recommended that shutdown and flushing of fluorine lines precede repair operations whenever possible.

3. Avoid development of explosive gas mixtures within the cell. All sources of air leak into the cathode gas space must be eliminated. Fluorine pressure should not be allowed to exceed 4 inches of waterhead. Correct barrier voltage must be maintained. Flushing of gas spaces with inert gas must precede starting and immediately follow shutdown of cell. The cell must be allowed to stand 24 hours with inert gas in anode and cathode chambers before the lids are removed. Explosion vents must be adequate and in good order.

Development and use of 600-ampere

FLUORINE CELL

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THE generation of fluorine gas by electrolysis of KHF$_2$ has been the subject of a number of papers in the literature, and several cells for the purpose have been described. The earliest successful unit was that of Argo *et al.* (*1*), which was modified by Simons (*3*) using an open-type cell with a single centrally located anode surrounded by a copper skirt to capture the fluorine as it was liberated. This design included a reversing switch to correct momentary polarization by passing current through the cell in the opposite direction; it represented one of the most practical sources of fluorine at the time work was begun in this laboratory.

THE design and operation of a 600–800 ampere high temperature fluorine cell are described. The cell contains about 100 pounds of KHF$_2$ as electrolyte and is constructed of Monel, steel, and copper. The total weight of the cell is about 400 pounds, and it occupies about 2 × 4 feet of floor space. The electrolyte temperature is kept constant by jacketing the cell with a bath. The bath is provided with a water-cooled reflux condenser. The life of the cell without repair is determined chiefly by the anode life and averages 200,000 ampere-hours. Operation results in the conversion of KHF$_2$ to KF. Regeneration is accomplished by the batchwise introduction of gaseous hydrogen fluoride through inlets for the purpose.

by polarization, an automatic depolarization device was tried which momentarily reversed the flow of current when the voltage on a given cell rose because of polarization. This did not, however, noticeably increase the effective output.

It was discovered, however, that a much more effective depolarization technique consisted of raising the applied voltage until normal current flow was re-established in spite of the polarized condition. This required voltages as high as 30 volts, and depolarization was indicated by a sudden increase in the current through the cell. The circuit was then broken, the voltage restored to normal, and the cell would operate at the rated voltage when it was again connected. As long as the anode was in usable condition, this technique was found to be effective and solved the polarization problem satisfactorily.

Initially a bank of eight such cells was constructed without major modification of the original design. The anode was attached to the central lead of a commercial spark plug, which was in turn inserted through the top of the skirt, so that replacement of anodes was a relatively simple matter. To avoid disconnecting the fluorine line, the skirt and anode were fixed, whereas the cathode, electrolyte container, and an appropriate electric furnace were mounted on a movable counterbalanced platform. These cells employed an electrolyte of approximate composition KHF$_2$ and operated at about 275° C. Regeneration was batchwise direct from a cylinder of anhydrous hydrogen fluoride gas.

The electric controls included suitable reversing switches, and, since the output from all of these units passed into a common manifold, it was necessary to provide electrical insulation in the tubes leading from each individual fluorine compartment. Portland cement was the most desirable insulating material then available. In operation, maximum current was in the neighborhood of 10 amperes at a voltage drop of approximately 7–8 volts. When the cell was polarized, this rose to as much as 13 volts and was accompanied by a drop in current to almost zero.

In an effort to overcome the interruption to production caused

In connection with the production of fluorocarbons in this laboratory (*2*) a more reliable and larger source of fluorine became necessary. This involved the choice of cell type. Successful units have been reported using various electrolytes and anodes operating from below 0° C. to the so-called high temperature cell described. One of the more popular working temperatures was at or near 100° C. where water could be used as a heating or cooling medium.

However, the high temperature cell was chosen for use in this laboratory because of its flexibility, low cost, and safety. The nature of the electrolyte was such as to permit operation over a relatively wide temperature range without difficulty. Although the electrolyte normally melted at about 250° C. in equilibrium with an atmosphere containing hydrogen fluoride, the cell remained operable as hydrogen fluoride was removed through electrolysis or evaporation at temperatures as high as 320–340° C. If too much hydrogen fluoride was added to the electrolyte at a given temperature, the excess boiled out as a gas. The melt possessed the added advantage, from the point of view of safety, of freezing quickly if it escaped from the cell. Once solidified, it could be handled with a minimum of danger to laboratory personnel.

