

[redacted]

APPENDIX

C O P Y

MODIFICATION TO HF ENCLOSURE 234-5 BLDG.
PROJECT D.O. 100991

COST SUMMARY

Earthwork	$ 150	
Concrete Work	350	
Carpentry	1,000	
Insulation	100	
Roofing & Flashing	100	
Painting	200	
Electrical Work	150	
Mechanical Work (Incl. Piping)	4,750	
Special Conditions	400	
Sub-Total Direct Cost		$ 7,200
IME (110% of Labor)		2,400
Total Direct Cost		$ 9,600
Design & Administrative Expense		1,000
Contingency 15%		1,400
Total Project Cost		$ 12,000

DECLASSIFIED

[redacted]

DECLASSIFIED

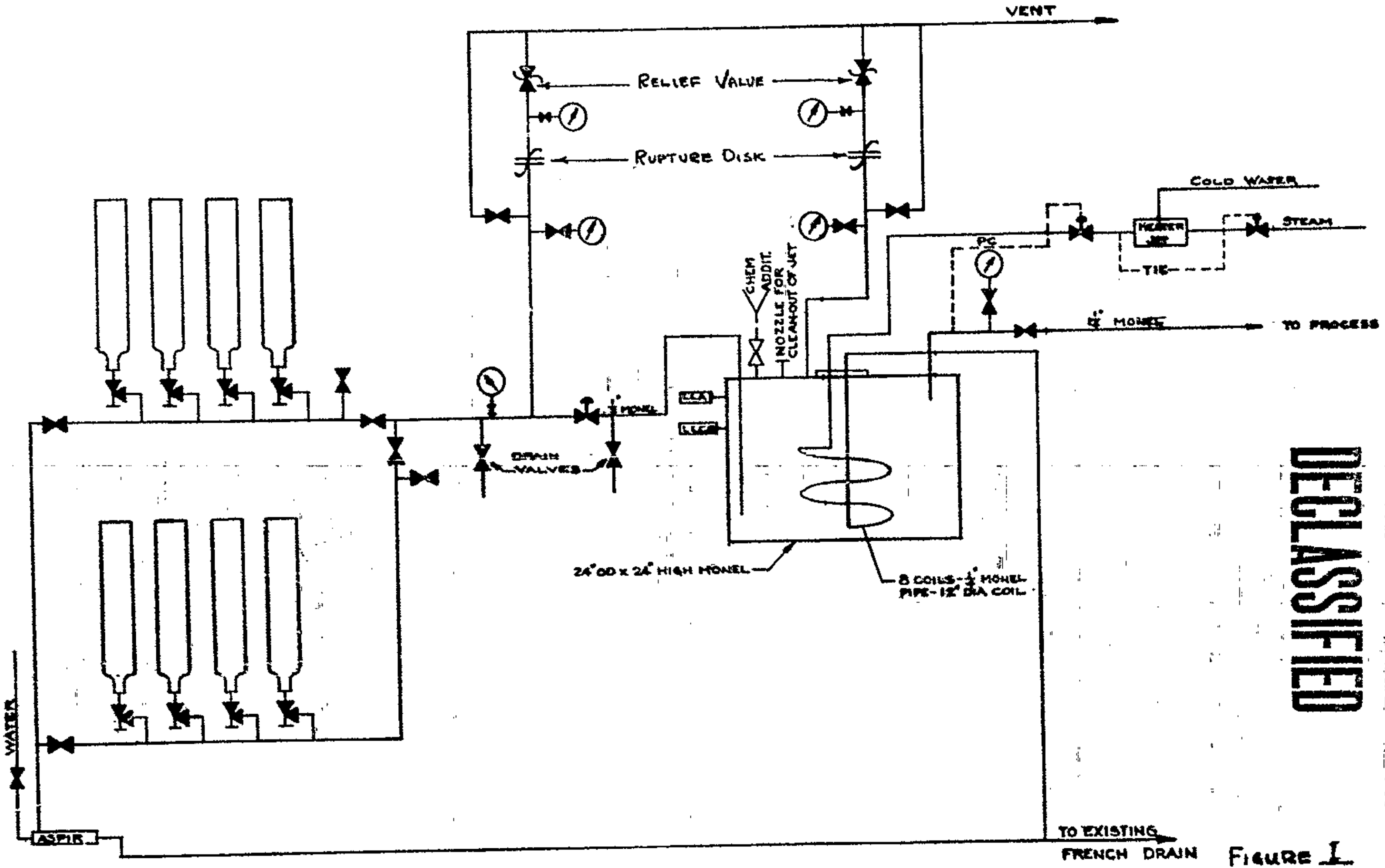


FIGURE 1

ENGINEER'S SKETCH
FLOW DIAGRAM
PROPOSED HF EVAPORATION
SYSTEM

234-5 BLDG.

