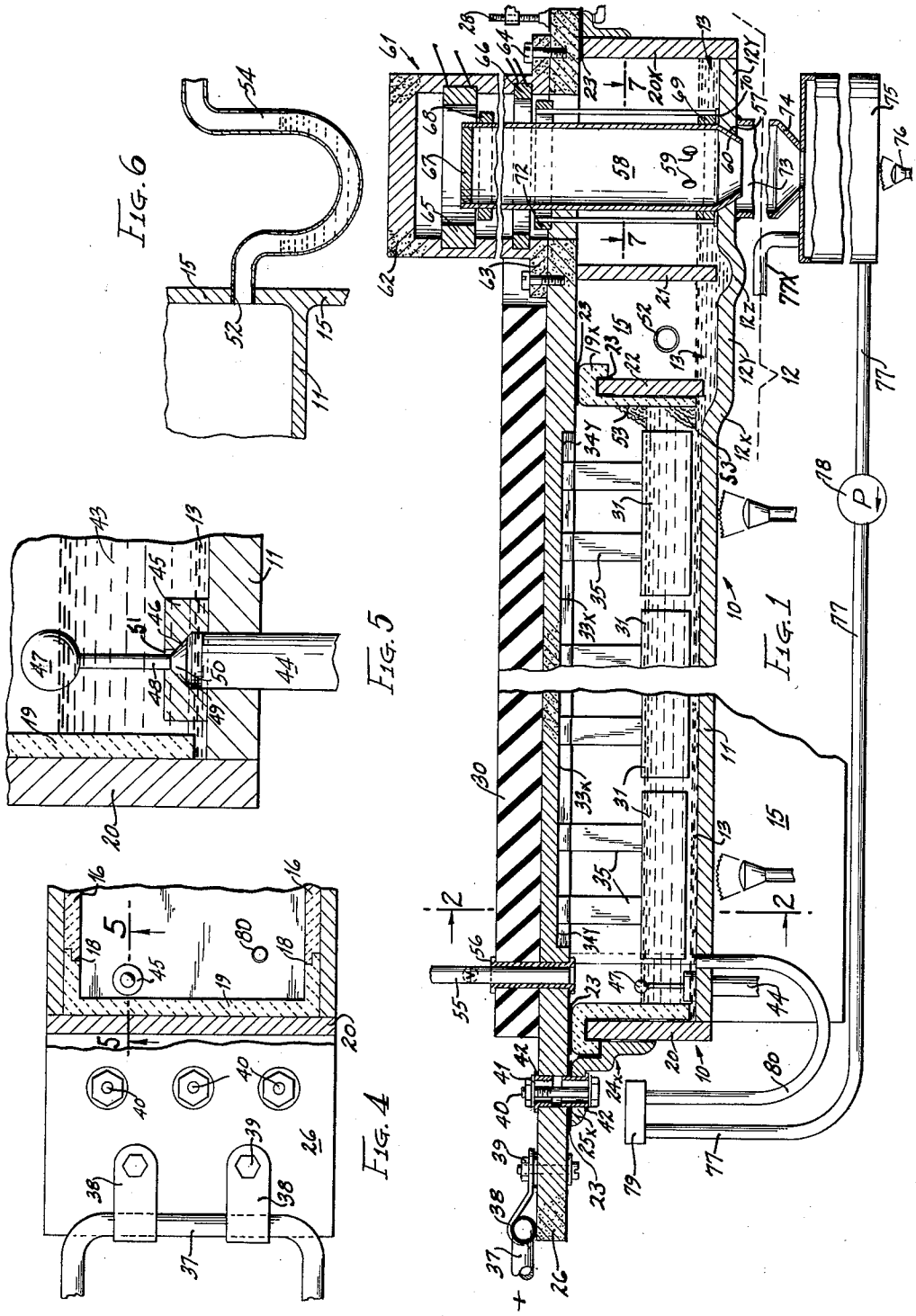
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ELECTROLYTIC CELL AND PROCESS THEREOF

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ELECTROLYTIC CELL AND PROCESS THEREOF

