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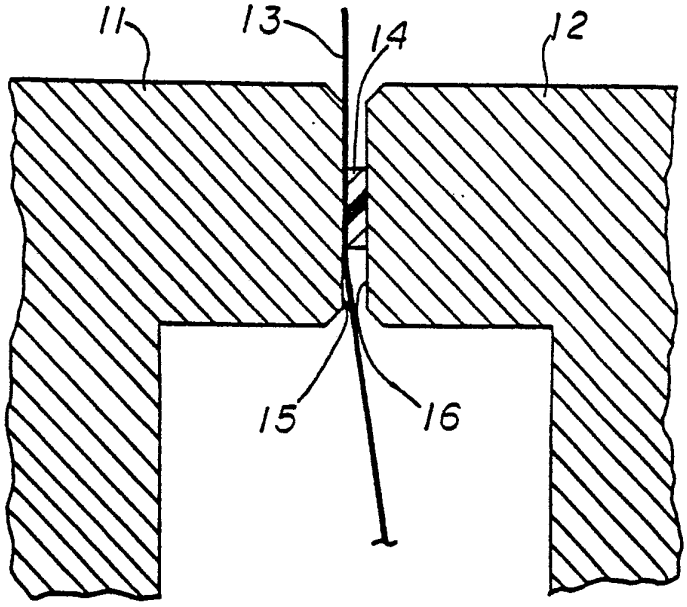

(57) Abstract

An electrolytic cell assembly of the filter press-type comprising a first frame (11), a second frame (12), a separator (13) is interposed between the frames to space apart an anode and a cathode, and a pre-compressed seal (14) is interposed between the first or second frames and the separator.

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SEAL FOR ELECTROLYTIC CELLS

This invention relates to a seal for electrolytic cells and a method of sealing electrolytic cells.

Electrolytic cells employing a separator and particularly cells used for the production of chlorine and alkali metal hydroxides by the electrolysis of aqueous solutions of alkali metal chlorides are generally of two types, the diaphragm-type and the membrane--type.

Generally, the membrane-type cell employs a sheet-like membrane of ion exchange material, such as those marketed by E. I. duPont de Nemours and Company under the trademark Nafion® and by Asahi Glass Company, Ltd. under the trademark Flemion®. Such cells are commonly of the flat plate or filter press-type having monopolar or bipolar electrode structures. U.S. Patent Nos. 4,108,742 and 4,111,779 issued to Seko et al. illustrate the bipolar system electrolytic cell. Illustrative

of another design is taught by Kenny in U.S. Patent No. 4,137,144 and the patent references cited therein.

In the filter press membrane-type cell, it is typical to clamp or otherwise compress the membrane in sheet form between the sides of the frame members. In addition, it has been common practice to interpose a gasket between one of the frame members and the surface of the membrane to form a fluid-tight, i.e., liquid- and gas-tight, seal upon compression of the frames and gaskets, to prevent leakage of electrolyte from one cell compartment to another or to the environment. This compression typically is applied manually or mechanically utilizing hydraulic rams or other types of pressure-applying apparatus to press the electrode frames and the separating gaskets together. Obtaining a fluid-tight seal, however, is desirably done without damaging the membrane.

The gasket material normally employed between the membrane and an electrode frame member of an electrolytic cell is of a resilient material, such as rubber or an elastomer. Commercial bipolar membrane electrolyzers generally use ethylene-propylene (EP) or ethylene-propylene-diene (EPD) as gasket material between the membrane and electrode frames. However, the above materials tend to deform and expand outwardly as pressure is applied to the frames via the frame members. As the gaskets deform outwardly, certain separators which are in contact with the gaskets tend to stretch when they are pulled under the pressure of the outwardly deforming gaskets. This stretching of the separator or membrane beneath the gaskets employed on adjacent electrode frames can cause the membranes to

break or tear when attempting to compress the frames into a fluid-tight cell. Furthermore, resilient gaskets require a higher compressive force to effect a seal which increases the risk of breaking or tearing the membrane.

Any tears or breaks in the membranes may reduce current efficiency during operation, greatly increasing electrical current usage while reducing the electrolytic operating efficiency of the cell. Too great a drop in current efficiency and/or electrolytic operating efficiency can require costly shutdown of the entire cell while the damaged membrane or membranes are replaced.