DECLASSIFIED

[redacted] DECLASSIFIED

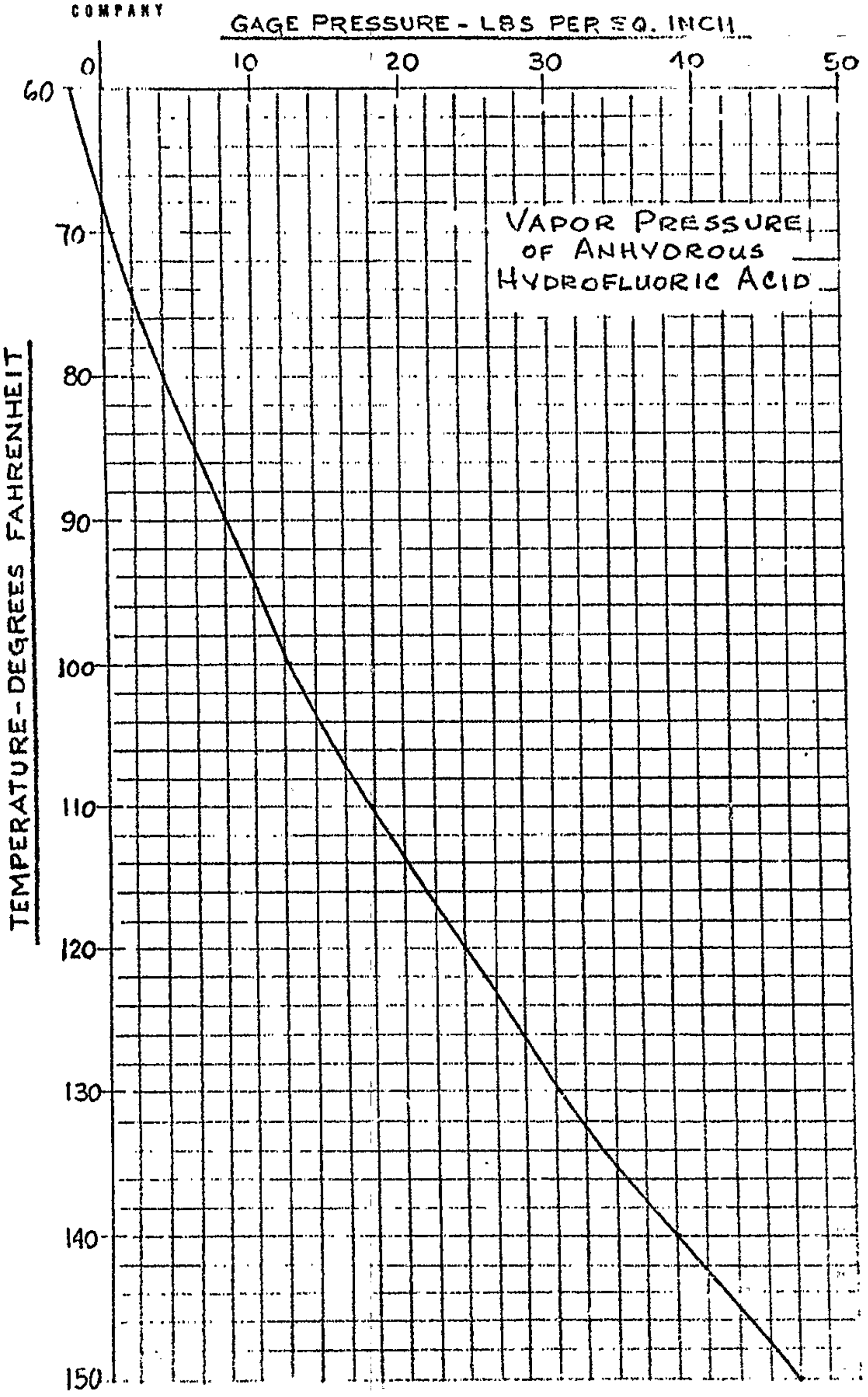
