

AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS

**FABRICATION OF EQUIPMENT FROM MONEL AND OTHER
MATERIALS FOR CORROSIVE SERVICE IN THE PRODUCTION
OF FLUORINE**

by

D. ROYSTON
N. W. D. CHRIMES

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ABSTRACT

Literature is reviewed on the fabrication of Monel, mild steel and brazed equipment for use in the production of fluorine. The results of an experimental study on the welding of Monel components for a fluorine cell experimental plant are presented and the fabrication of equipment used in the plant is described. The corrosion history of the equipment after three months of continuous operation is outlined.

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1. INTRODUCTION

Vessels and pipework used in the production of fluorine by the electrolysis of hydrogen fluoride have to contain anhydrous fluorine, hydrogen fluoride and molten potassium bihydrogen fluoride (KF.2HF). Mild steel is a satisfactory general material of construction; however, Monel is used in particularly corrosive environments such as the fluorine cell and the fluorine scrubber-tower. Corrosive attack on the vessels and pipework is generally directed at welds and care has to be taken to ensure that sound welds are made. This is most important with Monel where sound welds require careful preparation.

This report describes the experience gained with materials of construction, not only Monel and Monel welds but also mild steel and brazed joints, during the fabrication and operation of a 1500 A fluorine cell experimental plant. The plant was operated continuously for three months, producing and disposing of 2079 kg of fluorine by the processes shown in Figure 1. The plant has been described by Royston *et al.* [1975a] and the operation and performance have been described by Royston *et al.* [1975b].

2. LITERATURE SURVEY

The literature was surveyed to determine the procedures necessary to fabricate equipment used to process fluorine and fluorine compounds. Methods of fabrication of both Monel and mild steel were reviewed, although the major concern was the fabrication of Monel. The specifications of the nickel alloys and welding rods referred to in this report are listed in Table 1.

2.1 Equipment Exposed to Fluorine

2.1.1 Materials of construction

At ordinary temperatures and near atmospheric pressure, aluminium, brass, copper, magnesium, Monel, nickel and steel have a high resistance to attack by fluorine [Baxter 1952; Myers & DeLong 1948; Schmidt 1967]. Titanium has low resistance to attack [Schmidt 1967], whereas high silicon steels are subject to vigorous attack with pitting and corresponding high corrosion rates [Landau 1952; Myers & DeLong 1948]. To ensure corrosion resistance, Schmidt [1967] claimed that all metals which were to be in contact with fluorine must be radiographed to ensure that no inclusions, cracks, discontinuities or other imperfections were present. Any imperfection must be cut out and repaired to provide a homogeneous material. These comments indicate that imperfections provide sites for corrosive attack.

2.1.2 Fabrication by brazing and welding

Landau & Rosen [1951] suggested that, in soldering or welding vessels, measures should be taken to minimise the amount of flux allowed to remain in the pipe or vessel and, as far as possible, to remove flux prior to the use of equipment. This statement referred to fabrication in mild steel and the brazing or silver soldering of equipment. Schmidt [1967] stated that welding did not reduce the corrosion resistance of nickel or Monel if fluxes were either not used or completely removed. In addition, he stated that the primary objective in fluorine-tank fabrication was to achieve a smooth crevice-free interior. Poor seam welds which had flux and slag inclusions, pockets or bubbles, and oxidised surface flaking were considered particularly undesirable. For this reason, the quality of the weld seams for inlet and outlet connections was very important and it was good practice to X-ray all welds, as well as all stock material used in fabrication, to ensure good quality.

Checks and procedures to assess the quality of welds for use with fluorine were described by Downing [1951], Dykstra *et al.* [1955] and Clark [1960]. Clark described the fabrication of a USAEC 'E' type fluorine cell giving details of fabrication methods and mentioning the need for each weld to be X-rayed to ensure satisfactory quality. Dykstra *et al.* stated that stress relieving was required on all welded fabrications. Downing commented that the failure of the skirt to cover plate (Monel joined to mild steel) weld in fluorine-cell construction was frequent, and this weld had to be pressure tested for leaks before use.

In welding nickel, there is evidence that the use of a filler rod containing titanium (a deoxidising agent) produces welds which are less resistant to attack than welds in which parent metal alone had been used as the filler rod. Chilenskas & Gunderson [1965] carried out tests on the corrosion of nickel and nickel welds in high-temperature (~450°C) fluorine environments. Test pieces were made using nickel 200 plate alone, and nickel 200 plate welded using the tungsten arc inert gas (TIG) process with nickel 61 and parent metal as filler rods. The corrosion of the nickel 61 weld was very high by comparison with the weld prepared using parent metal, and higher than that of the parent metal itself. It was claimed that the high corrosion rate was due to the titanium content of the nickel 61 weld forming volatile corrosion products.

Similar corrosion tests undertaken by Smiley [1965] also indicated that parent metal welds gave greater corrosion resistance than those made with

nickel 61 filler metal. The nickel 61 welds showed signs of pitting and deep penetration in the weld areas, whereas no such attack occurred in samples welded with nickel 200 or 201 (parent metal).

2.1.3 Preparation before installation

Fluorine attacks organic materials and other contaminants, and so it is important that the components for use with fluorine are cleaned thoroughly before use. Landau [1952] stated that the reaction between fluorine and metals was limited if the surfaces were clean; however, contaminants (including surface moisture) give rise to ignition points. Fluorine escaping from a pipe can ignite with surface moisture leading to the ignition of the metal, producing an intense metal fire. Baxter [1952] stated that fluorine attacked most organic materials and grease or oil, and so they must be rigidly excluded from plant used to handle the gas. Fluorine fires can be initiated in metals by a patch of grease or even a set of finger prints. Similar comments were made by Schmidt [1967].

A specification for the cleaning of valves for use with fluorine was given by Trumbo [1970]. This pointed out the need for a contamination-free product and required valve assembly to be undertaken with gloved hands to avoid finger marks.

The CAPE packages of USAEC drawings for fluorine-production facilities carry notes listing the desired quality of the welds and materials [Kelly & Clark 1968]. These notes define the high standards of cleanliness required in the assembly of equipment and the quality of the welding.

2.2 Equipment Exposed to Hydrofluoric Acid

In the industrial handling of HF, mild steel is a common material of construction, and the silicon in the metal and the slag from welding are the major causes of corrosion. Heat treatment and radiographic inspection of welds are generally advised.

Thornton [1970], writing on HF alkylation plants, stated that it was well known that anhydrous HF will easily corrode metals having silicate slag inclusions that contain large amounts of silicon; in addition, all vessels routinely in contact with HF were specified to be heat treated after welding. Such vessels should also be 100 per cent radiographed for further assurance of quality, particularly for freedom from slag inclusions in welds and plates. No major repair welding should be permitted on such vessels without radiographing and heat treating the repair.

In New South Wales, the Department of Labour and Industry requires pressure vessels which are to contain HF to be designed to Class 1 pressure

vessel standards which includes radiographic examination of the welds. Experience with HF plants in New South Wales has demonstrated the need to eliminate slag inclusions from pipe welds in mild steel pipe as these lead to rapid corrosion and perforation of the weld.

