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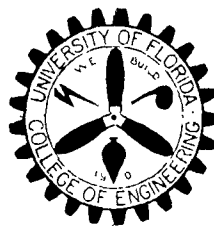
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ENGINEERING AND INDUSTRIAL EXPERIMENT STATION

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FIRST TECHNICAL REPORT
on
THE PREPARATION OF FLUORINE-CONTAINING COMPOUNDS
for
THE CHEMISTRY BRANCH, OFFICE OF NAVAL RESEARCH
under
CONTRACT NUMBER: Nonr-580(03); NR 356-333
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Figure 1 Electrochemical System

Figure 2 Gas System

Figure 3 Probe System

Figure 4 Distillation - Molecular Weight Curves

Table 1 Summary of Results of Electrochemical
Operations of $(C_2H_5)_3N$ in HF . . . 14

SUMMARY

Two separate electrochemical systems and the associated laboratory facilities have been established and are in operation. Work is in progress to erect two more complete and separate electrochemical systems. Laboratory work has entailed mainly the preparation and purification of starting materials for use in the electrochemical process.

One operation of the equipment using triethylamine and hydrogen fluoride as starting materials has been completed. The purpose of the operation was to test the component parts of the electrochemical systems, familiarize the personnel in the operation and to instruct them in the handling of hydrogen fluoride as a liquid and a gas.

In the main, the equipment performed satisfactorily. The desirability of certain minor modifications became evident.

The results of the electrochemical operation on this short trial run indicate a current efficiency of 91%, a fluorine efficiency of 68%, with the yield of $(C_2F_5)_3N$ being about 50% of all the fluorine-containing products isolated or detected. These figures do not necessarily reflect performance of the operation under other than break-in conditions.

INTRODUCTION

The major initial effort in this work was the design, construction, and installation of those components needed for the successful operation of the electrochemical facilities for the preparation of fluorine-containing compounds. At the same time associated research facilities were set up and an attempt was made to anticipate the direction and magnitude of the research work, when fluorine-containing materials from the electrochemical process became available. The direction and magnitude of the research work will dictate the type and quantity of starting materials that are necessary for use in the process to prepare specific fluorine-containing starting materials for purposes of investigation. Accordingly, several of the starting materials needed for the electrochemical process have been procured and modified for use.

It is to be noted that several federal agencies have an interest in this work. To date, sponsorship has come from (1) the Army Chemical Corps Procurement Agency, (2) Chemistry Branch, Office of Naval Research, and (3) the United States Air Force, through the Office of Naval Research. As a result, the facilities have been arranged to meet the anticipated needs of all these agencies. Two complete electrochemical systems are presently in operation and work has been started on two more. Each is, or will be, a self-contained system so that different fluorine-containing materials can be prepared in each simultaneously.

THE COMPONENTS OF THE ELECTROCHEMICAL SYSTEM

Reference is made to a complete schematic diagram (see Figs. 1 and 2) of the system. The following discussion and figures referred to will offer a more or less complete description of the equipment as installed with some modifications resulting from experience gained from a test operation.

I. The Electrochemical Units

The electrochemical units are the heart of the process. Each consists of the following elements:

- a. A 12" portion of 5" or 6" steel pipe to one end of which is welded a 125 lb. steel flange and to the other a dished base, with a 1/4" IPS hole centrally located.
- b. To the exterior of the steel pipe there is either welded a steel jacket, or soldered a copper coil, through which a coolant can be passed to adjust the temperature of the unit.
- c. To the hole in the base is piped a two valve system with a transparent portion of Kel-F (trifluorochloroethylene plastic) tubing for observation of any material withdrawn.
- d. The top (blind) flange is modified to accommodate the following items:
 1. Thermocouple or thermometer wells.
 2. The electrode pack. This consists of a series of alternate nickel cathodes and anodes, the size and number of which are governed by the anticipated operational requirements. Each plate is separated from its neighbors by a teflon spacer. Each anode is connected to the other anodes by means of a nickel bus bar to which it is welded. The same is true of the cathodes. A piece of threaded nickel stock is welded to each