Custom made gaskets, flat sheets or O-ring EPD gaskets, are usually made and installed in cells to minimize the damage to the membrane. However, these resilient gaskets will still substantially recover their size and shape after release of a compressive stress applied to them. Thus, a resilient gasket cannot be pre-compressed and the membrane must be installed with the gasket between the cell frames before compression. This increases the likelihood that the membrane will be damaged when being compressed with the resilient gasket.

In view of the foregoing problems, it is desired to provide a seal for electrolytic cells and a method for sealing a cell with such a seal without damaging a membrane of the cell.

The present invention is an electrolytic cell assembly comprising a first frame a second

frame, a separator interposed between the frames to space apart an anode and a cathode, and a pre-compressed seal interposed between at least the first or second frames and the separator. The seal may be a gasket of a fluorocarbon polymer material.

The present invention also resides in a method of sealing an electrolytic cell comprising interposing a seal between at least a first frame or a second frame and a separator, said separator spacing apart an anode and a cathode in compartments defined by the first and the second frames, and compressing the seal, separator and the first and second frames, characterized by employing as the seal a pre-compressed, permanently deformed seal.

The present invention further resides in a method of sealing an electrolytic cell comprising:

(a) interposing a sheet member between at least a first and a second frame,

(b) interposing a seal made of a permanently deformable material between at least the first or second frame and the sheet member,

(c) compressing the first and second frames, sheet member, and seal to permanently deform the seal,

(d) releasing the compressive force to remove the sheet member,

(e) substituting a separator for the sheet member, and

(f) compressing the first and second frames, the pre-compressed seal and separator to form a liquid and gas tight seal.

Figure 1 is a cross-sectional view of an electrolytic cell assembly showing a pre-compressed gasket seal interposed between an electrode frame and separator.

Figure 2 is another embodiment of the present invention which is a cross-sectional view of an electrolytic cell assembly showing a pre-compressed gasket seal interposed between an electrode containing a recess and a separator.

Referring to Figure 1, there is shown a filter press-type electrolytic cell assembly having a pair of adjacent filter press-type frames 11 and 12. In this case, for illustration only, the first frame 11 is an anode frame and the second frame 12 is a cathode frame. Typically, the anode frame and the cathode frame is a single frame structure wherein an anode and a cathode are attached on opposite faces or sides of the structure and electrically connected through the structure. This cell assembly is typical of a filter press-type electrolytic cell unit which may be monopolar or bipolar. Herein the invention will be described with reference to a bipolar electrode-type, filter press-type electrolytic cell. Between the anode and cathode frames 12 there is interposed a separator 13 and a pre-compressed seal 14. The pre-compressed seal 14 may be interposed between the separator and either one of the frames 11 or 12. Furthermore, although only one pre-compressed seal 14 is shown, this invention encompasses the use of pre-compressed seals on both sides of separator 13.

By "pre-compressed seal" is meant a seal which has undergone deformation by a squeeze force or compression and the seal remains in a substantially compressed state or permanently deformed state, prior to its end use. By "permanently deformed seal" is meant that a seal is compressed to a desired thickness and remains compressed at such thickness without substantial change until its end use, i.e., installation between cell frames 11 and 12, or until further compression. The "pre-compressed" feature of the seal of the present invention is an advantage over prior art seals because by pre-compressing the seal, the seal undergoes the major compressive forces required to deform the seal in absence of a separator which might be damaged by the major compressive forces.

Referring to Figure 1 again, the cell frames 11 and 12 contain a peripheral lateral surface or face 15 and 16, respectively. As shown, the seal 14 is positioned between the separator 13 and cathode frame 12 but obviously can also be positioned between the separator 13 and anode frame 11. The separator 13 is usually larger in size than the cell frames 11 and 12 and preferably, extends beyond the outer periphery of frames 11 and 12, while the seal 14 is generally confined to within the lateral face 15 of the anode frame 11 or, as shown in Figure 1, within the lateral face 16 of the cathode frame 12. The separator 13 is shown bent at a slight angle away from lateral face 15 only to illustrate the lateral face 15 more clearly. The separator, typically, contacts the lateral face 15 completely, i.e. over its entire surface. The seal 14 may line the entire lateral face 15 or 16 of the anode or cathode frames 11 and 12, respectively. Furthermore, a liner (not shown) on lateral face 15 or 16 can

be used to protect the frame structure from corrosive environments. For example, the cell frame 11 may contain a liner made of metal such as titanium or made of plastic such as polytetrafluoroethylene adjacent lateral face 15.