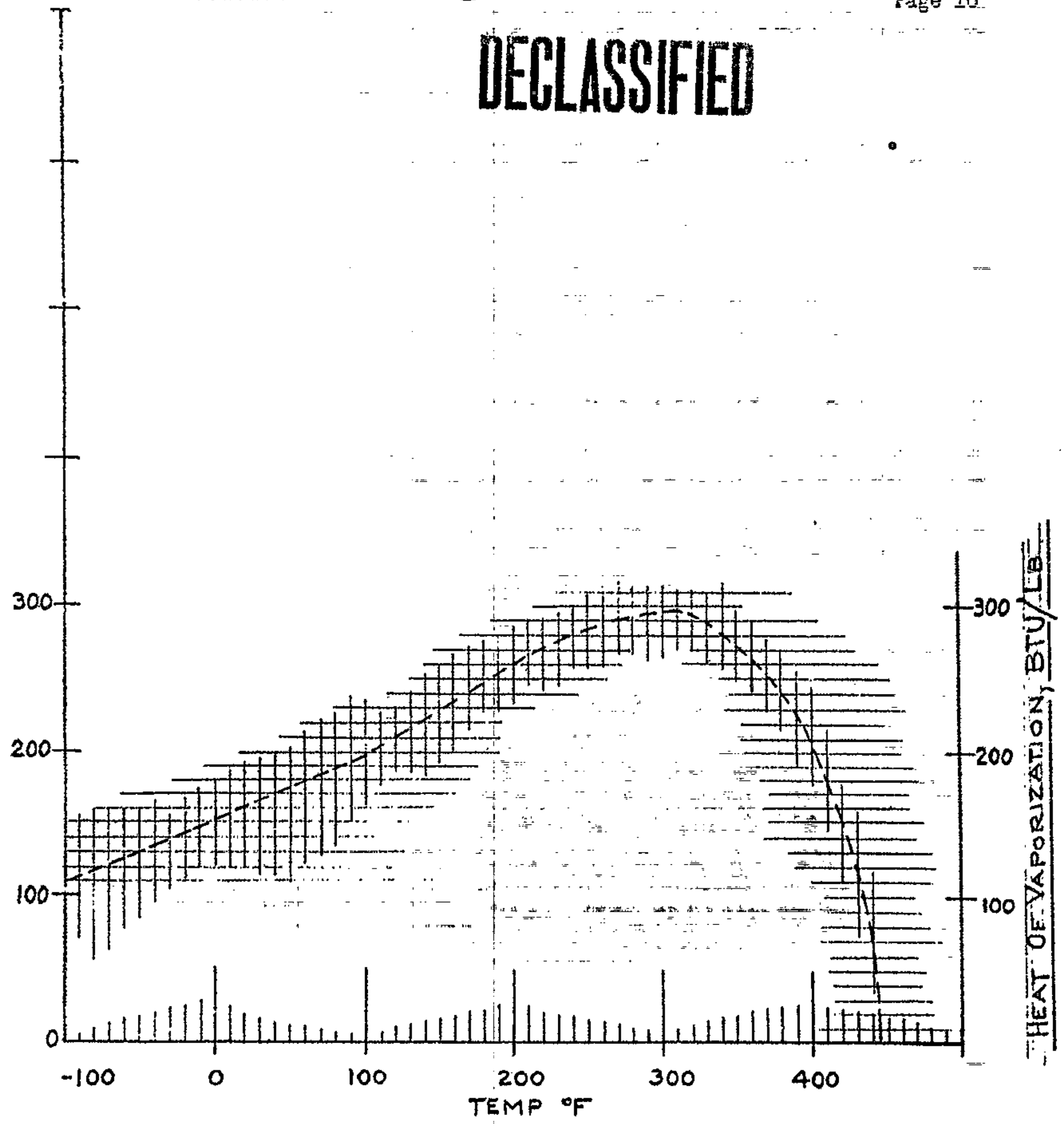