Edwards [1961] examined the corrosive effect of aqueous HF solutions on Monel welds using Monel 60 filler wire in argon-arc welding, and Monel 130 and 140 electrodes in metal-arc welding. Both stressed and unstressed samples were subjected to the corrosive attack of 40 per cent HF at 70°C with 1 per cent oxygen purge. The Monel 140 weld corroded rapidly, Monel 130 and 60 were much less affected, and Monel 60 was the least attacked. The attack on the Monel 140 was attributed to the presence of a second phase of niobium. The Monel 130 and 140 electrodes are no longer available commercially having been replaced by the similar Monel 190.

Edwards also found that corrosion of the parent metal by HF in the vapour phase, under condensing conditions, was marginally higher than in the liquid phase. Stressing the test piece gave no statistically significant increase in corrosion rates under the conditions of the test. However, points of very high stress (e.g. punch marks) did give rise to local cracks and, in addition, multiple repairs to attain radiographic soundness could produce accelerated corrosion. Edwards mentioned that radiographic soundness was the ultimate criterion employed for the acceptance of welds for equipment used with hydrogen fluoride.

The stress corrosion of Monel in hydrofluoric acid was discussed by Copsen & Cheng [1956] who cited several cases where high stresses had provoked the corrosion of Monel. They suggested that the stresses could be relieved by heating the item at 600-650°C for one hour followed by slow cooling. They also pointed out that the most severe corrosion resulted from exposure to moist aerated hydrofluoric acid vapour.

The design of plants for handling hydrofluoric acid was discussed by Hill & Knott [1960]. They suggested that plants should be designed to allow the radiographic inspection of all welds. Failures in welds were found to be where cutting and rewelding had been required and where the original weld had had marginal defects.

2.3 Fabrication Using Nickel Alloys

2.3.1 Cleanliness of materials

Prior to each stage in the fabrication of equipment where welding or heat treatment is to be carried out, the materials must be rigorously cleaned. Contaminants containing sulphur or lead produce surface cracking

at high temperatures leading to loss of strength and high corrosion rates in nickel and nickel alloys [Phillips 1961; Wiggin Publication 312a].

Phillips [1961] stated that the removal of all machining lubricants, dirt, grease, oil, paint, temperature-indicating crayon, marking crayon and processing chemical residue was essential before starting to weld. Similar comments were also made by Hill & Knott [1960].

Wiggin Publication No.312a dealt with the annealing of materials; it discussed the sources of, and methods for the elimination of sulphur contamination in annealing furnaces as well as methods for cleaning and pickling nickel alloys. In addition, it stated the need to eliminate surface contamination from materials before fabrication. It advised that sulphur may be introduced in the form of sulphate salts and sludge remaining on the work from a prior pickling operation, or in oil and lubricants used for machining or other fabricating operations. All such contamination should be completely removed before heating. It also advised the removal of all marking paint; this type of paint frequently contains metallic sulphide pigments which can cause embrittlement of the painted areas.

Barnett [1959] also commented on the importance of avoiding contaminants in welding nickel and nickel-based alloys. He listed the following elements as deleterious: sulphur, lead, bismuth, boron, carbon, silicon and certain gases. He stated that small amounts of the first three elements and some heavy metals caused hot cracking during welding, stress relieving or service at elevated temperatures. Gases such as nitrogen and hydrogen had a high solubility in molten nickel-based alloys and could cause porosity in the solidified weld.

In addition to cleaning the metal surface free of contamination, it is essential after heat treatment and before welding to clean the thin dark-coloured oxide film from the immediate vicinity of the area to be welded. A hot-rolled plate which has been machined in preparation for a butt weld needs no further cleaning other than the removal of machining lubricants or cutting fluids [Phillips 1961]. If the weld is made with a covered electrode, the resulting slag will promote surface corrosion if it is not removed before any further heat treatment of the weld [Phillips 1961].

2.3.2 Welding procedures

Wiggin Publication No. 3314 advised that nickel alloys should generally be welded in the annealed or solution-treated condition and, although a small degree of residual cold-work should not cause problems, severely cold-worked material was liable to stress cracking during welding. It

also advised that direct current should be used for electric welding operations and, in metal-arc welding, the electrode should be positive. In addition, both manual and automatic procedures can be used together with gas welding, although the application of the latter process was limited. The publication stated that care should be taken to ensure that the appropriate electrode or filler metal was used in welding alloys of the same type, of dissimilar type, or to other materials.

These considerations were explained in greater detail in Wiggin Publication No. 3367c, which dealt with surface preparation, precautions against distortion and surface finish and its influence on corrosion and joint design. Among other methods, TIG, metal-arc and resistance welding processes were discussed in this publication.

Phillips [1961] outlined the welding procedures for nickel and high-nickel alloys. Details were given of the necessary preparatory cleaning, joint preparation, jiggling, finishing and cleaning. The importance of correct weld alignment and proper jiggling facilities to avoid stresses in the weld area was pointed out. The use of the TIG welding method was described. Improved results with hydrogen in argon as inert gas were reported; however, these beneficial results were to be seen mostly in single- or root-pass welds and higher hydrogen concentrations could lead to porosity. Porosity could be minimised or eliminated by using a near vertical angle of the torch (workpiece horizontal) to avoid inspiration of air. High purity of blanket gases was also important, and it was necessary to use as short an arc length as possible. In addition, the covering of the entire weld, both front and back, with inert gas was recommended to minimise the risk of porosity in the welds.

Wiggin Publication No. 3367c also noted that where welding was only possible from one side, the root run was of prime importance. In such cases, the root run was best made using the inert gas-shielded TIG process with proper backing bars. Details were given of the design of suitable backing bars; copper was suggested as the best material to use.

Welding Dissimilar Metals

Procedures for joining dissimilar metals such as Monel and mild steel were given in Phillips [1961] and, using more recent techniques, in Wiggin Publication No. 3489. Cleanliness of materials, joint preparation and other factors required for a sound weld are the same as for welding Monel. In welding Monel to steel, it is important to avoid dilution of the weld by iron as this can lead to cracking. Maximum

dilution by iron before cracking of the weld becomes a problem is 30 per cent iron in Monel 190, 25-30 per cent iron in nickel 61 and 10-15 per cent iron in Monel 60 welds. Maximum dilution is seen in the first weld, successive layers containing less iron.

In laying a surface coat of metal with gas or metal-arc, techniques have been used to minimise dilution of the weld by iron and to give a ductile deposit suitable for structures which have to be stress relieved. An initial layer of nickel 61 is deposited followed by layers of Monel 60 [Phillips 1961].