The use of graphite for anodes, at that time believed impossible in the low temperature cells, offered the further advantage of low

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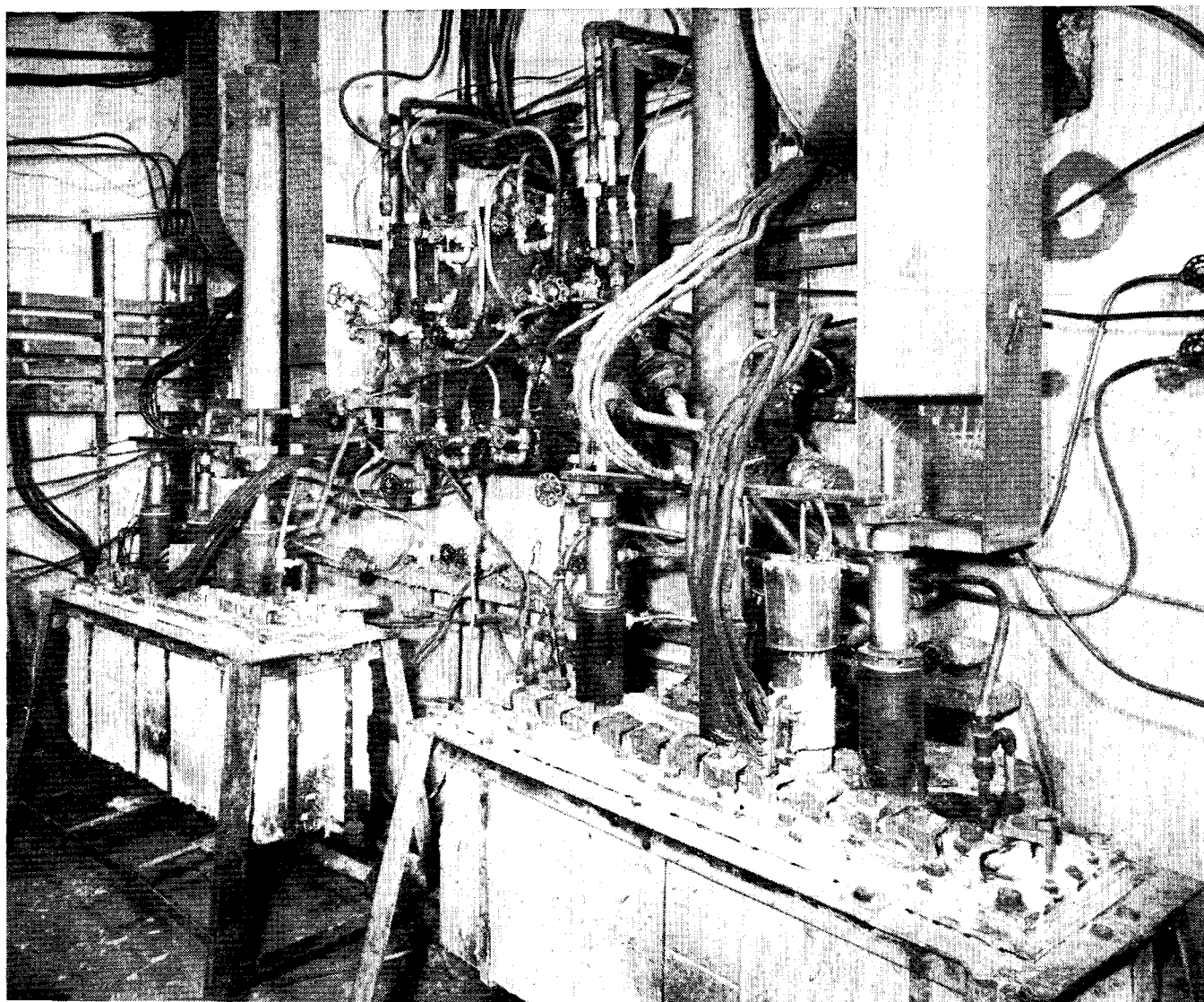
Close-up of Cells in Operation Showing Electric Leads, Insulators, Gas Lines, and Valve Board, with Sheet Metal Cover over Pop Valve on Cell 4 to Prevent Splashing

cost for replacement and removed the difficulty due to accumulation of fluoride sludge from the consumption of nickel anodes. All of these considerations resulted in the choice of the high temperature cell; despite significant developments in the lower temperature types, the authors believe that the high temperature modification still possesses a number of advantages.

600-800 AMPERE CELL

As a result of experience with several designs of intermediate units, which will not be described here, a satisfactory cell capable of delivering 0.75 pound of fluorine per hour was constructed (Figure 1). The cell as connected for use is schematically represented in Figure 2, which shows electrical and piping connections as well as the gas burners and heat insulation, angle iron support, and condenser. In this unit direct gas heating was selected as being more economical, but a thermostatic liquid was interposed between the gas flame and the electrolyte box to avoid local overheating and corrosion of the electrolyte container. Diphenyl oxide was selected as a thermostatic liquid since it boils at 259° C., which is above the melting point of the electrolyte and at the lower end of the operating range of such a cell. When the cell was not in operation, heat was supplied to maintain the electrolyte in a liquid condition. During operation, however, the cell did not require auxiliary heat, and the liquid served to keep the temperature within the desired range. The diphenyl oxide vapor was liquefied in water-cooled condensers and returned to the cell.