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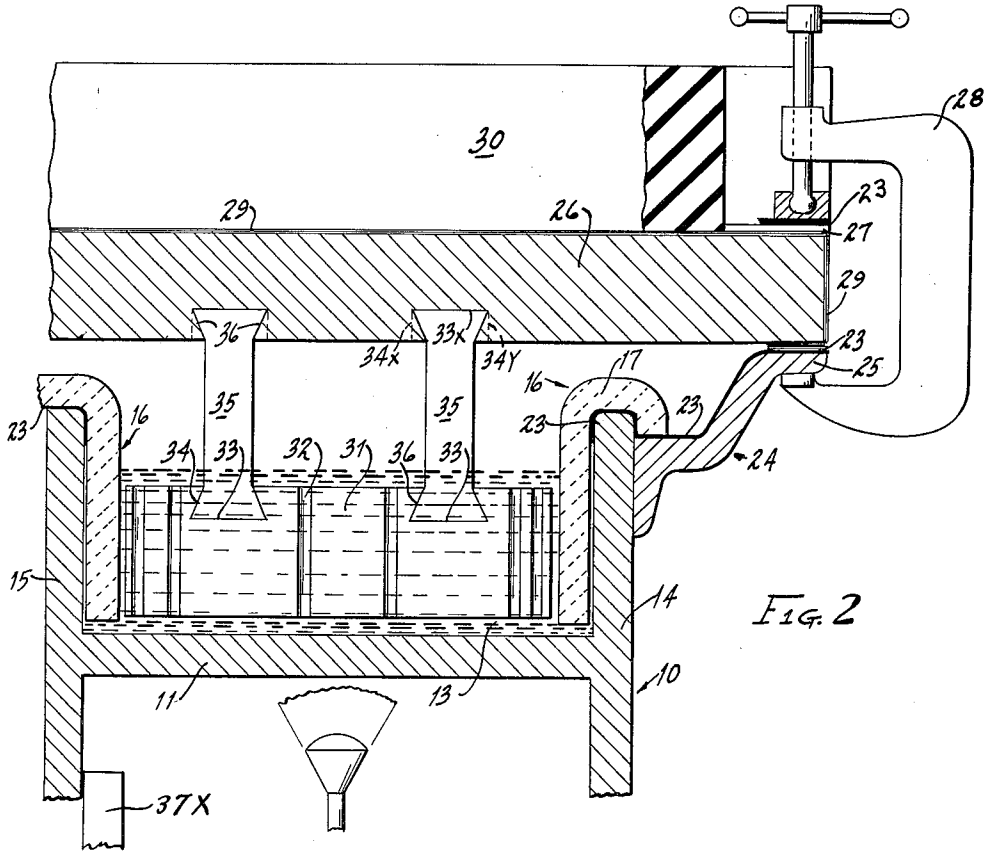



FIG. 2

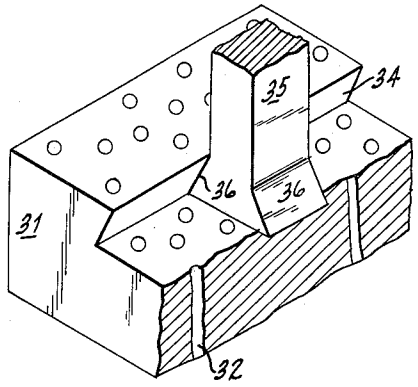



FIG. 3

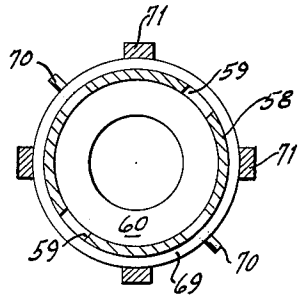



FIG. 7

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3,104,213

# ELECTROLYTIC CELL AND PROCESS THEREOF

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This invention relates to an electrolytic cell and the electrolytic process performed therein and more particularly to an electrolytic cell for the decomposition of alkali metal halide salts and whose interior is hermetically sealed from contact with air.

It is an object of this invention to provide an electrolytic cell operable with high current densities above those densities conventionally used in the decomposition of alkali metal halide salts.

It is another object of this invention to provide an electrolytic cell which may be cleaned free from dross accumulation within the cell without exposing the interior of the cell to contact with air, and therefore cleaning out of dross under hermetically sealed conditions.

It is another object to provide a method of cleaning the undersurfaces of the anodes under conditions of maintaining a hermetically sealed cell.