2.3.3 Welding processes, filler rods and electrodes

The TIG process eliminates the risk of slag inclusions in the weld and is preferred to metal-arc processes which use fluxed electrodes. In addition, it appears that certain alloying elements used in welding rods lower the corrosion resistance of a weld, and fluorine and HF corrode silicon-containing steel welds, titanium-containing nickel welds and niobium-containing Monel welds. It follows that these elements ought to be avoided in filler rods or electrodes for use with fluorine and fluoride-containing equipment. However, these elements may be necessary to obtain sound welds in terms of weldability of materials, weld porosity or other mechanical features, and their inclusion to give superior mechanical properties to the weld may outweigh the increased risk of corrosive attack.

The Composition of Standard Filler Rods

Monel 60 and nickel 61 filler rods are recommended for use with the TIG process when welding Monel 400 and nickel 200 and 201 respectively [Wiggin Publication No. 3367c]. These materials have the parent metal as a base but contain additions of titanium, silicon and aluminium. Barnett [1959] noted that welding rods for gas-shielded arc welding contained titanium, and that titanium deoxidation was used to provide welds without objectionable porosity and to allow the weld metal to flow well under the intense heat of the arc. Hinde [1958] pointed out the risk of porosity with argon-arc welds when a high titanium filler rod was not used, the porosity being due to a reaction between carbon and residual oxygen.

Welding Using Parent Metal

The welding of nickel presents welding problems very similar to those of Monel and the alloying additions in the filler rods used for these materials are very similar (see Table 1). It follows that welding techniques developed for use with nickel could also be applied to Monel.

Smiley [1965] reported a welding technique using the TIG process with filler wire swaged from strips of nickel 201 sheet (parent metal). He described the need for cleanliness of the workpiece, the pickling procedures to be adopted, and the welding technique which used a high heat input with a fast rate of travel. For example, arcs of 100 A for the root weld, increasing to 120 A for the face weld, were used when butt welding 76-mm nickel pipe. The rod was fed into the puddle in fast short jabs but the tip was never removed from the inert gas shield of the torch, thus reducing the possibility of oxide formation.

Joint bevel angles of 75 degrees and a 3.2-mm root gap were found to be the ideal conditions for a good weld. Both argon and helium torch and purge gases were employed in the tests; helium was found to be the best gas for both applications. A much higher back-purge flow was required for the root pass to prevent oxidation and to provide adequate cooling. Smiley, Brater & Pashley [1966] prepared a specification for this technique which is outlined in Section 2.3.4. As discussed earlier (Section 2.1.2), Chilenskas & Gunderson [1965] also reported that nickel welds prepared from parent metal has superior corrosion resistance compared with those made using the standard nickel 61 filler wire.

It seems reasonable to suppose therefore that, with care in weld preparation, sound welds may be obtained in Monel 400 using the TIG process with parent metal as filler rod.

2.3.4 Standards for welding nickel and Monel

Two specifications for welding nickel and Monel are discussed: the Union Carbide singular specification [Smiley, Brater & Pashley 1966] gave the procedure for TIG welding of low carbon nickel; the other, the UKAEA specification AESS 6026 [UKAEA 1965], dealt with the fusion-welded fabrication of nickel and nickel alloys. The first specification is presented for guidance only, the second has been used as the basis for a specification for the welding of nickel alloys at the AAEC Research Establishment.

Union Carbide Specification

This specification dealt with the TIG welding of low-carbon nickel plate in the range of thicknesses 3 to 11 mm for use in extremely corrosive environments.

The qualifications of the welder were defined and part of the specification dealt with the procedure for testing the performance of the welder. The specification defined and described the requirements of the base metal and filler rod (both nickel 201); the inert gas (helium 99.995 per cent

purity); the electrical characteristics (d.c., base metal positive); the position of the workpiece when laying the weld; the preparation of welds; the welding technique; the defects permitted in the weld; the cleaning of the weld; and the X-ray examination of the weld (to paragraph UW-51, Section VIII of ASME Boiler and Pressure Vessel Code 1971). Pre- and post-heat treatments were not required. A general section described the responsibility of the inspector.

The preparation for welding, including machining of the workpiece, was given in some detail. The cleanliness of the weld site was regarded as essential, and so procedures were described for the removal of dirt and the oxide film. It was noted that white cloth gloves were to be worn by the operator. The need for a uniform gap opening in the assembly before welding to avoid subsequent distortion was mentioned. The welding technique was also described in detail with descriptions of wire feedrate, character of the weld pool, care of the tungsten electrode, and the need for a draught-free working area.

This specification, by rigorously defining the cleanliness requirement of the work, the use of parent metal as filler wire, and describing comprehensively the use of the TIG welding process, sought to satisfy all the requirements for a sound weld.

UKAEA Specification AESS 6026

This specification related directly to Monel welding and dealt in great detail with all aspects of the fabrication of equipment in Monel (and other nickel alloys).

The types and acceptability of materials and their marking procedures were given. The cleanliness of materials was emphasised as was the need for sulphur-free environments in heating furnaces. Descaling and pickling procedures were given. The quality of welding equipment and operators was set out as well as the type and treatment of filler materials to be used. The welding techniques, inspection and testing procedures were outlined, and radiography was used to determine the quality of welds (details of these procedures are given in Section 3.1.1). Finally, details were given of the procedures for marking and packing the finished products.

3. EXPERIENCE WITH MONEL COMPONENTS

3.1 Experimental

Monel was used in the fabrication of the cell and the fluorine scrubber tower for the 1500 A fluorine cell experimental plant. The literature survey showed that welding Monel required care and, when using

it in fluorine environments, it would be desirable to avoid impurities in weldments. To gain experience of welding Monel, an experimental program was carried out using Monel 400 plate in configurations similar to those required in the fluorine cell. Most of the welds were made using the TIG process with filler rods of parent metal, Monel 40 and Monel 60. The first two materials are similar in composition, whereas the third contains titanium as a deoxidant and is recommended by the manufacturer for the welding of Monel 400 by the TIG process. For the thicker plate (9.5 mm), the TIG process was used for the root run with the metallic-arc process being used for subsequent runs.

3.1.1 Procedure

Several joint configurations for the principal components of the fluorine cell warranted investigation (Figure 2).

All the joints, except that of the skirt to cover plate, were of Monel 400. The skirt and cover plate were respectively Monel 400 and mild steel. A test sample was based on each of these to produce a welded joint approximately 15 cm long. Before welding, the material was annealed and cleaned according to the following procedure:

1. Bend test pieces (where necessary).
2. Machine weld preparation (use low sulphur lubricants; no grinding allowed; filing with new clean file permitted).
3. Shot blast.
4. Pickle, rinse with cold water and dry.
5. Anneal (no sulphur contamination).
6. Shot blast.
7. Pickle, rinse with cold water and dry.
8. Weld (handle with gloves in clean conditions for this and subsequent operations).

Initially, welds were made with a parent material (Monel 40 and Monel 60), but subsequently the use of parent material was discontinued because it is similar in composition to Monel 40 (Table 1). After welding, samples were radiographed and a section was taken from each for metallographic examination.