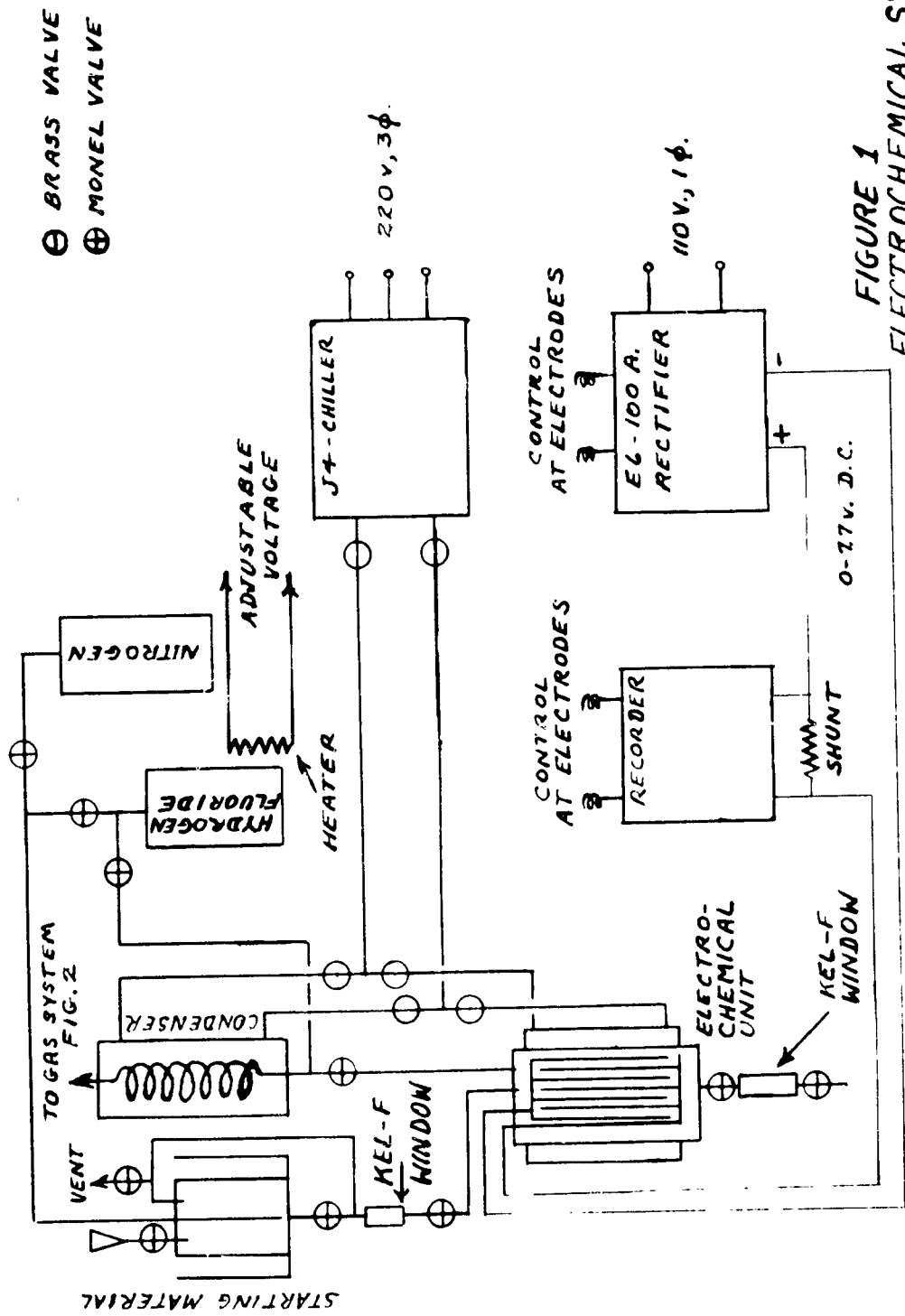


FIGURE 1
ELECTROCHEMICAL SYSTEM

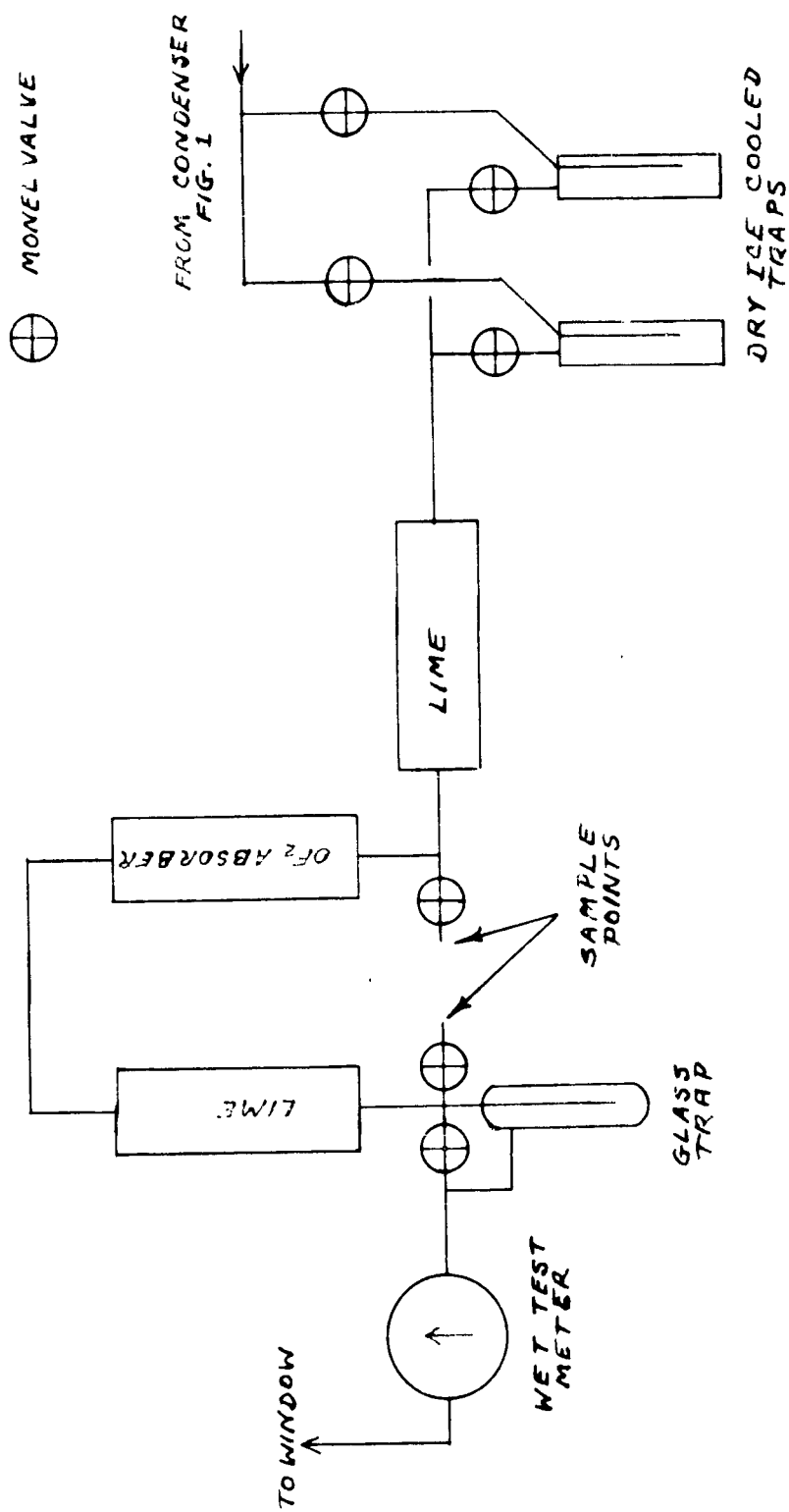


FIGURE 2 - GAS SYSTEM

bus bar and exits through the top flange through holes drilled therein. These supports are also the electrical leads. They are so shaped that when special teflon sleeves and gaskets are inserted over them, the electrodes are electrically insulated from the flange and all possible gas leaks are sealed. The current and voltage control connections are attached to these electrode supports.

3. A tube for adding starting materials.
 4. A gas exit line.
 5. A system of sensing probes for determining the liquid level in the unit.
 6. A system for returning liquid hydrogen fluoride to the unit that has accumulated in a set of cold traps.
- e. When the flanges are bolted together, a special teflon "chemical" gasket is used to seal the unit.
- f. The whole unit is insulated.

The units have a liquid capacity of between 0.75 and 1.25 gallons.

2. Condenser

The purpose of the so-called overhead condenser is to return hydrogen fluoride carried out of the electrochemical unit as a gas by condensing it on a cold surface. The amount of hydrogen fluoride refluxed in this manner can be considerable. First, the escaping hydrogen contains the hydrogen fluoride evaporated from the liquid; and, second, it entrains any spray that may be formed. Simple engineering calculations indicated that 3.6 square inches of surface maintained at -20°C would condense the hydrogen fluoride except for about 8 grams/hr. under a set of conditions at which the electrochemical units

would be operated. Because of a lack of precise data of the properties of hydrogen fluoride, and the heat transfer of the surfaces, and also because of qualitative experimental experience this figure seemed too low. Thus a spiral condenser was constructed from 3/8" nickel tubing offering a 12 foot path and a surface area of 105 sq. in. The flow rates were such that all gas would be in the condenser in the order of 0.5 minutes. The rate of hydrogen fluoride reflux was calculated to be in the order of 0.5 cc. per second.