FIGURE I

DECLASSIFIED

[redacted]

DECLASSIFIED



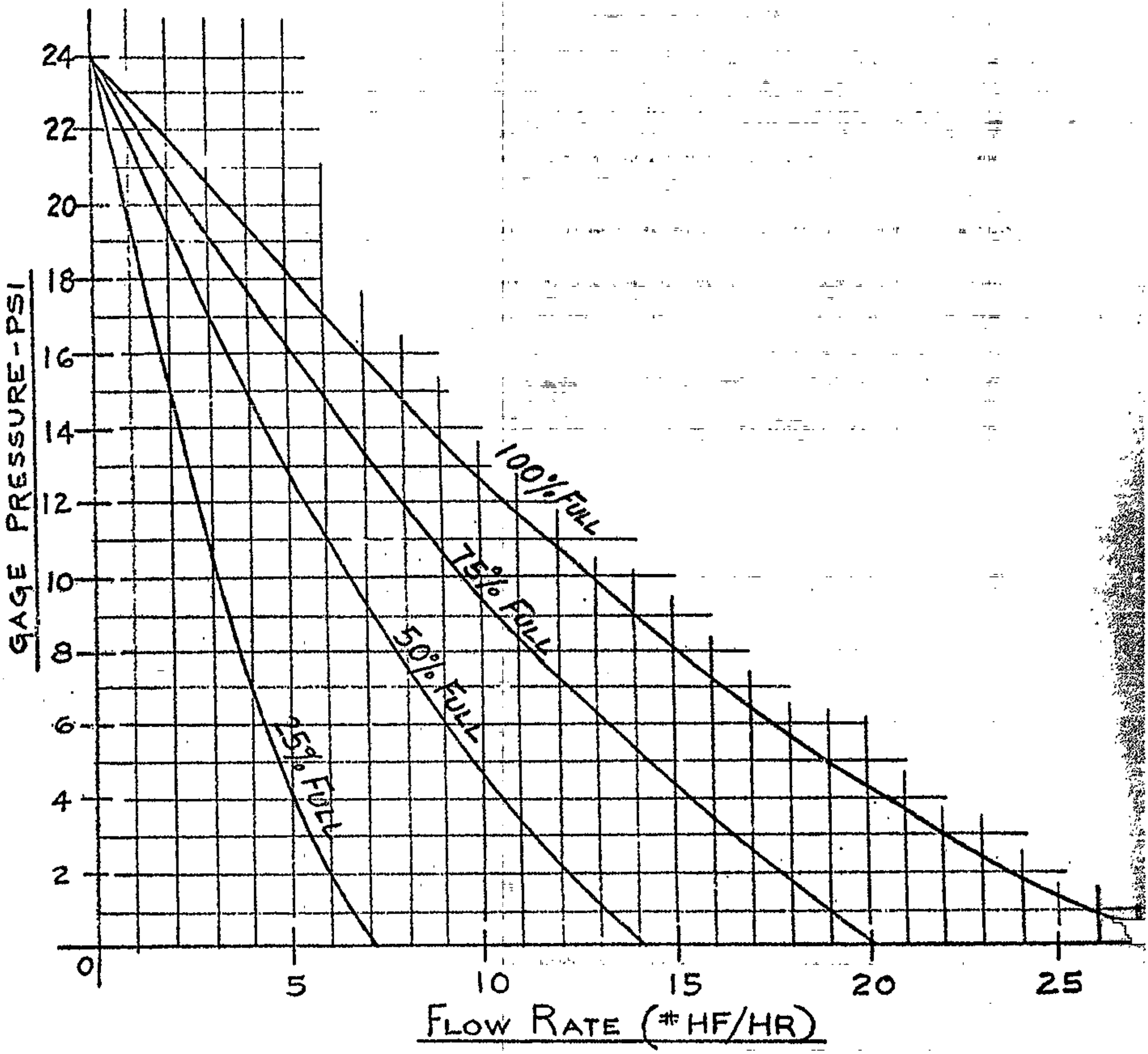
HEAT OF VAPORIZATION