Welding acceptability standards were, in general, based on those given in UKAEA Standard Specification AESS 6026 [UKAEA 1964]. These are summarised below:

Unacceptable defects

- (i) Cracks.
- (ii) Lack of penetration.
- (iii) Lack of fusion.
- (iv) Undercutting.
- (v) Worm holes.
- (vi) Any linear defects.
- (vii) Angular inclusions.
- (viii) Surface defects which reduce wall thickness or cause stress concentration.
- (ix) Porosity and spherical inclusions in excess of dimensions given in subsection (b), second column.

Porosity and spherical inclusions

Thickness of Parent Material	Maximum Dimension of Defect	Type and Frequency of Defect
1/8 in.(3.2 mm) & under	No definite limits of groupings can be given for this range of thickness and, although a defect free weld is generally expected, each case is considered separately	
Over 1/8 in. (3.2 mm) up to & including 5/16 in.(8 mm)	1/32 in.(0.8 mm) dia.in any portion of the weld	Spherical inclusions and porosity not larger than the limits stated and providing that no more than 12 defects exist in any one length of 6 in.(150 mm) of weld and that no more than 5 such defects are grouped within any one in.(2.5 mm) of weld length
Over 5/16 in. (8 mm) up to and including 1/2 in. (12.5 mm)	3/64 in.(1.2 mm) dia.in any portion of the weld	
Over 1/2 in. (12.5 mm) up to & including 3/4 in.(19 mm)	1/16 in.(1.6 mm) dia.in any portion of the weld	
Over 3/4 in. (19 mm)	Each case considered separately	

Welding Procedure

All welding was done with a Secheron Arcifix 320 unit, the majority of the welds being carried out by the TIG process (direct current, straight polarity). Where practicable, a backing supply of inert gas was used and the torch nozzle size was also selected to give adequate coverage of gas and prevent ingress of air. Welding-grade argon was used for all TIG welds, but additions of 5 per cent hydrogen were made for single-seam welds with the aim of reducing porosity. All the welds, except those of

the tank corner and skirt to cover plate, were made using the TIG process alone. For the corners, the TIG process was used for the root welds with the subsequent runs being carried out by the manual metallic-arc process with the electrode positive and using Monel 190 electrodes. For the skirt to cover plate weld, the metallic-arc process alone was used. Before welding, all joints were preheated to about 100°C using an oxyacetylene flame.

3.1.2 Results

Welds made using Monel 60 filler rods were generally less susceptible to porosity than those made with Monel 40 filler rods or with strip cut from parent material. Weld defects were almost entirely confined to gas defects. In the majority of cases these were spherical, *i.e.* 'pores', but some elongated defects known as 'worm holes' were occasionally seen. In some locations, the design of the component caused practical difficulties by restricting access to the weld, *e.g.* the skirt corner weld or where the welded seam changed from one type to another, *e.g.* the corner fillet to butt weld.

3.1.3 Conclusions and recommendations

The welds prepared with parent metal and Monel 40 were unacceptable owing to the defects which were formed. Monel 60 filler rod was recommended for use with the TIG process in the fabrication of the 1500 A experimental fluorine cell and fluorine scrubber tower, for the root runs of composite welds and where the TIG process alone was to be used. The recommended shielding gas was Ar with 5 per cent H₂.

3.2 Fabrication of the Fluorine Cell and Scrubber Tower

3.2.1 Cell tank

Construction

The inner cell tank was cuboid, of 1 m side, with the open top framed by a 75-mm wide flange (Figure 3). After fabrication, the inner tank was covered with a jacket for cooling water and this was constructed from 16-gauge (1.6 mm) Monel 400. The inner tank was formed from three pieces of 9.5-mm thick Monel 400 plate. One piece was shaped to form the base with two sides and the integral flanges; the other two sides with integral flanges were made from the remaining plate. The pieces were welded together with two vertical welds and one horizontal weld on each side. Holes were bored for the twelve cooling tubes which were passed through the tank and then welded in place. Joint preparations and welding of the inner Monel tank followed the techniques developed in the experimental program. The TIG process was used with Monel 60 filler rod for the sealing run, and Monel 190 with metallic arc for the subsequent runs.

details of the weld preparation and procedure are given in Table 2.

Examination and Repair

(a) Sealing Run

The welded joints were first submitted for radiographic examination with only the sealing run of each joint completed. When the sealing runs conformed to the requirements of the specified code [UKAEA 1964], the welded joints were completed and the vessel was submitted for final examination and acceptance.

The partially completed welded joints showed the presence of gas pores in isolated areas of each weld. Minor tungsten inclusions were also present in some vertical welds. Gas pores and inclusions larger than the minimum acceptable size (Section 3.1.1) were marked for repair and subsequent re-examination. Each weld contained a number of small isolated areas for repair.

The defects most difficult to locate and repair were generally situated in the corner areas of the flange welds and at the junctions of the vertical and horizontal welds. The partially completed welds were repaired by grinding and rewelding. Repairs to isolated areas were attempted up to three times before acceptance for final welding.

(b) Completed Weld

Radiographic examination of the completed welds showed the presence of moderate to pronounced gas defects in each weld. Several areas of porosity in each weld failed to comply with code requirements of size and grouping of pores. The defects were located and removed by grinding and the local area was re-examined for defects before final repair. The location and elimination of defects in each of the corner areas was extremely difficult, owing to difficulties in applying an adequate radiographic technique. Such areas required several examinations before final acceptance. Repair welding of ground areas sometimes introduced new defects, probably due to the grinding which had to be used in the restricted weldment, necessitating repeated attempts at repair and re-examination.

The vertical welds were finally accepted with minor gas pores present in isolated areas of the vertical welds. The horizontal welds were substantially free from defects.

Radiographic examination of the filler welds at each end of the twelve cooling tubes showed evidence of minor isolated gas pores.

3.2.2 Cell cover plate, skirt and diaphragms

The skirts were made from 6.5-mm Monel 400 plate and each skirt was

fabricated from four preformed pieces. The final welds were examined radiographically. The straight run welds were to the standard used with the cell tank. Owing to the difficult configuration, the flanged corners of the skirt contained some porosity and were difficult to repair. These areas were repaired and finally accepted with the porosity slightly in excess of that stated in the UKAEA specification.

The cover plate was made from 28-mm thick mild steel plate and had several mild steel branch pipes and sockets welded to it. These fillet welds were made using the standard metal-arc process.

The skirts were welded to the underside of the cover plate (a dissimilar metal weld) using the metallic-arc process with Monel 190 rod. Both the mild steel and dissimilar metal welds on the cover plate were generous fillet welds and could not be radiographed satisfactorily. Their acceptability was judged from the appearance and size of the welds.

After fabrication, the cover plate and tank were bolted together (to minimise distortion) and heat treated at 620°C for 1 hour to relieve the stresses induced in the mild steel cover plate during welding.

The diaphragms were welded from preformed angles of 6.5 mm thick Monel 400 and the standards used with the cell tank were readily achieved. Figures 3 (a,b,c) and 4 (c) show the cover plate and other components used in the cell. These components were pickled and degreased before assembly and use.