The condenser was a 12 foot length of 3/8" nickel tubing (.035" wall) bent to form a coil with an I.D. of 1.5", and O.D. of 2.25" and approximately 12" long. This was jacketed by a 15" length of 3" pipe to which an inlet and outlet was welded for the passage of coolant, and the ends were capped. The ends of the coil passed through the caps by means of compression glands containing teflon sleeves. These glands prevented both fluid and electrical leakage. The jacket was adequately insulated to minimize heat leak, and the condenser was placed in the line by means of suitable adapters.

3. Starting Material Addition

The starting materials are organic substances added to the electrochemical unit before and during operation. As these might be either liquids or solids, a system compatible to both had to be devised. Liquids were added to the electrochemical units by the application of a slight positive pressure to the containing vessel. To add solids is a more formidable task. One convenient method is to make a solution of solids in hydrogen fluoride, and add the solution as indicated above. In anticipation of such a procedure, a unit was designed to serve the purpose (see Fig. 1). Essentially it was a cylindrical copper container with a capacity of about one liter. Four tubes enter the container, three in the top and one in the bottom. The first tube in the top

runs all the way to the bottom of the container and is used both to add HF when necessary, and dry N_2 gas to the system either to pressurize it or stir it by bubbling through the liquid. Nitrogen enters through a tee in the HF line. A second tube is flush with the top and is used as a vent to some safe area. The third tube is for adding solids and is larger than the other two. The tube in the bottom admits the liquid into the electrochemical unit. It is double valved on both ends of a piece of Kel-F tubing of known volume. A line from the nitrogen source by-passes the container and is connected at the top of the Kel-F tube. A metal jacket around the main copper cylinder serves as a container for a coolant such as dry ice or ice. This is further insulated with several inches of rock wool. Monel needle valves are judiciously placed in the system to permit the operations outlined.

4. Addition of Hydrogen Fluoride

Hydrogen fluoride is added to the electrochemical units by distilling it from a cylinder heated by a gas-coil heating tape. The heat is controlled through a variable transformer. The HF gas is condensed in the overhead condenser and flows into the electrochemical unit as a liquid. When a certain desired liquid level is attained, it is indicated by the sensing probe system.

It might appear more efficient to add the HF as a liquid directly from the cylinder. However, this has several distinct disadvantages. The first is the added hazard of a liquid leak. The second involves the purity of the HF obtainable. All commercial grades are presently only at best 99% pure. Distillation keeps the water content of the distillate lower and prevents any solid impurities from disturbing the electrochemical process which can become very sensitive to microscopic amounts of foreign matter under certain conditions.

Again, a system of monel valves judiciously arranged reduces any possible hazard of HF leakage to a minimum.

5. Refrigeration

The refrigeration for the overhead condenser and the electrochemical unit is supplied by a unit with the following characteristics:

Compressor H.P.	1.0
Pump H.P.	0.25
Pump capacity	3.0 gallons/min.
Power characteristics	220-3-60 volts/phase/cycles
Refrigeration capacity	2863 BTU at -24°C

This unit is designated a J-4 junior chiller (Mayer Refrigerating Engineers). The gas used is Freon 21 and the circulating coolant is an alcohol water mixture with a freezing point of -40°C . .03% triethylamine is used as a rust inhibitor. The pipes through which the coolant is circulated are insulated with a thickness of rock wool insulations recommended for -40°C . Extra insulation and seals were added to the coolant reservoir to safeguard against some obvious heat leaks.

Precautions had to be taken to remove all the heat generated by the compressor and the heat exchanger from the area, and to protect the equipment from the corrosive effect of HF vapors.

6. Power Supply

It is desirable that the D.C. power supplied to the electrochemical units have certain characteristics. The voltage and current should be as pure D.C. as possible. It is necessary that the voltage to the electrochemical units undergo a minimum of variations with a change in current. It was felt that a properly designed and constructed rectifier and filter circuit would meet the rigid requirements better than a motor-generator, and require less maintenance. To this end selenium rectifiers with voltage control were obtained for operation.

These have the following characteristics:

input voltage range	95-130 volts AC, single phase, 60 cycles
output voltage range	adjustable from 4.5 to 7 volts DC
voltage regulation	$\pm 0.2\%$ from 1/10 to full load
ripple voltage	1% RMS at 60 cycles
capacity	100A DC

The signal disadvantage of such an instrument is the low efficiency. The efficiency is in the order of 25%.