The frames 11 and 12 may be any shape used in typical electrolytic cells. For example, the frame members may be in the shape of rectangular bars, C or U channels, cylindrical tubes, elliptical tubes I-shaped or H-shaped. Preferably, the cross-sectional shape of one electrode frame unit containing frames 11 and 12 is I-shaped.

The frames 11 and 12 may be constructed of any material resistant to corrosion by the electrolytes and the products of electrolysis. For example, the anode frame which is in contact with an anolyte solution contained in the anode compartment may be made of metals such as iron, steel, stainless steel, nickel, titanium, or alloys of these metals. The cathode frame which is in contact with a catholyte solution contained in the cathode compartment may be made of iron, steel, stainless steel, nickel, or alloys of these metals. Similarly, plastic materials such as polypropylene, polybutylene, polytetrafluoroethylene, fluorinated ethylene propylene, and chlorendic acid based polyesters can be employed for the anode and cathode frames.

The separator 13 of the present invention may be a hydraulically permeable or impermeable separator. Preferably, inert flexible separators having ion exchange properties and which are substantially impervious to the hydrodynamic flow of the electrolyte and the passage

of gas products produced during electrolysis are used. More preferably used are cation exchange membranes such as those composed of fluorocarbon polymers having a plurality of pendant sulfonic acid groups or carboxylic acid groups or mixtures of sulfonic acid groups and carboxylic acid groups. The terms "sulfonic acid groups" and "carboxylic acid groups" are meant to include salts of sulfonic acid or salts of carboxylic acid which are suitably converted to or from the acid group by processes such as hydrolysis. An example of a carboxylic acid type cation exchange membrane is commercially available from Asahi Glass Company under the trademark Flemion®. Another example of a suitable membrane having cation exchange properties is a perfluorosulfonic acid membrane sold commercially by E. I. duPont de Nemours and Company under the trademark Nafion®.

The seal 14 of the present invention may be a gasket, and like the frames 11 and 12, the gaskets should be made of a material which is corrosion-resistant to the electrolyte and the products of electrolysis. In the production of chlorine and caustic, for example, the gasket 14 must, of course, be substantially inert to acid, brine, chlorine, hydrogen and caustic such as are present in the cell during normal operating conditions. The gasket 14 is also preferably non-conductive and should be of a material having high volume resistivity and good sealability after it has been compressed. A main feature of this invention is that the material of gasket 14 be non-resilient or substantially permanently deformable. The gasket 14 is preferably made of a fluorocarbon polymer material and more preferably, of a polytetrafluoroethylene (PTFE). Preferably, a gasket

made of a porous expanded PTFE material sold as GORE-TEX® by W. L. Gore & Associates, Inc. Elkton, MD is used. The gasket 14 of Figure 1 may be attached to the lateral face 16 of frame member 12, prior to applying a compressive force to the cell assembly, by an adhesive such as cement or epoxy, known in the art. The cement or epoxy used should also be inert to the electrolysis environment.

An alternative embodiment of the present invention is shown in Figure 2 which shows a membrane 23 and a pre-compressed, first gasket 24 interposed between an anode frame 21 and a cathode frame 22 having lateral faces 25 and 26, respectively. The structure and materials of construction with respect to the membrane, the pre-compressed, first gasket and the electrode frames of Figure 2 are identical to those illustrated in Figure 1 except that in Figure 2, there is shown a recess 27 in the lateral face 26 of frame member 22. It is within the scope of this invention that either the lateral face 25 or 26, or both, of the cell frames 21 and 22 can contain a recess or plurality of recesses. In addition, Figure 2 shows a second gasket 28, either uncompressed or pre-compressed, fitted into the recess 27. Second gasket 28 may be of an identical material or of a different material than first gasket 24. Although the gasket 14 of Figure 1, and the gaskets 24 and 28 of Figure 2, are illustrated as being generally rectangular in cross-section, various other suitable shapes may also be employed so as to obtain improved sealing effects. For example, the gaskets may be circular or toroidal in cross-section and the recess may be generally rectangular with its corners rounded. The recess may also be a generally semi-circular or a triangular groove viewed in cross-section or any other

shape desired. Any well-known adhesive such as cement or epoxy material may be used at the interface 29 of gasket 28 and pre-compressed gasket 24 to hold the pre-compressed gasket 24 in place to the lateral face 26 of cathode frame 22 prior to applying a compressive force to the cell assembly. It is also within the scope of this invention that gaskets 24 and 28 be one piece.