3.2.3 Scrubber tower

Construction of the Scrubber Tower

The scrubber tower consisted essentially of a rolled cylinder of Monel, 1520 mm long, 254 mm diameter and 3.2 mm thick (Figures 3 & 5).

The longitudinal weld in the scrubber tower was prepared under the same conditions as the successful experimental test butt weld in Figure 2. After construction, the scrubber tower was degreased before use.

Examination and Repair

Visual examination showed a pronounced groove on the inner surface of the tube in the region of the weld. Radiographic examination confirmed the presence of pronounced root grooving or under-flushing over the entire length of the weld, but there was no evidence of other internal defects in the weld. The weld was judged unacceptable. Repairs were effected by cutting out the entire weld area, rerolling the plate to a slightly smaller diameter and rewelding the longitudinal seam.

The size and frequency of occurrence of the pores in the new weld was

considered unacceptable for 3.2-mm thick Monel plate. Repair by welding the isolated defect areas frequently resulted in the displacement but not the total removal of the gas pores. Severe distortion of the plate (which was subsequently removed) was frequently encountered after these repairs.

3.2.4 Discussion and conclusions

The major problem in fabricating these two components was the tendency for porosity to occur in:

- (a) vertical welds;
- (b) at junctions between horizontal and vertical welds, *i.e.* in corners;
- (c) areas which had been repaired by grinding and rewelding; and
- (d) second and subsequent runs of multi-run welds.

The extent of porosity appeared to be more acute than was encountered in welds prepared in the experimental test program. Porosity was generally still present when Monel 60 filler rod containing deoxidants was used.

In spite of a considerable amount of experimental work and close control of welding and associated processes, the welding of Monel to the desired standard [UKAEA 1964] presented a very significant problem. In view of the difficulties that arose, it was necessary when welding Monel to relax the standards for weld defects in some circumstances.

3.3 In-Plant Experience

3.3.1 Fluorine cell

Corrosion of the Monel components in the cell was limited to a band 15 mm wide at the electrolyte operating level which had a smooth polished appearance and was corroded to a maximum depth of 0.5 mm. None of the welds in the cell (even at this band) showed any sign of corrosion. The mild steel cover plate showed no sign of corrosion and the skirt to cover plate dissimilar metal weld looked unmarked. The diaphragms also suffered no apparent corrosion and could have been returned directly to service.

The Monel components had a pink (copper) coloration when removed from the cell. After cleaning this changed, in some areas, to a green (nickel) colour.

Figures 6 & 7 show the condition of the cover plate and other cell components following cleaning after 3 months of duty in the fluorine cell.

3.3.2 Fluorine scrubber tower

In the scrubber tower, fluorine gas reacted with a 10 per cent by weight potassium hydroxide solution to produce potassium fluoride and oxygen. This reaction was vigorous, highly exothermic, and took place

at the end of the nozzle through which fluorine entered the tower. The nozzle was made of 0.5 in. N.B. Schedule 40 Monel 400 pipe (15.8 mm i.d.).

The replaceable nozzle corroded at a rate of approximately 25 mm of its length per week. The first nozzle was not replaced in time and the reaction perforated the Monel tower where the nozzle entered the tower. The perforation could not have been caused by a failure of the weld at this point as the surrounding parent metal was also badly corroded.

On removing the replaceable nozzle, it was noticed that a fillet weld attaching the nozzle pipe to a Monel flange had pitted corrosion marks. This weld could not be examined radiographically but clearly some imperfection had been present to allow pit marks (one was 2 mm diameter) to be formed. The surrounding weld appeared to be sound.

The tower was repaired with a welded Monel patch on the outside and, by careful welding, a smooth profile of weld metal was placed on the inside and the tower was returned to service with a new longer nozzle having a double wall thickness. No further problems were encountered with the tower wall which, on inspection after 3 months' duty, showed no corrosion except where it had previously perforated. This corrosion zone had not been enlarged since the repair. The nozzle was replaced a second time as a preventive measure. The corrosion rate of the second nozzle remained approximately the same as the first; however, the fillet weld at the flange was found to be sound on the second occasion. The first nozzle corroded by intergranular corrosion of the outside, the second by transgranular corrosion of the bore of the pipe.

3.3.3 Conclusions

No failures occurred of the welds in the fluorine cell and scrubber tower where high standards of fabrication had been applied, despite the corrosive nature of the environments in which these materials were used, as illustrated by the corrosion band in the tank and the perforation of the tower. Possibly, welding standards can be relaxed slightly in terms of porosity requirements if sufficient weld metal is present. However, X-ray examination of welds is necessary and it is absolutely essential to avoid inclusions, especially slag.

4. EXPERIENCE WITH MILD STEEL COMPONENTS

4.1 Manufacture

All mild steel components (with the exception of two vessels) within the fluorine cell experimental plant were subject to corrosive attack by fluorine, hydrofluoric acid or molten potassium bifluoride and were designed

and fabricated to Class I pressure vessel standards. Class I standards require radiographic inspection of welds and defects to be judged by the standards of the ASME code [ASME 1971]. Care was taken in fabrication to eliminate slag inclusions and, where radiographic inspection could not be carried out, ample thickness of weld metal was provided in the joints.

The exceptions to this standard were two demisters which were fabricated to a good standard (judged by visual inspection), the welds being well laid and having a generally smooth finish which was similar to that of the other mild steel vessels. One of the vessels was subject to fluorine with 10 per cent by volume of hydrogen fluoride vapour, the other contained hydrogen with the same vapour. Both operated at atmospheric pressure.

Some of the joints in the pipework used to vent HF vapour were borderline cases when inspected. These were not repaired and were also used to gain experience with relatively inferior welds.

All mild steel vessels and pipework were alumina-blasted to a dull grey metal finish and cleaned, before being put into service, by flushing in turn with trichloroethylene, cold water, two per cent alkaline solution, cold water and methanol; finally, dry nitrogen was used to dry the equipment. A similar procedure, with the alkaline wash omitted, was used for copper pipework.

4.2 In-plant Experience

No vessel or pipe leaked during 3 months' continuous operation of the plant.

After dismantling the plant, none of the vessels exhibited any sign of corrosion, with all welds appearing as laid.

4.3 Conclusions

No corrosion occurred in the welds which were fabricated to the standards adopted in constructing the plant. The parent metal in many cases was very thick and, in future construction of equipment for use with anhydrous fluorine and hydrogen fluoride, a thickness suitable for the pressure duty and weight of the vessel may be allowed. A corrosion allowance of 1 mm appears to be entirely satisfactory. However, care must still be taken in welding, though for non-pressure vessels some relaxation from the ASME code for Class I vessels could be allowed in terms of porosity if sufficient metal thickness is present. However, slag inclusions must be avoided.