The voltage-control electronic circuit is of rugged construction and lives up to the claims made for it by the manufacturer, Sorensen and Company, Inc.

As this equipment could not be kept in the same area as the electrochemical units, the voltage drop in the lines became significant. As a result the voltage control sensing points had to be extended so that they were located right at the plates of the electrochemical unit. In this way control was at the unit and not at the output of the rectifier. Lines were fused to protect the control circuit of the rectifier and a common ground was established for all electrical equipment.

7. The Recording System

The purpose of the recording system was to obtain a permanent, accurate record of the current and voltage supplied to the electrochemical unit. The recorder chosen for the purpose was an 8 point Minneapolis-Honeywell Brown Elektronik Recorder, modified to measure and record DC voltages from 0 to 10 volts with an accuracy of 0.01 volts and DC currents from 0-100 amperes with an accuracy of 0.1 ampere. The instrument records one point every 6 seconds and when all circuits are active, reads and records continuously four DC voltages and four DC currents.

8. The Probe System

A determination of the liquid level in the electrochemical equipment is a necessary criterion for successful operation. Three 1/8" x 6" nickel rods, covered by 1/4" x 1/8" teflon tubing, are inserted through the top flange of the electrochemical unit by means of compression glands. The levels of the three probes are so adjusted that a signal light is observed, when the level contacts each probe-base. As the lights go out, it is an indication that the level has dropped below a pre-set level; and HF is added to reestablish this level.

Figure 3 is a schematic of the electric circuit for each probe. When the liquid level is below the end of the probe, an almost infinite resistance exists between the probe and ground; when solution contact is made with the probe, a continuous circuit with from 2000 to 20,000 ohms resistance is established.

In order to energize a signal light with a minimum voltage drop and current through the liquid, the circuit was designed around a 2050 thyatron as shown in figure 3. With the probe circuit open during the positive half-cycle of the plate voltage applied by transformers, T2, the grid-circuit voltage supplied by T1 stays below the critical firing potential so that no current flows through the light P1. During the next half cycle the plate is negative so that no plate current can flow. Closing the probe circuit connects the grid directly to the cathode. The grid is then no longer forced negative, when the plate voltage is positive; and the thyatron conducts for a full half cycle and current flows through the light P1. R1 acts as a sensitivity control and can be adjusted for satisfactory performance. The thermostatic time delay relay REL prevents plate voltage being applied to the thyatron, until the filament has reached the proper operating temperature.