In the following description of the cell construction the numbers refer to Figure 1. The outermost or coolant box 1 and 18 was of welded steel construction and contained approximately 6 gallons of liquid. It was provided with suitable drain and filling pipes 17 and 71 and with elbows and flanges 33 and 34 for attaching the reflux condensers. The condensers consisted of six $3/4$-inch copper tubes in a 5-foot long water jacket and were constructed entirely of copper and brass.

Electrolyte container and cell cathode, 11, was a narrow Monel box, $4 \times 16 \times 34$ inches provided with Monel flange 5 near the top. All seams were electrically welded and tested for gas leaks. The flange on the Monel box rested on the similar flange, 21, of the iron box below, and thereby sealed the diphenyl oxide compartment. A clamp flange, 4, of steel rested on the Monel flange and was provided with clamp studs, 25, near the inside periphery, which served to bolt down the cell head when in place.

The term "head" was a convenient designation for the assembly which consisted of the anode, skirt, diaphragm cage, and all auxil-

iary equipment in one integral unit. It could be lifted out of the electrolyte container in one piece and normally was the only part of the cell to require maintenance or repair work. All parts above and within the electrolyte box were, when assembled, attached to a single plate of copper called the headplate, 2. When lowered into place, this rested on the machined top edge of the Monel box using gasket 3 and was held securely in place by the headplate clamps, 24.

Symmetrically arranged on the headplate were three cathode tubes, 41, of 3-inch i.d. copper tubing; these supported the skirt, diaphragm cage, and anode assembly by means of clamp rings 36, 37, and 39. Tetrafluoroethylene polymer (Teflon), 38, 40, was used as packing and for electrical insulation between the cathode and skirt assembly.

On early models the cathode tubes were shorter and reduced the over-all height of the head proportionately. It was found, however, that the packing could not withstand the higher temperatures near the headplate; in later models, consequently, it was raised to take advantage of air cooling. In addition, the annular space between the cathode tube and the skirt tube was continuously swept with anhydrous hydrogen fluoride to provide a protective blanket below the packing and insulation. The unsymmetrically located fourth tube, 63, on the headplate served as an outlet, 54, for the gases from the cathode (hydrogen and entrained hydrogen fluoride) and carried a pressure release valve, 55–62.

The skirt, 8, which projected about 1 inch below the surface of the electrolyte during operation, served to divide the anode gas compartment from the cathode gas space and, in addition, supported the diaphragm cage. Although the cell did not require a wire screen diaphragm below the skirt to separate the gases as they were generated at the anode and cathode, the cage assembly, 14, 15, and 20, was provided to confine fragments from broken anodes and prevent them from short-circuiting between anode and cathode. This could result in an arc which would puncture the cathode box and require a major repair. The bottom plate, 14, was the same width as the skirt and served to prevent any hydrogen liberated at the bottom of the cathode box from rising into the fluorine compartment and reducing the cell efficiency.

The skirt and cage assembly were $^3/_{16}$-inch sheet Monel electrically welded, and the anode tubes, 42, were of 2-inch Monel tubing. The end tubes were provided with specially insulated packing glands, 43–46, through which the anode bar risers passed and which served to support the anode assembly. The central tube, 53, served as outlet for fluorine and entrained hydrogen fluoride. In this case also provision was made for a protective hydrogen fluoride sweep in the annular space between the anode lead and the Monel tube.

The anode assembly consisted of two $^3/_4$-inch diameter copper anode bar risers, 52, carrying the anode bar, which were supported in packing glands and were insulated from the skirt by Teflon packing and a mica sleeve in the packing nut, 46, above the polymer. The anode bars which proved most satisfactory, 7, were $1^1/_4 \times 2 \times 24$ inch copper and carried sixteen 1-inch-diameter carbon rods, 13, of Stackpole Carbon Company's grade K-1 carbon. In assembling the anode bar, the carbons were drilled and tapped to fit the $^1/_4$-inch copper dowel pin, 12, after which the upper portion of the carbon was machined to a drive-fit in the sockets of the bar. The bar was bored 0.003 inch smaller than the carbon, and the carbon and copper dowel pin were then driven into the socket, the dowel pin fitting into a centrally located hole through the anode bar. The upper end of the dowel pin then projected through the anode bar and was silver-soldered at the top to ensure a durable electrical contact. When first used, it was customary to bore the sockets in the anode bar to a diameter of 0.960 inch. After carbon renewal, which required remachining to clean out corrosion products, etc., this diameter was increased to a maximum of 0.990 inch before the bar was replaced. As a rule a bar could be used four or five times.

The anodes were estimated to have an effective area of about 2 square feet when the electrolyte was maintained at its normal level about $1^1/_2$ inches below the bottom of the anode bar.