5. BRAZED AND SILVER SOLDERED JOINTS

Brazed components were used in the fluorine cell. The anode hanger bars consisted of a chrome-molybdenum steel (AISI 4140) bar to which copper (electrically refined, tough pitch) electrode posts were brazed (Figure 4a). The brazing rod used was OXWELD 25, a low-fuming bronze type produced by the Union Carbide Corporation, Linde Division, (Tonawanda, N.Y.). In the cell, the brazing was subject to splashes of cell electrolyte (KF.2HF) and an atmosphere of fluorine with 10 per cent hydrogen fluoride at a temperature of up to 100°C. After 3 months' exposure to this environment, the brazing had a copper colour but was generally unmarked. The surrounding steel bar was corroded up to 2 mm deep in the areas near the brazing and up to 9 cm wide in a distinct band at the electrolyte operating level (Figure 7a). The copper components in the anode assembly (electrode posts, brazing and bolt washers) survived this corrosive environment far better than those of alloy steel (Figure 7a). It appears that copper would be a more suitable material for the hanger bar than the alloy steel (AISI 4140) used in the present cell. the hanger bar than the alloy steel (AISI 4140) used in the present cell.

The cathodes were constructed of commercial grade mild steel with copper electrode posts brazed to mild steel plates (Figure 4b), following the same procedure as that used with the anodes. The assembly showed no discernible signs of corrosion after 3 months of exposure to the cell operating environment (Figure 7b).

Silver soldered joints were used with brass fittings on copper lines in nitrogen service lines and, in some areas, these were occasionally subject to HF vapour. With the exception of one poorly soldered joint which leaked during commissioning trials, none of these joints leaked during service.

6. CONCLUDING COMMENTS

- . Fabrication in Monel to high standards requires the careful preparation of the workpiece and a high level of skill by the welder. Slag must be eliminated from the welds, and their quality has to be checked by using radiography.
- . The fluorine cell and fluorine scrubber-tower were fabricated in Monel 400 using Monel 60 filler rod with the TIG process and Monel 190 with the metallic-arc process. Porosity was the major problem with the welds and this occurred particularly in vertical welds and in corners, and was also found in reworked welds and in the second and subsequent runs of multi-run welds. These defects

were more prevalent than those found in the experimental tests and some consideration had to be given in certain circumstances to relaxing the standards of UKAEA specification AESS 6026 [UKAEA 1965] which were applied.

- . Plant experience showed that all the Monel welds remained sound and were not preferred centres of corrosive attack. However, significant corrosion of Monel did occur at the electrolyte level in the cell tank and, in particular, of the nozzle in the fluorine scrubber-tower.
- . With mild steel equipment, construction to Class I pressure vessel standards was followed and none of the welds leaked nor was any corrosion apparent at the surfaces of the mild steel welds.
- . The brazed joints were subject to little if any corrosion and at dis-assembly of the cell, brazing on the anode hanger bar and the cathode was sound and unmarked.

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TABLE 1

COMPOSITIONS OF NICKEL ALLOY PLATE AND WELDING RODS

REFERENCE	Nickel				Monel					
	Plate		Filler Rods		Plate	Filler Rods				
	200 ASTM	201	RN41	ERN61	400	RN40	ERN60	190	130	140
Ni	99.0	99.0	97.0	93.0	63.0- 70.0	68.0- 70.0	62.0- 69.0	62.0- 68.0	62.0- 70.0	62.0- 70.0
Cu	0.25	0.25	0.25	0.25	Ba1	Ba1	Ba1	Ba1	Ba1	Ba1
C	0.15	0.02	0.15	0.15	0.3	0.3	0.15	0.15	0.4	0.15
Mn	0.35	0.35	0.35	1.0	1.25	2.0	1.0	4.0	4.0	2.5
Fe	0.40	0.40	0.4	1.0	2.5	2.5	2.5	2.5	2.5	2.5
S	0.01	0.01	0.01	0.01	0.024	0.02	0.02	0.015	0.25	0.025
Si	0.35	0.35	1.0	0.75	0.5	0.5	1.5	1.0	1.0	1.25
Ti		0.10	0.5	2.0- 3.5			1.5- 3.0	1.0	1.0	1.5
Al				1.5			1.25	0.75	1.5	0.75
Nb+Ta										3.0
P								0.02		
Other			0.5	0.5		0.5	0.5	0.5		
	ASTM B162-69	WIGGIN PUBLICATION No. 3367c	ASTM B304-56T (1956)	ASTM B304-56T (1956)	ASTM B127-61 (1969)	ASTM B304-56T (1956)	ASTM B304-56T (1956)	WIGGIN PUBLICATION No. 3489	WIGGIN PUBLICATION No. 3050	WIGGIN PUBLICATION No. 3050