It is a further object to provide a method for the inexpensive production of alkali metals, for example sodium, potassium and lithium, together with chlorine.

It is another object to provide an electrolytic cell of inexpensive manufacture.

These and other objects of this invention will become apparent upon reading the following descriptive disclosure taken in conjunction with the drawing in which:

FIG. 1 is a longitudinal section view of the cell,

FIG. 2 is in part a transverse section view taken on line 2—2 of FIG. 1,

FIG. 3 is a partial section view showing the manner of securing a graphite anode block to a graphite holder,

FIG. 4 is a top front end view, partly in section, showing the manner of securing the electrical feed conduit to a graphite cell conductor plate,

FIG. 5 is a vertical section view, broken away in part, showing the float valve used to maintain a desired level of fused molten salts within the cell,

FIG. 6 is a transverse section view, broken away in part, and showing the manner of maintaining a trap having fluid therein to prevent introduction of air into the cell, and

FIG. 7 is a transverse view taken on line 7—7 of FIG. 1 and showing a cylindrical sleeve in section and a slideable ring disposed about said sleeve and seated upon a pair of prongs integral with said sleeve adjacent the sleeve base.

Turning to FIGS. 1 and 2, the cell of this invention is made from an elongated metallic or non-metallic element having a channel therein, for example a steel I-beam **10**, the cross plate **11** of which is disposed downwardly in a predetermined area **12** adjacent the rear of the cell.

The cell I-beam **10** is sloped toward the right (FIG. 1) so that the feed end is at a higher level than the rear end of the cell thereby permitting liquid metal, preferably lead **13**, to flow from the front end over the cross beam **11** to the rear end area **12**.

The slope of the cell may vary within a suitable range but a slope of 1 degree or less is preferred. And while I prefer to use liquid or molten lead as a moving stream cathode, other liquid or melted metals are operable in this cell.

The construction of the cell is best seen in FIG. 2 wherein the manner of hermetically sealing the cell interior from contact with the outside atmosphere is shown. The construction shown on the right of FIG. 2 is repeated on the left side of the I-beam **10** but is not shown as it is a mere repetition of the shown structure.



The beam shape **10**, having a cross-plate **11** and upright sidewalls **14** and **15**, is provided with a continuous refractory blanket **16** along the entire interior walls of the anode chamber of the cell, such blanket **16** serving as an electrical insulator and also as a protector of the walls **14** and **15** against chemical attack by the chemical contents within the chamber. The continuous blanket **16** is provided with a top curved lip **17** which completely covers the rounded top edges of walls **14** and **15**. The refractory blanket **16** also comprises an integral piece **19** which covers the interior cell wall of transverse steel plate **20** welded to the front end of the I-beam **10** in a hermetical manner. In FIG. 4 the single refractory end piece **19**, extends from the top ship-lap joint **18** to the bottom ship-lap joint **18** over the top of end plate **20** (FIG. 1) as well as over the respective top edges of side walls **14** and **15**.

As shown in FIG. 1, the rear end of I-beam **10** is provided with a vertical plate **20X** welded hermetically, as in the case of vertical plate **20**, to the rear end of the cross-plate **11** and the respective rear ends of the side walls **14** and **15**.

FIG. 1 shows the cross-plate **11** as deformed downwardly in an area **12**, this area being characterized by a suitable slope **12X** leading the area **12** to a relatively horizontal non-sloped area **12Y** in which a trough **12Z** is located.

A transverse plate **21** is hermetically welded to the opposed walls **14** and **15** of the I-beam **10** and suitably above the trough wall **12Z** to permit passage of liquid between the bottom of plate **21** and the top of trough **12Z**.

A second transverse plate **22** is hermetically welded to opposed side walls **14** and **15**. As shown in FIG. 1, plate **22** does not extend to the top of the side walls **14** and **15**, however the bottom of plate **22** extends suitably downward adjacent slope **12X** so that the bottom edge of plate **22** is substantially in the same plane as the top of cross-plate **11**. An integral refractory insulator unit **19X** is disposed over the top edge of plate **22** upon flexible mica strips **23**. Mica strips **23** are also placed upon refractory end elements **19** and **19X**.

Turning to FIG. 2, the exterior surfaces of side walls **14** and **15** are each provided with longitudinal support flanges **24** welded hermetically to the side walls.