Regeneration was accomplished by introducing hydrogen fluoride through the Monel tubes, 19, at each end of the headplate. Auxiliary equipment included the thermocouple well, 35 and 16, and an inspection port, 68, 69, and 70.

The schematic drawing (Figure 2) shows the piping and electrical connections required for operation. The anode connection is made directly to the flat copper bus bar between the anode bar risers; the cathode connection can be placed at any convenient point. It is shown as a strap around the hydrogen outlet tube.

All sweep and regeneration lines were soft-drawn $^1/_2$-inch copper tubing and were provided with flanged insulators, using Teflon where the insulation was in contact with the gases and fiber under the bolt heads. Hard-drawn $^3/_4$-inch copper tubing was used to connect the cell to condensing units maintained at $-70°$ C., where hydrogen fluoride was liquefied, continuously withdrawn, vaporized, and returned through the sweep lines to the cell. The hydrogen fluoride from the fluorine side entered the anode tubes as shown and formed a continuous sweep down the annular space surrounding the two anode risers. Similarly, hydrogen fluoride from the hydrogen side swept the three spaces below the cathode tube packing assemblies. The tees shown in the gas sweep lines were connected to an independent source of anhydrous hydrogen fluoride, and a slow supplementary sweep was continuously added. If the hydrogen fluoride in the permanent gases was not condensed, the sweep could be provided entirely in this manner. The regeneration line, connected to four Monel tubes in the corners of the headplate, must be provided with a vent to keep electrolyte from sucking back and plugging the lines.

The hydrogen fluoride sweep was found to be a necessity for successful operation. In addition to supplying much hydrogen fluoride to the electrolyte during electrolysis and protecting the packing glands, it also provided hydrogen fluoride which combined with and liquefied any electrolyte which had splashed up and solidified on the cooler parts of the head; this greatly reduced short circuiting and sludge accumulation.

The valves which proved most successful for handling hydrogen fluoride were ordinary all-brass globe-type water valves. It was necessary to lubricate the threads with perfluoro oil or similar material to prevent sticking. The life of a valve in such service was from six months to a year, and no commercially available device was found more satisfactory. The valves handling fluorine were of the same type but had a considerably shorter life and tended to stick despite lubrication. All connections were therefore made with flare fittings to permit easy and rapid substitution.

Disassembly was easy, the entire head lifting out as a unit weighing about 75 pounds. By removing the Monel cage, the anode bar assembly was readily dropped out and could be replaced. This entire operation, including reconnection, could be carried out by two men in 20 minutes.

PERFORMANCE DATA

NORMAL OPERATING PROCEDURE. The units operated on a continuous schedule. The following data outline the performance of a cell:

Current, amperes	600–650
Current density, amperes/sq. ft.	300–325
Total voltage drop, anode to cathode, volts	6.0–8.0
Voltage drop, anode to skirt, volts	4.0–5.0
Voltage drop, skirt to cathode, volts	2.0–3.0
Electrolyte temperature, ° C.	260–310
Water (15° C.) through condenser, gal./hr.	10