In carrying out the method of the present invention, an uncompressed gasket is attached, preferably with an adhesive, to the lateral face of the cathode or anode frames of an electrolytic cell of the filter press-type. A sheet, preferably having a thickness and flexible properties similar to a membrane to be employed in the electrolytic cell assembly, is then positioned between the anode or cathode frames and the gasket. Preferably, the sheet may be of paper or plastic material and more preferably of Kraft paper. Preferably, the thickness of the paper sheet is in the range of from 0.125 to 0.25 mm. A compressive force or load, which can be applied to the assembly by any known hydraulic form or clamping means, is applied to the cell assembly. After compressing the gasket to a desired thickness, the paper sheet is removed from between the gasket and electrode frame member. At this point, the gasket is pre-compressed and generally permanently deformed, i.e., release of the compressive force leaves the gasket in its compressed form.

Generally, a compressive load of from 10 to 100 percent of the final compressive load required to obtain a desired electrolytic cell brine gap or a final desired gasket thickness is used to pre-compress the

gasket. For example, the gasket may be compressed to a thickness of from 2.0 to 0.38 mm using a compressive load of from 2750 kPa to 17240 kPa. After removing the paper sheet from between the gasket and electrode frame, a membrane is substituted for the paper sheet. The membrane and pre-compressed gasket are then compressed betweem the cell frames to provide a complete liquid- and gas-tight electrolytic cell assembly. Since the gasket is already in a compressed form, stresses applied to the membrane due to gasket deformation are negligible and, thus, damage to the membrane is minimized.

The following examples are provided to illustrate the invention only and not to be limited thereby. The examples describe use of a pre-compressed gasket in filter press-type electrolytic cells employing a membrane.

Example 1

A pair of 0.61 by 0.61 meter dummy flat plate-type cell frames were used to test a gasket of GORE-TEX® material. A 12.7 by 4.76 mm thick rope of GORE-TEX® material was glued on a flat lateral face of one of the frames. The rope was lapped at its ends to provide a seal. No gasket was attached to the other frame. The two cell frames were installed on a 0.61 by 0.61 meter hydraulic squeezer. A sheet of Kraft paper, approximately 0.25 mm thick, extending beyond the periphery of the cell frames was installed between the frames. The gasket between the frames was pre-compressed first to about 1.6 mm thick with about 1380 kPa on the gasket. The cell frames were then opened up and the Kraft paper removed. A Nafion® membrane No. 324 was then installed between the gasket and a cell frame. The cell frames with the membrane were then squeezed together again.

Hot water at a temperature of from 85°C to 93°C was circulated through the cell at an internal cell pressure of from 34 to 103 kPa. The squeezer hydraulic force was increased proportionally to the internal pressure in order to hold the gasket pressure constant at about 2413 kPa. The test was run for about 140 hours. No leakage was observed. The cells were then shut down and opened up for inspection. The membrane showed no sticking and no thinning on gasket area.

Comparative Example A

A pair of 10 cm by 10 cm dummy flat plate-type cell frames were used to test an uncompressed gasket of GORE-TEX® material. A 9.5 mm wide by 3.2 mm thick rope of GORE-TEX® material was glued on a flat lateral face of one of the frames. The rope was lapped at its ends to provide a seal. No gasket was attached to the other frame. A Nafion® 324 membrane was installed between the frames. The two cell frames were squeezed between the platens of a hydraulic squeezer. Hot water at a temperature of 90°C was circulated through the cell at a pressure of from 34 to 207 kPa. The hydraulic force was increased proportionally to the internal pressure to hold the gasket pressure at 3447 to 4137 kPa to stop leakage.