The flanges **24** are disposed below the top edge of the side walls **14** and **15** and are provided with a horizontal portion **25** for supporting a thick electric current conductor plate **26**, for example graphite. This conductor plate **26** is insulated from support flanges **24** by a plurality of flexible mica strips **23**. A steel strip **27** is placed along both top edges of the conductor plate **26** and mica strips **23** are placed thereupon. A plurality of spaced-apart conventional C-clamps **28** are used to secure conductor plate **26** to flanges **24** in an air-tight hermetically sealed manner. The use of insulating mica is to prevent electrical contact between conductor plate **26** and steel C-clamps **28** and steel flanges **24**.

The conductor plate **26** is provided with a top glass coating **29** to counteract the porosity of graphite and thereby make it impervious to air passage and thus effect an air-tight condition on the air exposed graphite surfaces.

A thick blanket of heat insulator material **30** is disposed upon the conductor plate **26** in order to prevent loss of heat from the cell. The heat insulating material may be any conventional high heat resistant material such as asbestos, etc., and cut-outs are provided along its sides in order to accommodate the C-clamps **28**.

As shown in FIGS. 1 and 2, the lower edge of refractory blanket **16** is disposed suitably above the top surface of cross-plate **11** and suitably close to the graphite anodes

31. The blanket 16 is an electrical insulator and prevents loss of electricity from the anodes 31 to the adjacent I-beam side walls 14 and 15. The conventional synthetic flexible sheet mica 23 is also disposed upon flanges 24 and thence intermediate the top of a respective I-beam side wall 14 and 15 and the portion 17 of blanket 16 disposed thereover.

The anodes 31 are of rectangular construction and provided with a plurality of vertical boreholes to permit chlorine gas to bubble up these boreholes. As shown in FIGS. 1 and 2 the bottom wall of the anodes 31 is disposed above the molten lead surface in the molten salt yet suitably close to the lead surface, thereby permitting electrolysis of a suitable layer of molten salt with the chlorine gas liberated passing up the anode boreholes 32 and the alkali metal formed passing into the flowing lead.

The anodes 31 are provided with a pair of grooves 33 having under-cut edges 34. The conductor plate 26 is also provided with a pair of grooved longitudinal channels 33X having under-cut edges 34X. Channels 33X, each terminate at each end in a suitable rectangular cavity 34Y (shown in outline) having no under-cut edges. A plurality of holder bars 35 each having top and bottom opposed taper edges 36 are placed into the respective receiving cavities 34Y and then slid into grooves 33X. Next the bottom tapered edges of four holder bars 35 are slid into the grooves 33 of an anode so that at least two bars 35 are disposed in each groove 33 in spaced apart relationship.

As shown in FIGS. 1 and 2, each anode 31 is supported by four holder bars 35, keyed into like grooves into both the anode 31 and into the conductor plate 26.

As shown in FIG. 4, the conductor plate 26 extends beyond the I-beam front end and its welded closure plate 20. An electrical water cooled copper pipe 37 is secured to the conductor plate 26 by a pair of conventional hold-down straps 38 secured by metal conventional nut and bolts 39. The return electrical direct current is made through electrical conduit 37X (FIG. 2).

As shown in FIG. 1, the front end of the conductor plate 26 is secured to the respective section of transverse flanges 24X by means of conventional nuts and bolts 40 passed through suitable apertures in both the conductor plate 26 and flange portion 25X. The nut and bolt combinations 40 are provided with washers 41 resting upon flanged electrical insulator tubes 42. As shown the tubes 42 are disposed about both the top and the bottom of the nut and bolts 41 and do not touch one another, thereby permitting movement of one insulator tube 42 toward the other during the tightening process of hermetically sealing conductor plate 26 to flanges 24X. The air column between the insulator tubes 42 is itself an insulator, hence a single tube is not necessary. As shown a sheet of flexible mica is disposed upon flanges 24X and the top edge of wall 20 and beneath element 19. Mica is also placed on top of refractory element 19.