CURRENT EFFICIENCY. Current efficiencies were normally of the order of 90–95% when a unit was placed in operation. With aging of the anode assembly, however, the over-all current efficiency dropped, frequently reaching as low as 55% before the cell was rebuilt with a new set of anodes. This drop was due partly
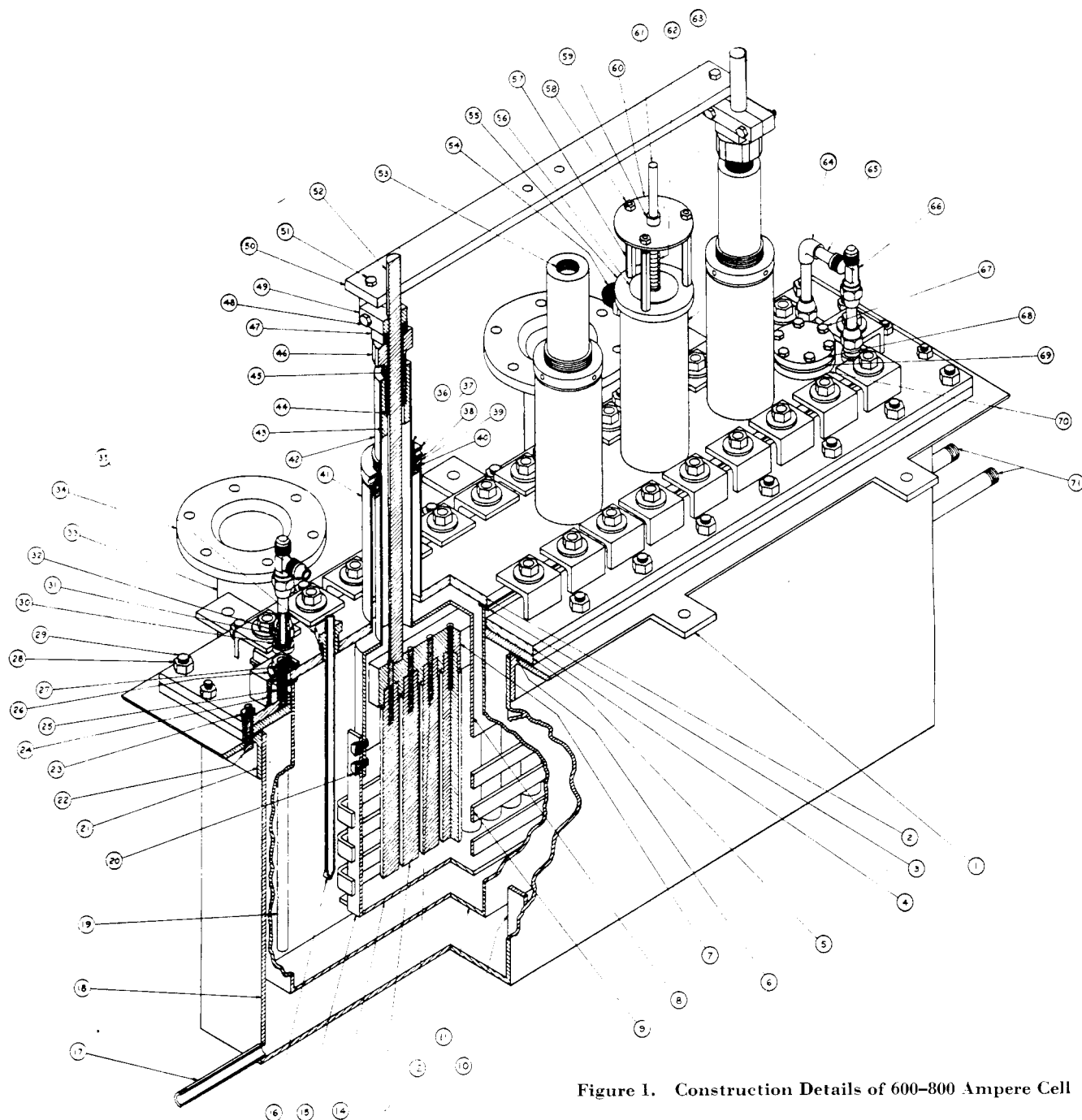
Figure 1.   Construction Details of 600–800 Ampere Cell

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1. Lug	19. Regeneration tube	37. Lock ring	55. Valve seat plate
2. Headplate	20. Monel bolt	38. Packing	56. Valve plunger
3. Headplate gasket	21. Coolant box flange	39. Cathode riser ring	57. Spacer
4. Clamp flange	22. Flange bolt (end)	40. Packing	58. Spacer nut
5. Cathode box flange	23. Flange nut (end)	41. Cathode tube	59. Bushing
6. Coolant box gasket	24. Headplate clamp	42. Anode tube (diaphragm riser)	60. Guide plate
7. Anode bar	25. Clamp stud	43. Anode packing gland	61. Valve stem
8. Skirt	26. Clamp washer	44. Packing	62. Valve spring
9. Diaphragm cage bar	27. Clamp nut	45. Insulation sleeve	63. Hydrogen outlet tube
10. Coolant box rib	28. Flange nut (side)	46. Anode packing nut	64. Elbow
11. Cathode box	29. Flange bolt (side)	47. Spacer	65. Nipple
12. Copper dowel pin	30. Packing gland	48. Clamp block bolt and nut	66. Tee
13. Carbon	31. Packing nut	49. Clamp block	67. Inspection port bolt
14. Diaphragm case bottom	32. Packing	50. Anode bus bar	68. Inspection port cover
15. Diaphragm case end	33. Condenser elbow	51. Anode bus bar bolt and nut	69. Inspection port gasket
16. Thermocouple well	34. Condenser flange	52. Anode bar riser	70. Inspection port ring
17. Coolant drain pipe	35. Thermocouple nut	53. Fluorine outlet fitting	71. Coolant overflow
18. Coolant box	36. Diaphragm clamp ring	54. Hydrogen outlet	
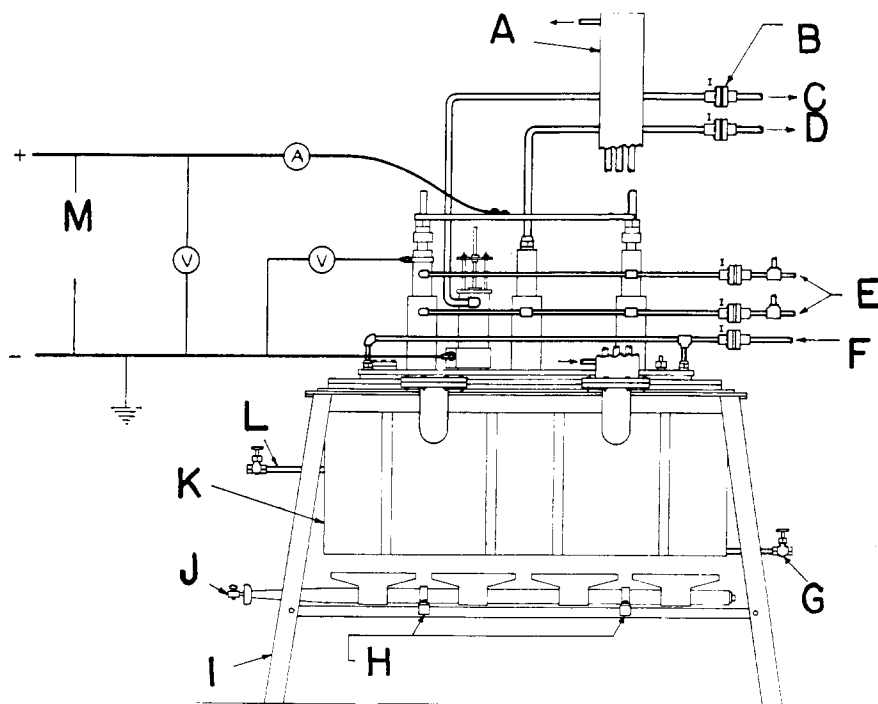
Figure 2. Piping Arrangements on 600–800 Ampere Cell