The test was run for 48 hours. The cell frames were then opened up and the gasket and membrane were inspected. It was observed that the area of the membrane under gasket pressure was stretched out and thinned.

Example 2

An electrolyzer consisting of four rectangular flat plate-type cell frames (1.22 met. by 2.44 met.)

was gasketed as follows: A 6.35 mm diameter rope of GORE-TEX® material was glued inside a 6.35 mm groove located on the cathode side of the cell frames. The rope was pressed down into the groove so that the top surface of the GORE-TEX® material was flush to the cell frame lateral face. Then a 12.7 mm wide by 4.76 mm thick rope of GORE-TEX® material was glued centered on the top of the 6.35 mm diameter rope and extended around the entire cathode gasket face. The rope was lapped at its ends to provide a seal. No gasket was placed on the anode side of the cell frames.

A sheet of Kraft paper, approximately 0.25 mm thick extending beyond the periphery of the cell frame was attached with masking tape to the anode side of each cell frame.

The gasketed cell frames with attached paper sheets were installed one at a time on a cell skid between the two platens of a hydraulic squeezer. The cell frames were then compressed together at a pressure of 8274 kPa on the hydraulic cylinder (42676 kg force on the squeezer) which was equivalent to a pressure of about 3723 kPa on the gasket. The 12.7 mm GORE-TEX® rope was compressed from the initial 12.7 mm wide by 4.76 mm thick shape to a 15.9 mm wide by 1.6 mm thick gasket. The hydraulic pressure was then relieved. The cell frames were spread out and the paper sheets removed. A sheet of Nafion® No. 324 membrane was then installed between adjacent cell frames. After installing the membranes, the cell frames were compressed again applying a pressure of approximately 11377 kPa on the hydraulic cylinder.

Anolyte and catholyte were circulated through the cells and power was turned on to energize the electrolyzer. The cells were operated at a temperature of 90°C and at an internal pressure ranging from 34 to 138 kPa. The squeezer hydraulic cylinder pressure varied accordingly from 11377 up to 15169 kPa to stop gasket leak. The gasket thickness varied from 1.27 mm to 0.76 mm. The average gasket pressure was held at about 3447 kPa.

During eight months of operation, the electrolyzer was shut down five times for cell modifications and membrane change outs. Used gaskets were taken off the cells during each shutdown and visually inspected. No damage was seen on the gaskets or on the membranes in the gasket area. New gaskets were installed on the cells according to the above procedure after each shutdown.

CLAIMS

1. An electrolytic cell assembly comprising a first frame, a second frame, a separator interposed between the frames to space apart an anode and a cathode, characterized by employing a pre-compressed and permanently deformed seal between at least the first or second frames and the separator.

2. The cell assembly of Claim 1 wherein the separator is a membrane and the seal is a gasket made of a fluorocarbon polymer.

3. The cell assembly of Claim 2 wherein the fluorocarbon polymer is a porous expanded polytetrafluoroethylene material.

4. The cell assembly of Claim 3 wherein at least one of the frames has a recess therein, and wherein the recess contains a gasket.

5. A method of sealing an electrolytic cell comprising interposing a seal between at least a

first frame or a second frame and a separator, said separator spacing apart an anode and a cathode in compartments defined by the first and second frames, and compressing the seal, separator and the first and second frames, characterized by employing as the seal a pre-compressed, permanently deformed seal.

6. A method of sealing an electrolytic cell comprising:

(a) interposing a sheet member between at least a first and a second frame,

(b) interposing a seal made of a permanently deformable material between at least the first or second frame and the sheet member,

(c) compressing the first and second frames, sheet member, and seal to permanently deform the seal,

(d) releasing the compressive force to remove the sheet member,

(e) substituting a separator for the sheet member, and

(f) compressing the first and second frames, the pre-compressed seal and separator sufficient to form a liquid and gas tight seal.