The process performed in this cell is as follows: The molten salt 43 (FIG. 5) is fed into the air free hermetically sealed cell through pipe 44 by way of a self-regulating float valve. The pipe 44 is secured as by welding into an aperture in the front end of cross-plate 11. A cylindrical collar 45 having an inner beveled shoulder 46 is secured by welding around pipe 44 to plate 11. A metal hollow sphere 47 is secured by welding to a light weight rod 48 and a circular washer 49 having a top beveled wall 50 adapted to mate with beveled wall 46 is welded to the bottom of rod 48. As shown in FIG. 5, the washer 49 is disposed within collar 45. Clearly the floating of sphere 47 on the top surface of molten salt 43 causes washer beveled wall 49 to contact collar beveled wall 46 thereby preventing passage of more liquid salt through the collar aperture 51 into the cell. As the molten salt level in the cell falls, the sphere 47 falls thereby opening a passage between beveled walls 46 and 50 thereby permitting more molten salt to enter the cell.



Turning to FIG. 1, the portion of the I-beam 10 between transverse walls 22 and 21 is provided with an aperture 52 in side wall 15. This aperture is used for removal of dross 53 in a manner to be described. A conventional U-shaped pipe trap 54 is secured in aperture 52 and being filled with molten salt it provides a seal preventing passage of air into the chamber formed between transverse walls 21 and 22.

An important feature of this invention is the provision of means of removal of dross from the cell and the cleaning of the anodes without disturbing the hermetical relationship in the cell. The cell, FIG. 1, is provided with a conduit 55 which passes through suitable apertures in both the conductor plate 26 and the heat insulator blanket 30 in an air-tight hermetically sealed manner. A double flanged collar 56 is disposed about conduit 55 in both of the apertures passing through the respective conductor plate 26 and blanket 30 to effect a hermetical seal at these apertures. Conduit 55 is used for removing the produced chlorine from the cell to other sections of the plant like heat exchangers where the temperature of the chlorine is reduced sufficiently to permit its further processing. No air is permitted to enter the cell through conduit 55 at any time. In starting operation the air present in the cell and in the connecting pipe lines is evacuated through conduit 55 and after the air is removed, argon gas is passed into the cell through conduit 55 to replace the air removed.

The means for removal of dross and for cleaning the anodes under hermetically sealed conditions comprise a pair of electromagnetically operated elements operated under hermetical conditions in an air-tight chamber and out of contact with air.

To this end the rear end of the I-beam 10 is provided with a chamber formed between rear end wall 20X and transverse wall 21.

An outlet hole 57, having a beveled opening, is provided in cross-plate portion 12Y between plate 21 and end plate 20X. A tall cylindrical sleeve 58 of suitable material is provided with four apertures 59 spaced 90 degrees apart and with a beveled nose 60 normally disposed in beveled outlet hole 57.

As shown in FIG. 1, conductor plate 26 is provided with a suitably large hole to permit sleeve 58 to pass through and a graphite cap 61 having a suitably tall cylindrical portion 62 having a bottom integral flange 63 is bolted about the said large hole by means of bolts 64.

The cap 61 is air cooled exteriorly by means of a fan (not shown) and the interior wall of the cap is provided with a top circular electromagnetic coil 65 and a bottom circular electromagnetic coil 66 suitably spaced away from coil 65 as not to interfere therewith electromagnetically.

The top of the sleeve 58 is provided with a weighted plug 67 secured within the sleeve as by welding. A circular collar 68, subject to magnetic attraction, is welded to the sleeve 58 below the end thereof at a point suitable for attraction by the electromagnet 65 upon the latter being energized.

A ring 69, is slidably disposed about sleeve 58 (FIG. 7) so that it rests on the integral holding sleeve lugs 70. The pair of lugs 70 keeps ring 69 suitably above the surface of cross-plate wall 12Y.

The ring 69 is disposed normally below apertures 59 but may be raised to close apertures 59 as will be described hereinafter.

The ring 69 is secured by welding to four upright standards 71. The tops of standards 71 are secured together by a metal ring 72 subject to magnetic attraction which ring is suitably energized by the lower electromagnet as desired. The ring 72 is also of sufficient weight to normally keep lower ring 69 below the level of the molten lead.

The I-beam outlet 57 leads to a conduit 73 which is provided with a constricted throat 74 at a suitable dis-

tance away. The molten lead containing sodium metal dissolved therein as an alloy is poured into retort **75** which is heated by flame **76** to drive off alkali metal vapors, for example sodium vapors, through top vapor conduit **77X** to a collecting tank (not shown).