A. Tubular condenser for refluxing coolant
B. All tube insulators (designated by *I*) are flanged, using Teflon insulation between flanges; bolts are insulated with fiber bushings; only Teflon can come in contact with the gases
C. H₂ and HF out
D. Fluorine and HF out
E. Fluorine side: HF gas sweep to protect packing H₂ side
F. Regeneration line: supplies HF under 5–10 lb. gage pressure; provided vent to prevent sucking back
G. Coolant drain
H. Electrically insulated mount for burners
I. Supporting frame
J. Gas burner: four No. 60-G Johnson Longways burners and 1½-inch Johnson air-gas mixer and valve. Gas supply: ¾-inch pipe size required
K. Outer steel box insulated on sides and ends only with 1½-inch sheet magnesia, supported by ⅛ × 1 inch steel straps
L. Coolant fill and overflow
M. Source of 800-ampere 30-volt (maximum) direct current; one side grounded; normal voltage, 8–10 volts

to polarization, which became increasingly troublesome as the anode aged, and partly to an undetermined phenomenon, probably at the anode, which reduced the output without affecting the current density.

ANODE ASSEMBLY LIFE. The limiting factor in the operating life of a unit was almost always deterioration of the contact between the carbons and copper anode bar. Despite the precautions taken to provide a sound mechanical and electrical contact, the resistance at this point increased with age with an accompanying decrease in output and efficiency. It was necessary, therefore, to renew the anode bars periodically by removing the carbons, remachining the sockets, and inserting new carbons. A number of efforts were made to overcome this trouble but were not completely successful. Using an anode assembly of this type, the following data were typical of experience over a period of about six months: The average life of an anode assembly was 187,000 ampere-hours, and the life of the oldest when removed was 241,000 ampere-hours. These figures were for units in production, where a low cell efficiency was quickly apparent and replacement was immediately made. The units were operable for much longer periods, but the output would be low during the latter part of the run.

ANODE BREAKAGE. With the copper studs, anode breakage from explosions in the cell or normal operation was sharply reduced. In general, about one in fifteen anode assemblies had a broken carbon when removed.

LIFE OF CELL PARTS. Exclusive of the anode assemblies, the all-Monel unit was little troubled by corrosion. Earlier models with copper parts exposed to the electrolyte were not satisfactory in this respect. The headplates and parts above them were largely copper but gave little evidence of corrosion; Monel was the only satisfactory material for the skirt and riser tubes in contact with fluorine. The estimates of the life of the component parts were as follows: cathode boxes, skirts, and cages, 18 months; copper head parts, 1 year; and skirt riser tubes, 6 months. In most cases the life could be prolonged by increasing the gage of metal. Destruction of cages by arcing is not included in the figures given. Occasionally when a carbon broke it fell in such a way as to strike an arc between the anode and the cage, and a portion of the latter had to be replaced.

SLUDGE. The accumulation of sludge or insoluble metallic fluorides in the electrolyte was not a serious problem, provided all the metallic parts exposed to the melt were of Monel metal. Copper was not satisfactory from this point of view and was replaced throughout the unit during the development. Adequate clearance (1.5 inches) was provided between the bottom of the cathode box and the bottom of the cage, so that the cell required cleaning infrequently. It was the custom to remove such accumulation from the bottom of the cathode box with a Monel strainer whenever an anode assembly was replaced.