7. The method of Claim 6 wherein the sheet member is made of paper or a synthetic resinous material.

8. The method of Claim 6 wherein the seal is permanently deformed to a thickness to provide a liquid and gas tight seal.

9. The method of Claim 6 wherein the seal is a gasket made of a fluorocarbon polymer.

10. The method of Claim 9 wherein the fluorocarbon polymer gasket is a porous expanded polytetrafluoroethylene.

Fig. 1

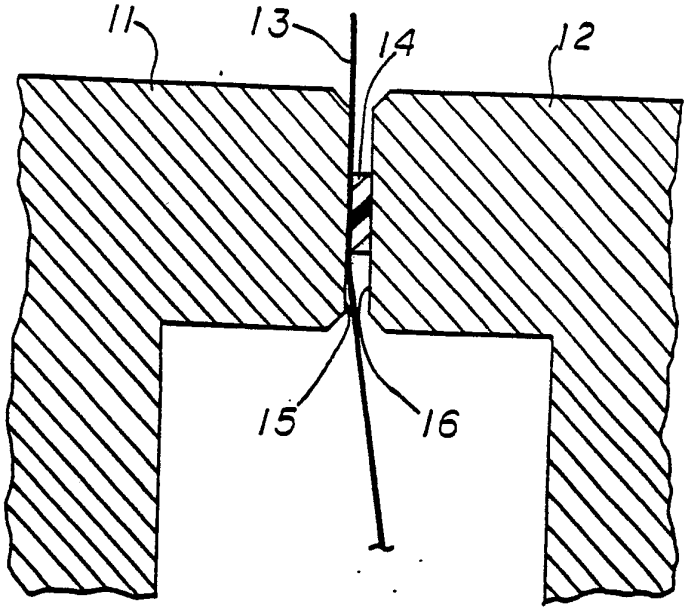
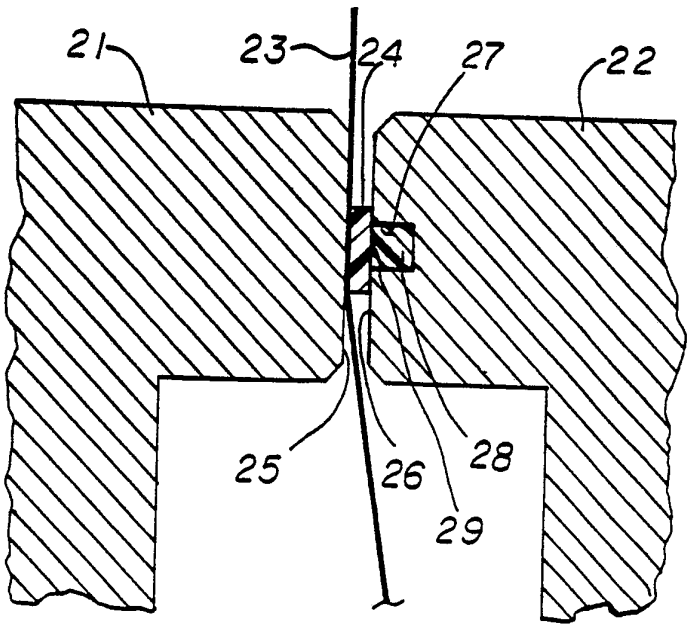


Fig. 2



INTERNATIONAL SEARCH REPORT

International Application No PCT/US85/01821

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

INT. CL.[4] C25B 9/00; C25B 13/02; C25B 13/08

U.S. CL. 204/253, 204/279

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
U.S.	204/253, 254, 255, 256, 257, 258, 279

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
X	US, A, 4,344,633, Published, (Niksa), 17 August 1982	1,2,3,5
X	US, A, 4,013,535, Published, (White), 22 March 1977	1,5
Y	US, A, 3,378,480, Published, (Reinshagen et al) 16 April 1968	1,2,3,4,5
A	US, A, 4,175,025, Published, (Creamer et al), 20 November 1979	1-5
A	US, A, 4,342,460, Published, (Eng), 03 August 1982	1-5
Y	US, A, 4,368,109, Published, (Ford), 11 January 1983	1,5
Y	US, A, 4,431,502, Published, (Ford), 14 February 1984	1,5
Y	US, A, 4,207,165, Published, (Mose et al), 10 June 1980	1,2,5

* Special categories of cited documents: [15]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
November 19, 1985	03 DEC 1985
International Searching Authority [1]	**Signature of Authorized Officer [20]**
ISA/US	Donald R. Valentine

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No [18]
A	US, A, 4,367,134, Published, (Kircher), 04 January 1983	6-9
A	US, A, 4,430,179, Published, (Ford et al), 07 February 1984	6-9