OF
ANHYDROUS HF

FIGURE III

[redacted]

DECLASSIFIED

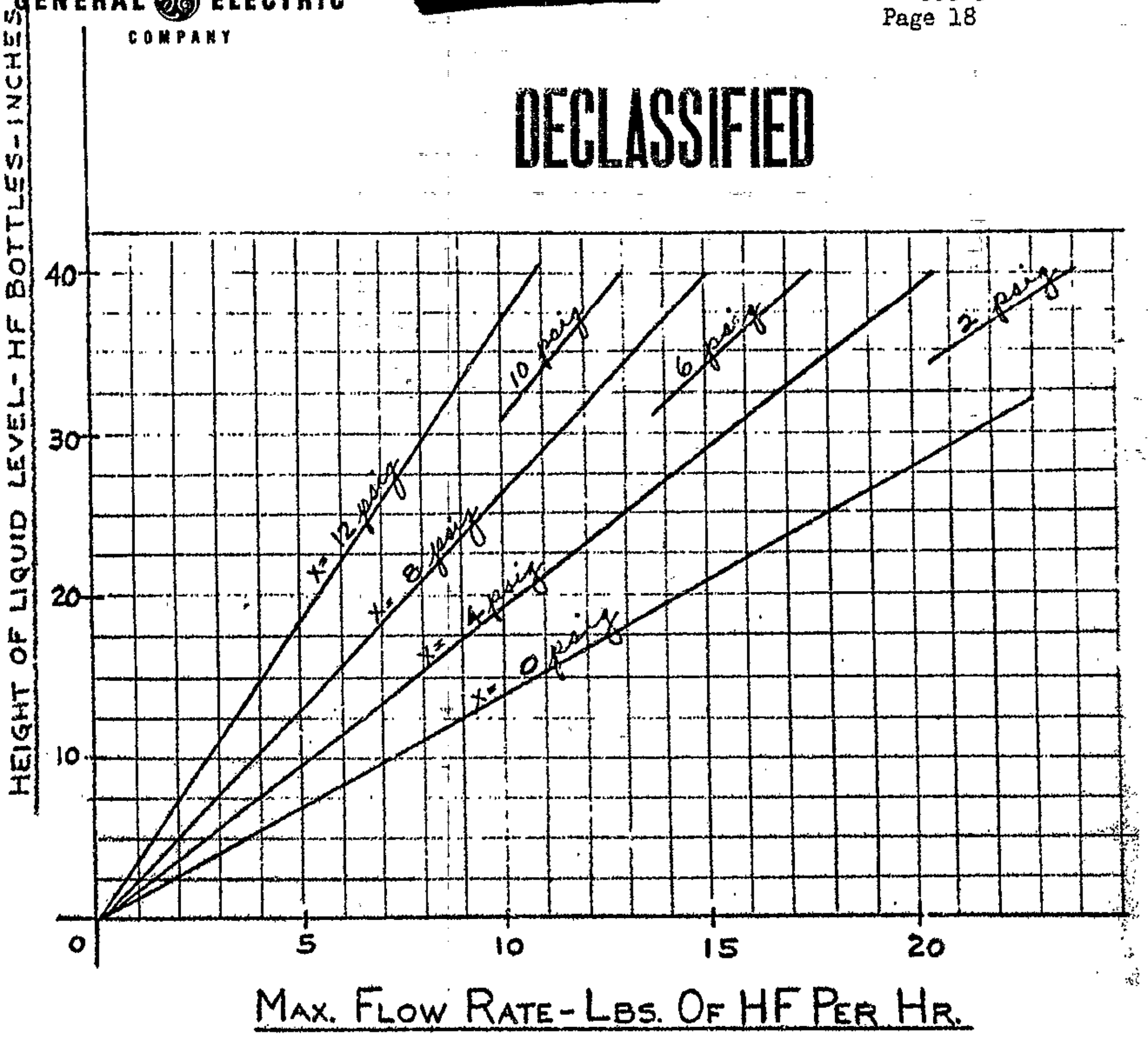
[redacted]