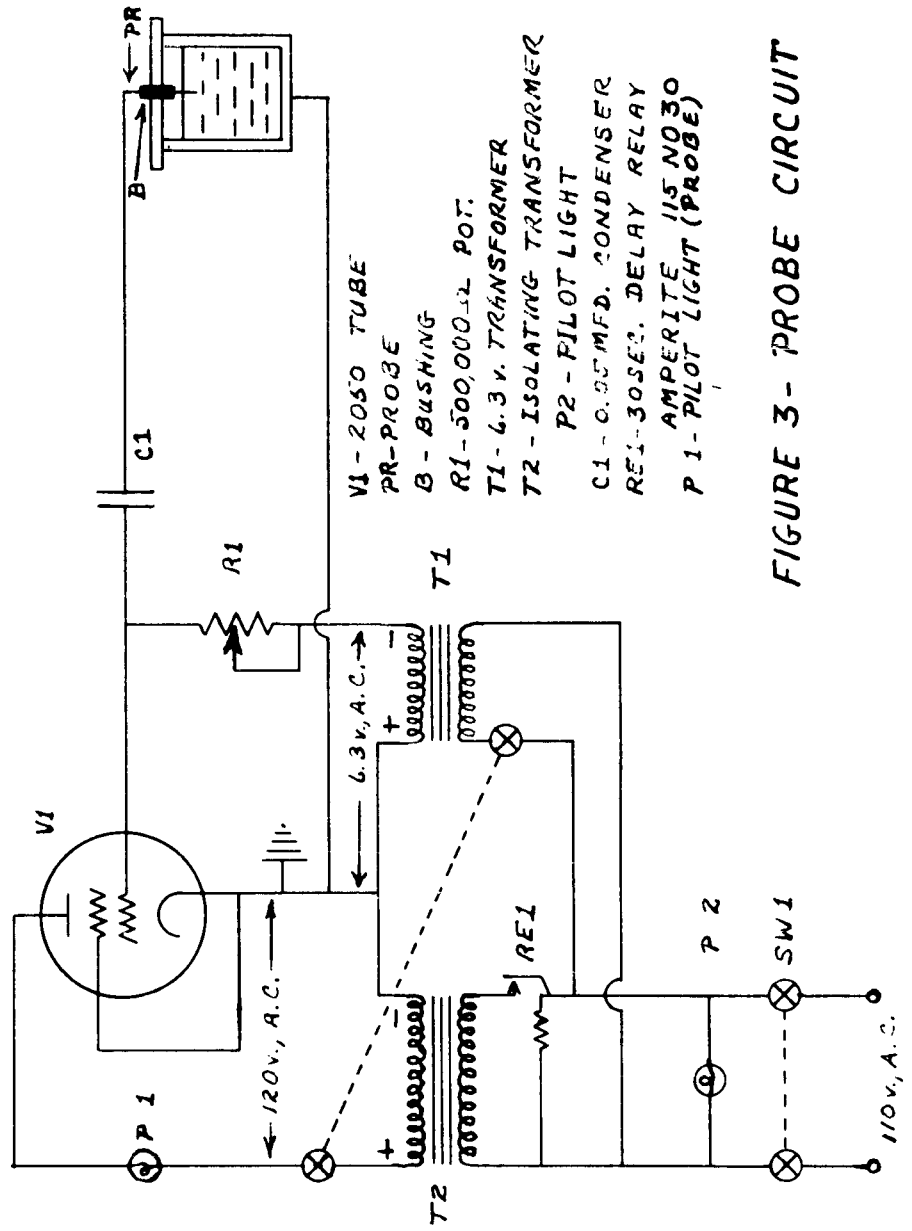


FIGURE 3 - PROBE CIRCUIT

9. The Gas Handling System (see Fig. 2)

The gas handling system contains facilities for the removal of HF, various products, and by-products of the electrochemical process and also metering the hydrogen generated. As mentioned in Section 2, a certain amount of HF is carried beyond the overhead condenser as well as a certain amount of HF insoluble product. In fact, all materials that have an inappreciable vapor pressure at, say, -70°C will tend to condense in the dry ice cooled copper trap in the gas line beyond the condenser. However, any materials that have an appreciable vapor pressure at -70°C will be carried from a dry ice cooled trap by the hydrogen, in accordance with the volume of hydrogen involved and its vapor pressure. For example, it is calculated that HF with a vapor pressure of some 4 mm. of mercury at -70°C is carried over to the extent of approximately 0.16 grams for every mole of hydrogen passed. Thus it is necessary to add to the system a chemical trap containing sodium fluoride or lime to remove this carry-over of HF. Both these substances present certain advantages and disadvantages as an agent for the removal of HF, and their choice is motivated by other operational conditions.

When water or other oxygen-containing materials are present in the electrochemical unit by chance or design, one of the by-products is invariably oxygen fluoride, OF_2 . It is known that in the presence of hydrogen, OF_2 can form an explosive mixture. However, OF_2 can be removed by a number of reducing agents. Actually, plain rubber tubing is very effective for this purpose. If hydrogen is used, water and HF are formed and can be removed by a lime trap. Analysis points for OF_2 before and after the absorber were provided to check the quantity of OF_2 in the gas stream and the efficiency of the absorber. These points can also be used to check for HF and CO_2 when necessary.

A liquid air cooled glass trap can be placed in the line after the lime trap to collect low boiling product, and the residual hydrogen is metered continuously by means of a monel wet test meter and exhausted out-doors.

The various products captured from the gas phase or removed from the bottom of the electrochemical unit must undergo purifications and separations.

OPERATION OF THE SYSTEM

In order to test the operation of all the components, it was decided to test each of the two systems in sequence so that any difficulties arising could be met with a minimum of hazard. As the contents of the electrochemical unit tested first was run into the other unit after the initial test was completed, some saving in HF, starting material, and time was accomplished.

The starting material used in the test was triethylamine, B.p. $86-88^{\circ}\text{C}$. This material offered the advantage of being converted to a main product with an ultimate research use and being a material that presented a minimum of operational difficulties. In the future it will be referred to as "amine".

The procedure for operation was as follows:

HF was distilled from a tared cylinder into the electrochemical unit, with refrigeration supplied to the condenser and the cell. This was continued until the lights associated with the probes were illuminated. The weight of HF transferred was in the order of 5.5 lbs. From experience, it is known that this will contain about 1% water. This can be removed in the form of OF_2 by passing a current through the solution. At a voltage of 5.8 volts the current rapidly increased to 22 amperes and slowly declined to 4 amperes after $3\frac{1}{2}$ hours. From the recorder record the average current showed that 2 faraday of current was passed and that approximately 9 grams of water were removed. OF_2 analyses, before

and after the absorber during this operation, showed that the OF_2 was being quantitatively removed. The amount of OF_2 generated was compatible with the current passed.