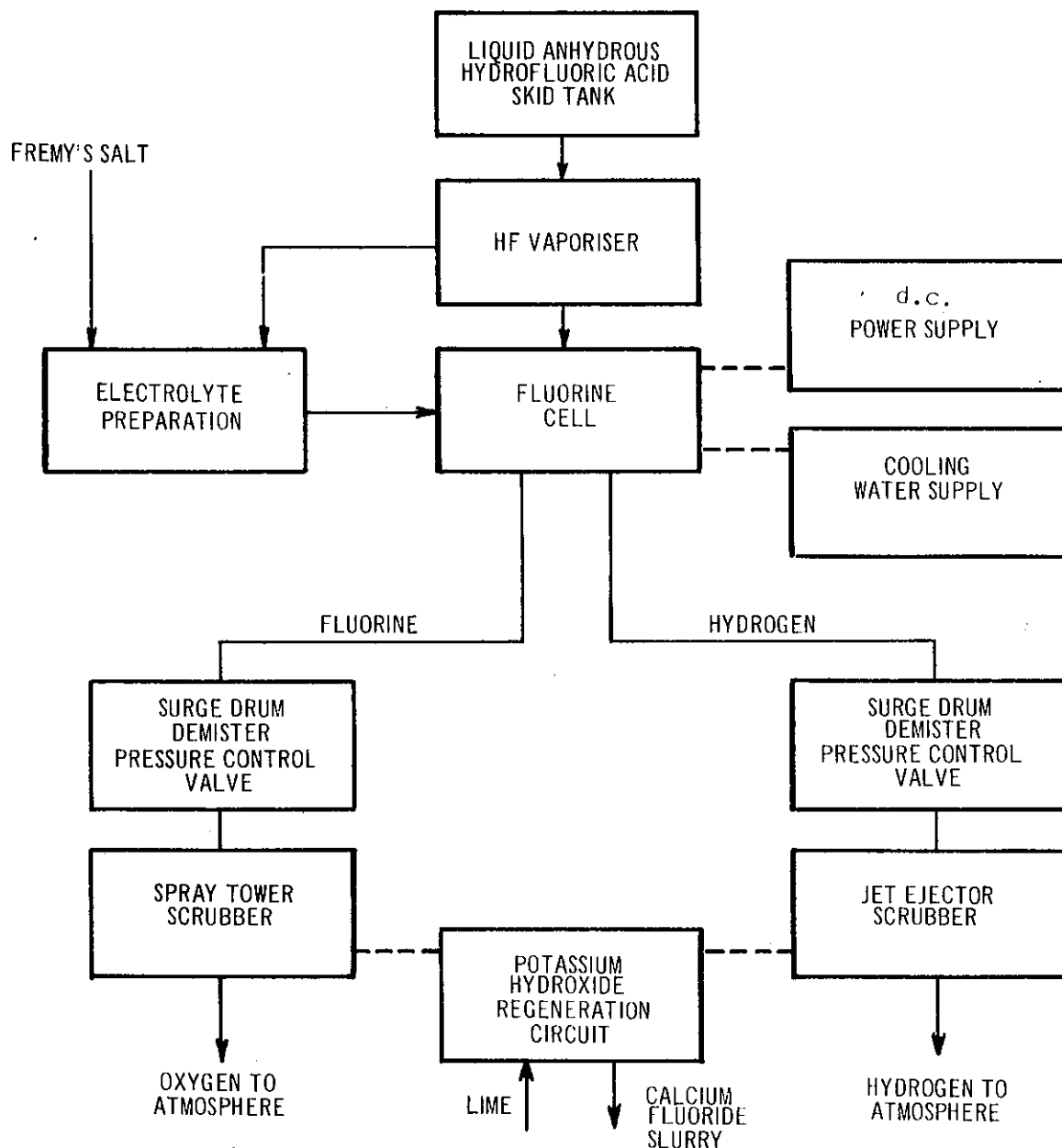
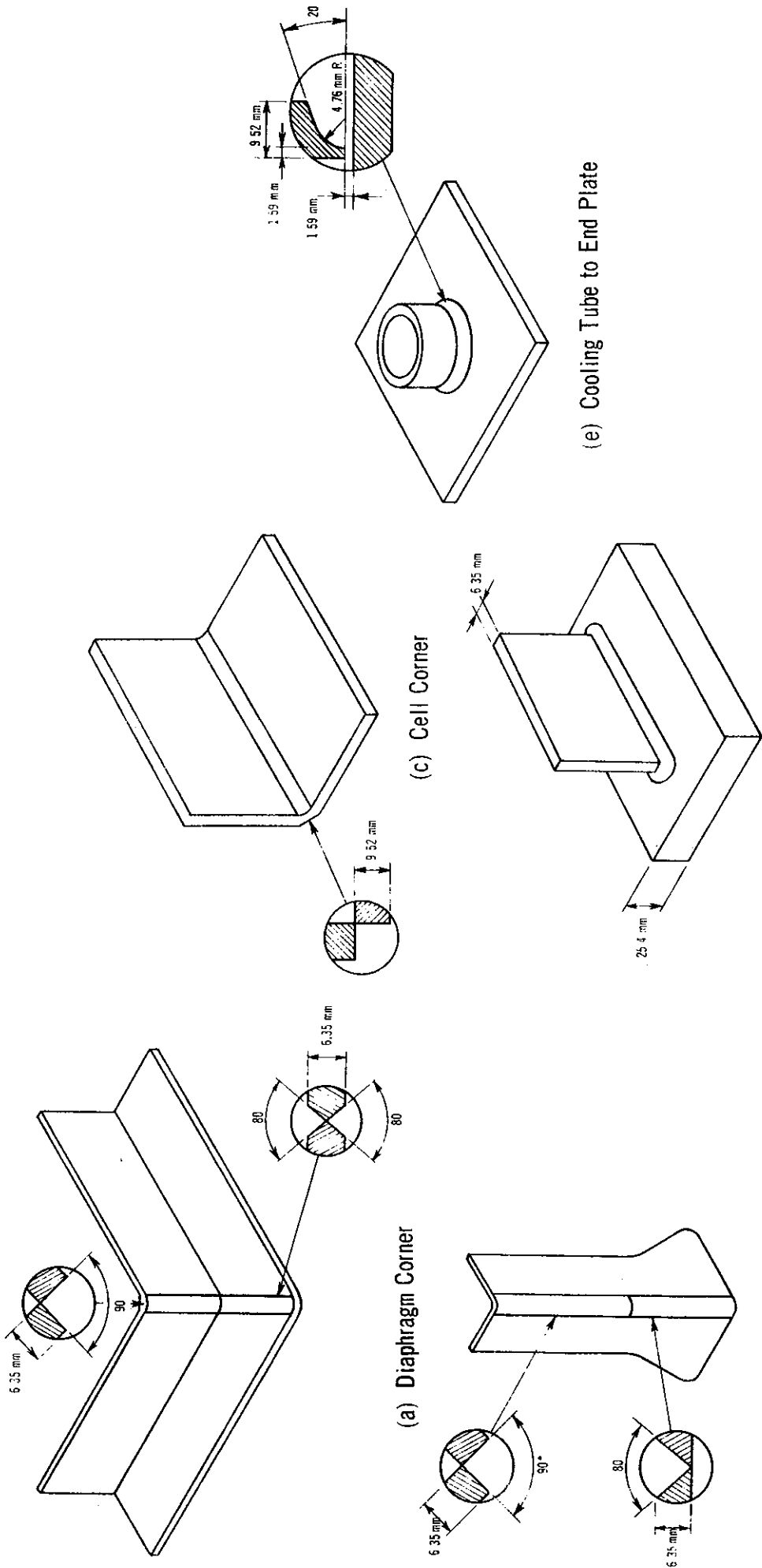