VARIATION OF MAXIMUM FLOW RATE OF HF FROM FOUR BOTTLES LOCATED IN ROOM AT 120°F WITH PRESSURE AT VARIOUS LIQUID LEVELS IN THE BOTTLES.

FIGURE IV

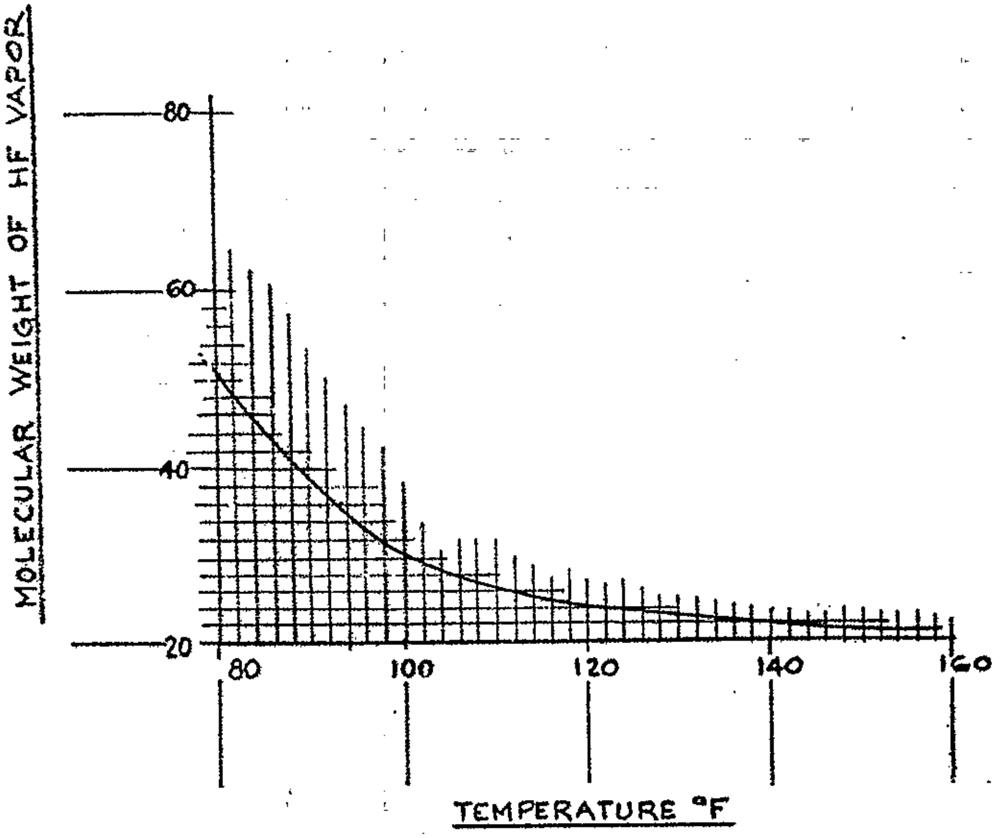
[redacted]