A more serious problem was the accumulation of corrosion products on the copper anode bars. The precise composition of the corrosion products was not determined, but the hydrogen fluoride sweep did not serve to keep the bar clean. As a result there was frequently a heavy crust on the bar when it was removed, and it was necessary to provide maximum clearance between the bar and the skirt without impairing cell efficiency. The standard skirt was 2½ inches wide inside, providing ⅝ inch between the anode bar and the inside of the skirt. No operating difficulty was experienced with the skirt close to the cathode (⅝ inch), even though the voltage drop from cathode to skirt was relatively low.

HYDROGEN FLUORIDE CONTROL. On the basis of the earliest cells the hydrogen fluoride concentration in the electrolyte had been thought to be practically constant, and the presence of suspended solid matter in the melt had been assumed to be potassium fluoride precipitating out as the hydrogen fluoride was consumed. Working on this assumption, regeneration (or addition of hydrogen fluoride) was not carried out until the melt failed to conduct current as a result of the presence of too much solid. During such

operation the temperature of the electrolyte was allowed to rise and sometimes reached 360° C. When the larger unit was placed in service, however, it was found that operation was not practical above 310° C. because of the limited capacity of the condenser for diphenyl oxide vapor. It was also found that, as hydrogen fluoride was removed during fluorine production, the voltage, as well as the temperature went up; this resulted in decreased power efficiency. Therefore, it was not practical to operate the cell for an extended period without replacing the hydrogen fluoride which had been consumed. Regeneration was placed on a regular schedule calling for 2–3 pounds of anhydrous hydrogen fluoride for every 2000 ampere-hours of operation.

The protective hydrogen fluoride gas sweeping, previously mentioned in connection with the design of the insulated packing glands, was found to be important from the standpoint of regeneration. This sweep, part of which was continuously supplied from cylinders of the gas, was found to be absorbed in the electrolyte and to supply part of the regeneration. The condensing system thus served as a reservoir of liquid hydrogen fluoride which was continuously vaporized and swept through the compartments above the electrolyte. As the electrolyte lost hyrogen fluoride because of electrolysis, the sweep gas from the reservoir dissolved in it, providing part of the regeneration required. In addition, the condensing system made it possible to store temporarily excesses of hydrogen fluoride without overloading the electrolyte.

It was impossible to accomplish all of the necessary regeneration by adding hydrogen fluoride to the sweep lines, probably because of the relatively small area of exposed melt and the lack of stirring. As a result the cell required further regeneration and hydrogen fluoride was added through the regeneration lines at the bottom of the electrolyte. Since the amounts added under the regular schedule were in excess of the requirement at the time of regeneration, a portion of the gas was vaporized and stored in the condensing system. It was therefore unnecessary to add significant amounts of hydrogen fluoride to the sweeping lines from an independent source, since the normal regeneration procedure constantly replenished the supply in the condenser. It was believed also that addition of hydrogen fluoride to the bottom of the melt served a useful function in periodically stirring the electrolyte and equalizing the hydrogen fluoride concentration.

The exact mechanism of the change in conductivity and composition of the electrolyte was not determined. In an effort to explain the presence of suspended solids, as well as to determine the actual change in hydrogen fluoride concentration, analyses were made of samples of the electrolyte in operating cells. The samples were taken at the exposed surface of the melt before and immediately after routine regeneration and indicated a difference in composition of 1.3 to 1.4% hydrogen fluoride by weight. However, no analysis gave a hydrogen fluoride concentration below 25.6%, the theoretical value for KHF$_2$ corresponding to a mole fraction of 0.5. No further investigation was warranted, since operation was sufficiently flexible for practical purposes and since the cells could be studied only under production conditions when it was unlikely that equilibrium prevailed in the electrolyte.

Despite the wide range of practical operation, it was desirable to keep the hydrogen fluoride concentration high enough to permit operation at 260–270° C., since at this temperature corrosion was slowest and conductivity of the melt seemed highest, with less power loss as heat. It was also desirable to keep liquid hydrogen fluoride in the condenser to furnish an adequate sweep for the cell head. If this sweep became too slow, the splashing of electrolyte soon resulted in a short circuit and there was danger that a skin of solid electrolyte would form at the surface, trapping the gas below and resulting in explosions.

POLARIZATION. If the proper material for the anode were selected, these cells never exhibited permanent polarization. The units did, however, occasionally show such characteristics temporarily and could be restored to normal conditions very simply by shutting off the current for a few seconds, or, more directly and quickly, by increasing the applied voltage to a point such that the current came back up to 600 amperes or more. The polarization then appeared to break down, and, after the voltage was reduced, normal operation resumed. A great deal of work was done to determine the cause of this phenomenon, but it was never possible, while using the units for production, to identify it with any single characteristic. There was a strong likelihood that some relation existed between polarization and the age of the anode assemblies, since, in general, polarization grew worse with the length of time in service.