FIGURE 1. PROCESS USED FOR THE PRODUCTION OF FLUORINE



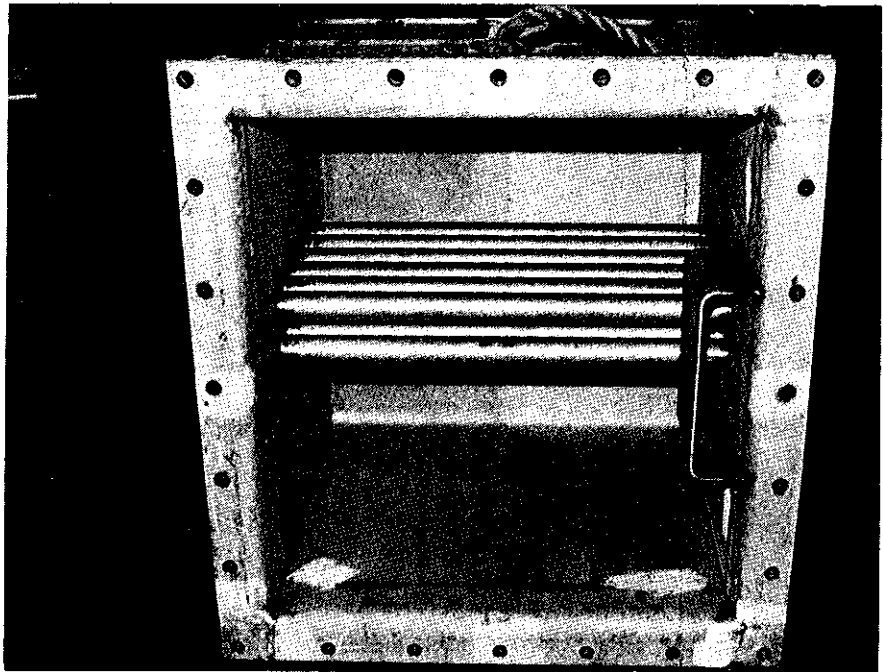
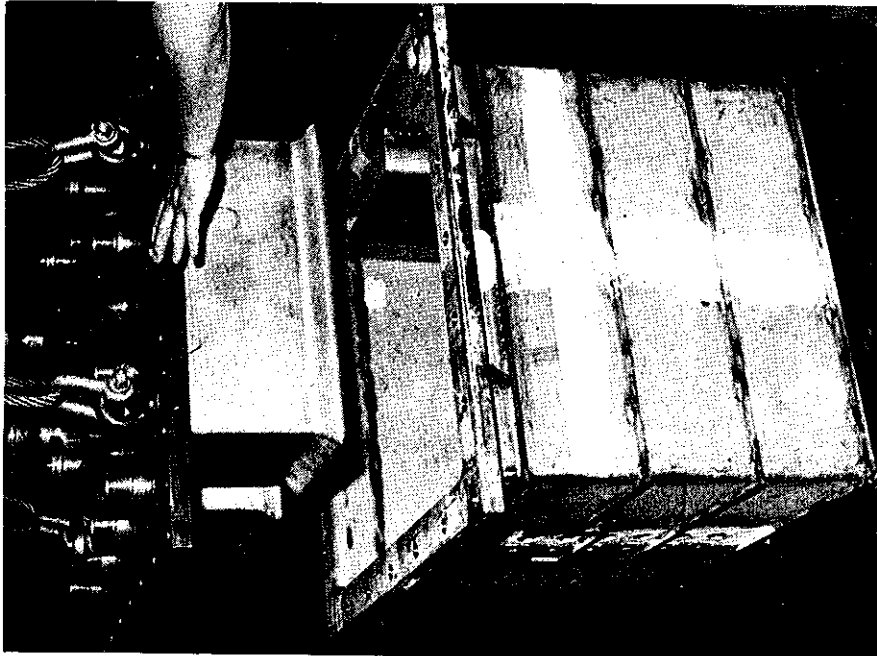
(b) Skirt Corner

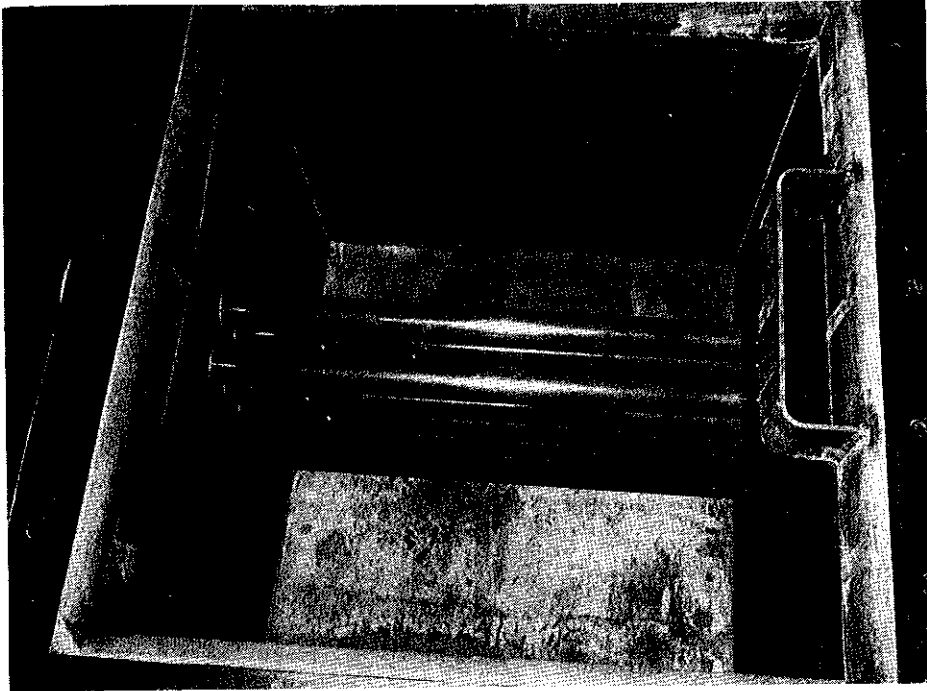
(d) Skirt to Cover Plate

(c) Cell Corner

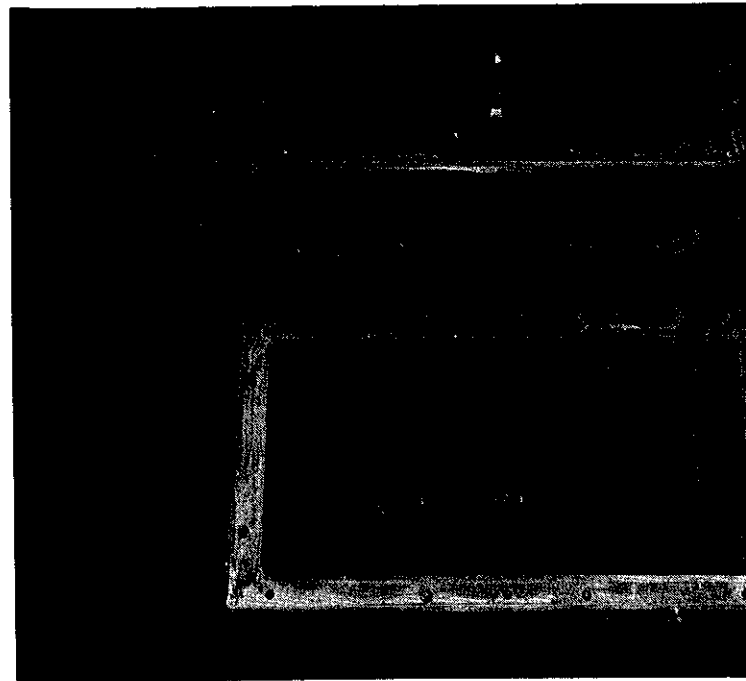
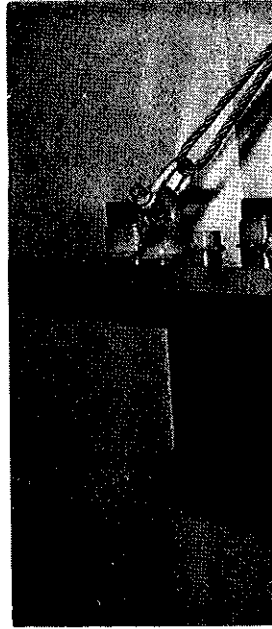
(e) Cooling Tube to End Plate

FIGURE 2. JOINT CONFIGURATIONS USED IN THE 1500 A EXPERIMENTAL FLUORINE CELL





(a) Cell tank



(c) Cover plate, underside

**FIGURE 6. CELL COMPONENTS AFTER 3 MONTHS' DUTY
CELL TANK, COVER PLATE**



(a)

FIGURE 7. CE
AI