VARIATION OF MAXIMUM FLOW RATE OF HF FROM FOUR BOTTLES IN SHACK AT TEMPERATURE OF 120°F WITH LIQUID LEVEL IN THE BOTTLES AT VARIOUS OPERATING PRESSURES

FIGURE V

[redacted]

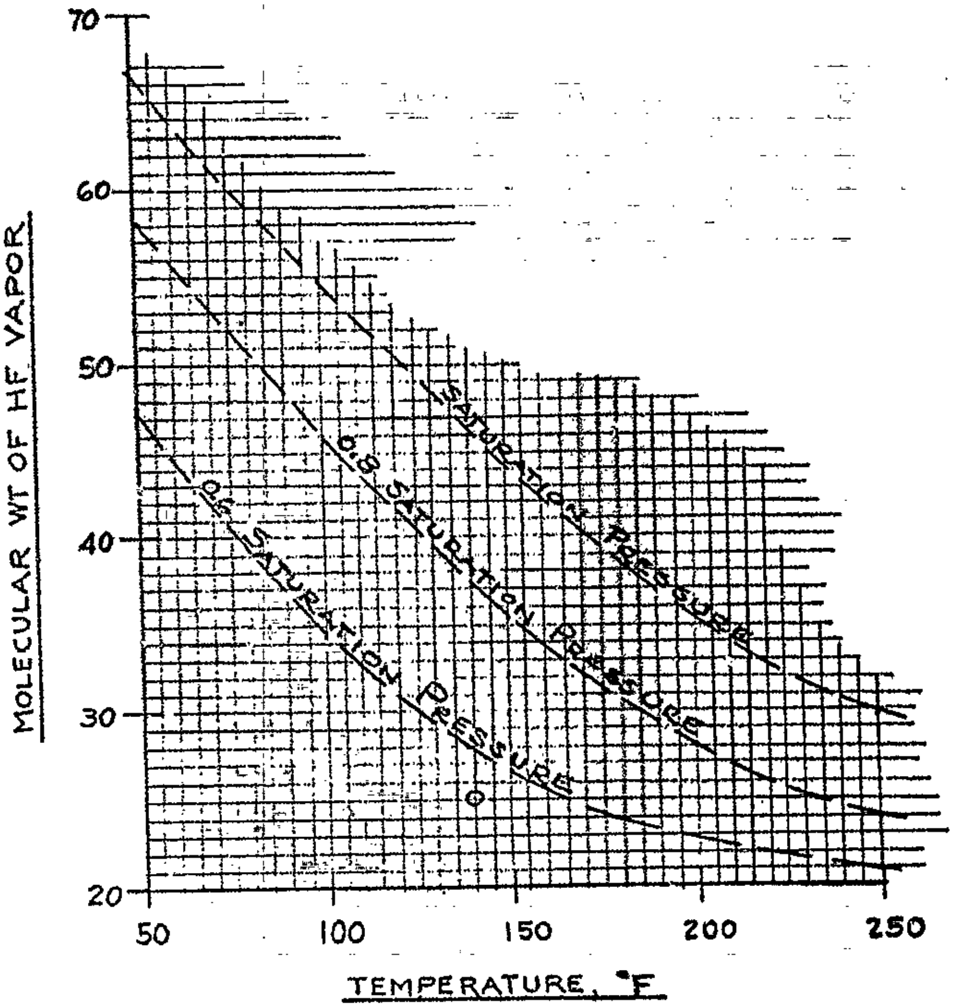


VARIATION OF MOLECULAR WEIGHT OF HF VAPOR WITH TEMPERATURE (AT 14.4 LBS./SQ. IN.)

FIGURE VI

DECLASSIFIED

[redacted]



VARIATION OF MOLECULAR WEIGHT OF HF VAPOR WITH TEMPERATURE AT SATURATION PRESSURE AND FRACTIONS THEREOF

FIGURE VII

DECLASSIFIED