At this point 240 cc (175 g; 1.75 mole) amine was added to the dried HF. Since the amine forms a salt with HF with the liberation of heat, this operation was done slowly and under condition wherein both the overhead condenser and the electrochemical unit were cooled. The unit was operated intermittently for a period of 94 hours, being inoperative overnight, on week-ends, and those periods during which modifications were in effect. For 7 hours in the middle of this operational period a liquid air cooled trap was inserted in the line to determine the quantity and character of material passing through the dry ice cooled traps. The voltage impressed was maintained between 5.34 and 5.48 volts while the current spread varied between 42.5 and 56.0 amperes. The voltage was controlled to the nearest .01 volt by the rectification equipment while the current was a function of temperature, voltage, and concentration. During operation, amine was added at the initial rate of 15 grams for every 50 liters of gas metered. This was later reduced to 12 grams. Accordingly 500 grams, in excess of the initial 175 grams, was added in 94 hours.

There were three sources of product, that remaining in the dry ice cooled trap, that found in the liquid air cooled trap, and that removed from the bottom of the electrochemical unit. The first was removed by decanting the complete trap contents into a polythene bottle and syphoning the lower layer of product into another bottle by means of slight air pressure. The residual HF was returned to the electrochemical unit. No appreciable quantity low boiling product material was detected from this source. The material from the liquid air cooled trap was examined for boiling point range and molecular weight.

range and stored for further examination. The material run out from the bottom of the electrochemical unit was added to the high boiling product found in the dry ice cooled trap.

After use in the first electrochemical unit, the liquid contents were transferred to the second unit and used for an additional period of 34 hours intermittently under essentially the same conditions. However, the average input of amine was reduced, since a shut-down was anticipated and the build-up of excess product intermediates would only serve to reduce the yield efficiencies. At the time of shut-down the residual products were removed from the bottom of the electrochemical unit and the remaining contents discarded via a special disposal system.

SUMMARY OF RESULTS

Table 1 gives the average results of the electrochemical operations for the two units and the overall results for the pertinent factors involved. It is to be noted that the voltage control was better than the ranges indicated and that these values were manually imposed to obtain suitable current values under various conditions of temperature and concentration. The variation in current was imposed to enable a study of the HF carry-over under various conditions of temperature and current compatible with the ability to handle it. The current efficiency can be defined as the ratio of the volume of hydrogen actually produced under a given set of conditions to the volume that theoretically should be produced at that set of conditions. The fluorine efficiency, within the limitation of the information available is the ratio of the weight of fluorine in all isolated products over the fluorine that theoretically should have been exchanged in the electrochemical processes. The denominator is evaluated from the number of faradays of electricity passed based on the volume of gas evolved.

TABLE 1

SUMMARY OF RESULTS OF ELECTROCHEMICAL OPERATIONS OF $(C_2H_5)_3N$ in HF

	<u>Unit 1</u>	<u>Unit 2</u>	<u>Units 1 and 2</u>
Voltage range	5.34 - 5.48 v.	5.36 - 5.46 v.	
Average current	46.75 A	42.0 A	
Time	94 hrs.	34 hrs.	128 hrs.
Faradays (current)	164	53	217
Gas metered	1941 liter	604 liter	2545 liter
Gas* (corrected)	1802 l.	568 l.	2370 l.
Faradays (gas equivalent)	151	48	199
Current efficiency	92%	91.5%	91.8%
Product 1. at $-70^{\circ}C$	408 g.	211	619
2. from bottom of unit	185 g	110	295
[I] 3. at $-185^{\circ}C$	[7 hrs.] 45g (610g)	[5 hrs.] 25g (170)	(780)
Est. total product	1203	491	1694 g
Fluorine efficiency [II]	64%	81%	68%
(Based on 100% current efficiency)			

* These values corrected for product metered. All gas values at ambient conditions. Factor of 24 l/mole H_2 .

I () values estimated.

II Values of 74.4% fluorine used for high boiling material and 75% for low boiling material.