Although this tendency was annoying, it was not serious if the cells were operated in parallel from a current source of variable voltage. Successful series operation was somewhat more difficult; in practice, here the current source was a low-voltage direct current generator capable of supplying up to 30 volts output, controlled by the field excitation.

VARIATIONS. In some units a mechanical motor-driven stirrer was installed in the inspection port and resulted in much longer operation without polarization. It was noted that temperature control was easier and that a higher current density could be maintained, but the advantages were not sufficient to warrant the additional complications in maintenance.

The use of corrosion-resistant anode bars would be a major advantage, but since most corrosion-resistant alloys have higher electrical resistance, the additional heating more than overcame the benefit. A Monel bar was used, but the contacts deteriorated rapidly.

To improve the contact between the metal anode bar and the carbon, flat carbon sheets were used as anodes; within the limits imposed by the cell's width, however, the supporting bars could not be made heavy enough. The result was warping and breaking of the carbon. To have increased the width of the cell to accommodate heavier bars would have resulted in loss of the benefits to be derived from the small space separating the anode and cathode surfaces.

It was believed that a completely satisfactory anode assembly for such a unit could result only if the carbon-metal contact were not exposed to fluorine at high temperatures. This would mean that the carbon anode would be brought through the top of the fluorine compartment and the metal connected on the outside.

ACKNOWLEDGMENT

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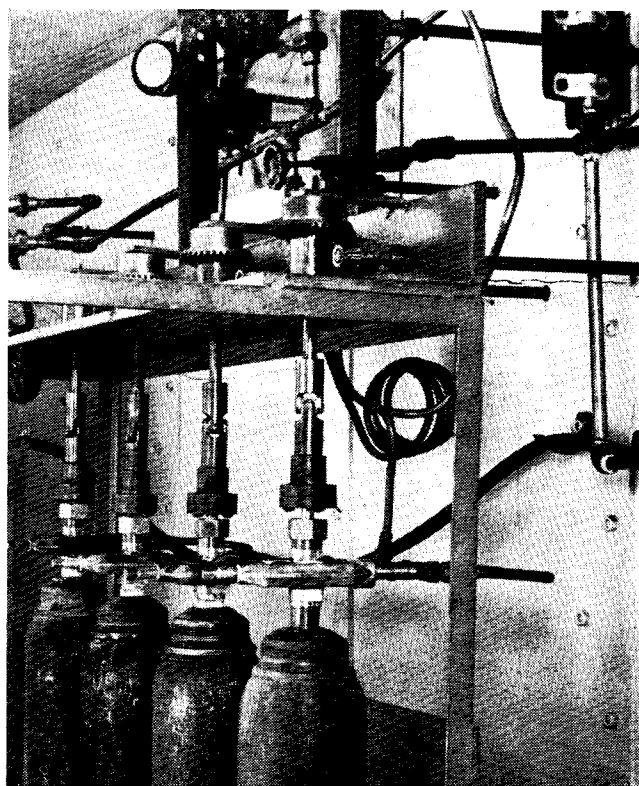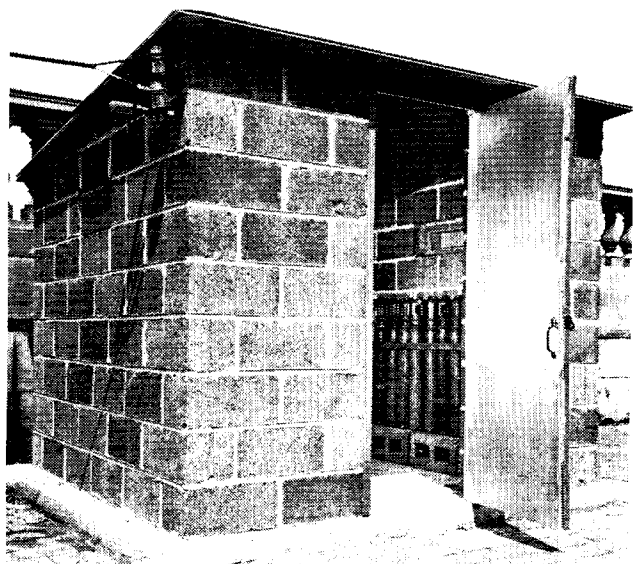
Loading the cylinders of fluorine behind a barrier of half-inch steel

FILLING COMMERCIAL CYLINDERS OF ELEMENTAL FLUORINE

PHOTOS COURTESY PENNSYLVANIA SALT MANUFACTURING COMPANY

Room on roof where cylinders are stored for a week to detect leaks

Closeup view of steel cylinders behind the steel barricade