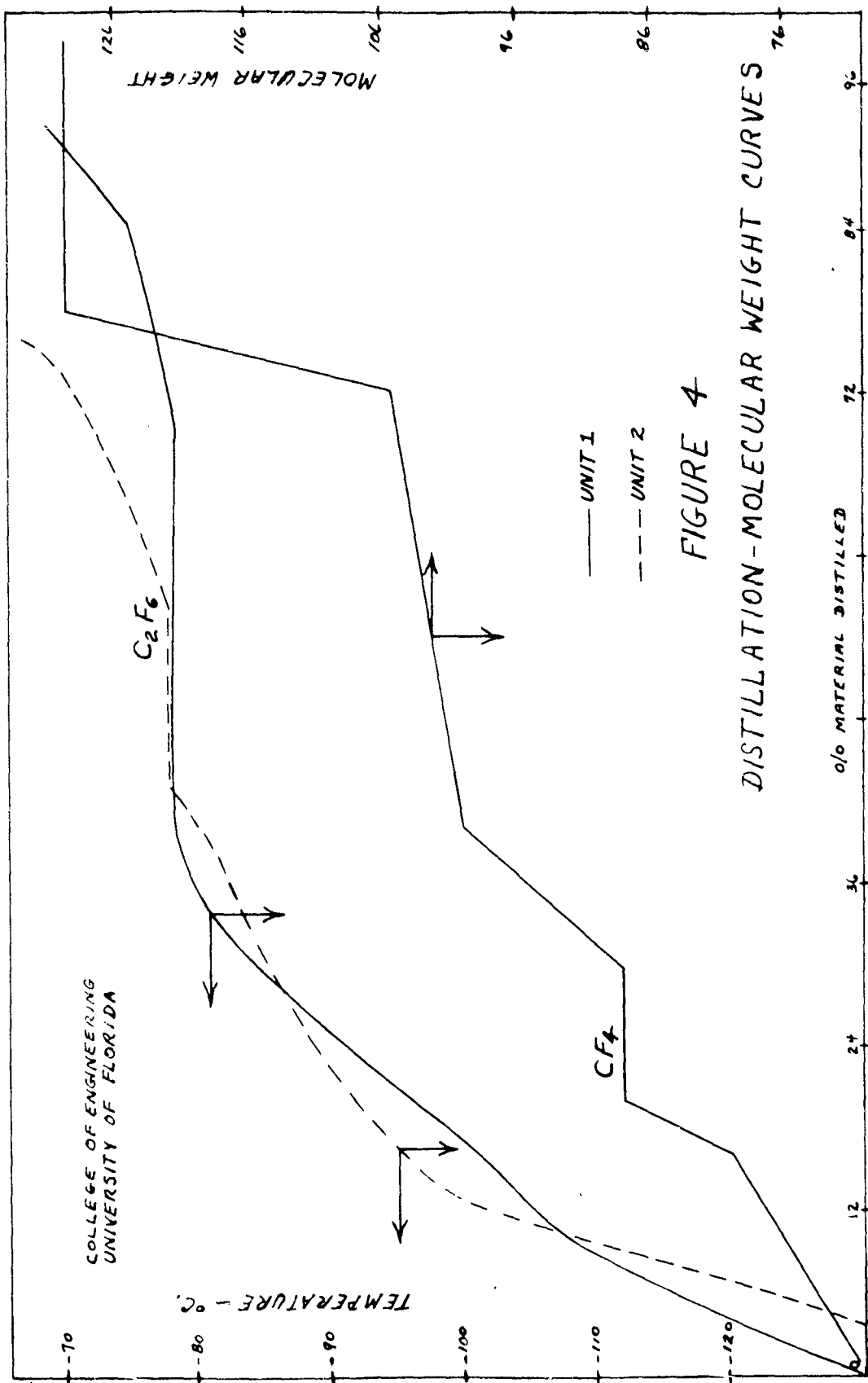


FIGURE 4
DISTILLATION-MOLECULAR WEIGHT CURVES

It is common in this type of electrochemical process for a large quantity of numerous intermediates to accumulate before isolatable products are evolved. Overall, the 32% not accounted for, can be assumed to have been present in the solution that was discarded.

The discrepancy in the rate of product collection in the liquid air cooled traps cannot be accounted for on the basis of the limited data obtained in this trial operation.

A simple relation between the boiling points, molecular weights, and quantity of material by weight is found in Figure 4. It is assumed empirically from the information and the starting materials that the majority of this product is NF_3 , CF_4 , C_2F_6 and possibly some higher boiling materials.

The bulk of the high boiling material is presumed to be $(\text{C}_2\text{F}_5)_3\text{N}$,
B.p. 70°C .

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