The lead free from sodium or other alkali metal is pumped through conduit **77** and pump **78** to a reservoir **79** located at a suitable height to form a head giving a sufficient steady flow of molten lead **13** through conduit **80** into the cell.

In order to clean dross from the cell the sleeve **58** is rapidly lifted by energizing the electromagnetic coil **65** which attracts sleeve ring **68** thereby lifting the sleeve **58** and the slidable ring **69** disposed on the sleeve lugs **70**.

This lifting of the sleeve **58** interrupts the normal flow of molten lead through apertures **59** and thence into conduit **73** and instead permits a relatively more rapid flow of molten lead into aperture **57** and conduit **73**. The level of molten lead drops in the chamber located between transverse plates **20X** and **21** as well as in the chamber located between transverse plates **21** and **22** thereby permitting passage of the molten floating salt adjacent to refractory **19X** and the dross thereon under transverse plate **22**. The molten salt with the dross pass into the chamber therebeyond having the aperture in the side wall **15**. Release of the energizing action, acting on the top electromagnetic coil, causes the weighted sleeve to fall and cover the outlet aperture in the cross plate. This causes the level of the dross and molten salt to rise in the said chamber and to pass out of the aperture in wall **15**.

Where it is desirable to clean the bottom surface of the anodes **31**, the cell current is turned off and the bottom electromagnet is energized. This causes the slide ring to rise and cover the sleeve apertures **59**. The result is that the liquid level of the molten lead rises to touch and wash the undersurfaces of the anodes **31** in the cell. In operation the anodes become fouled due to sulfur dioxide formation thereon, encrustations due to foreign matter in the salt, etc. The flow of heavy lead against these gaseous inclusions and deleterious deposits cleans the anodes **31**.

After the lead has washed the anode undersurfaces, the energizing action on the lower electromagnet is released and the ring falls downwardly to its normal position on lugs **70** of the sleeve thereby uncovering sleeve apertures **59** to permit the molten lead to pour therethrough. The cell current is then turned on and the operation of the cell is resumed with the cleaned anodes.

This invention can be modified by the use of a mechanical means of opening and closing outlet port **57**. The mechanical means may be lever-operated means, operating through conductor plate **26** in an air-tight manner.

This invention has been described generically as it is of broad scope and not to be limited to the illustrative example shown herein. For example, this cell is also usable in the manufacture of lead/sodium alloy required in the production of tetraethyl lead. In such an application, there would be no need for chamber **75**, nor for return pipe **77**, nor for pump **78**.

While the heating of the electrolytic cell of this invention is schematically shown by means of a gas furnace, other means of heating the cell are preferred as they will not injure the body. The heating of I-beam **10** by means of combustion gases, in contrast to heat application by use of direct flame, is preferred; also, the heating of I-beam **10** may be done by infrared radiation.

It is a feature of this invention that the conductor plate **26**, disposed over the anode chamber, is of integral construction devoid of anode receiving apertures used in the prior art cover plates.

It is another important feature of this invention that the salt is fused outside of the cell and is introduced into the hermetically sealed cell without the introduction of any air therewith. Thus the salt is melted in a unit outside of the cell and is introduced into the cell as a liquid, free from air and moisture. The elimination of air and of moisture within the apparatus of this invention prevents anode wear in the chamber and also prevents explosions from chemical combination of hydrogen with chlorine and from chemical combination of oxygen with sodium, all disadvantages in prior art electrolytic cells. Also the air and water free operation of this apparatus permits the employment of a high current density up to twenty or more amperes per square inch of anodic surface, compared to the current density conventionally employed of approximately three amperes per square inch of anodic surface. Thus this invention relates to a cell which will produce about seven times more end-products such as chlorine and alkali metal, compared to the conventional cell of substantially the same size.

The dross **53** within the anode chamber is generally of such a nature as to be disposed floatingly upon the lead **13** but it may also be of such a nature as to be disposed both upon the lead **13** and the molten salt **43**.

I claim:

1. An air-tight electrolytic cell for air free and moisture free electrolysis of molten alkali metal salts comprising a substantially rectangular three chambered cell having a front wall and a rear wall and a bottom, a first transverse wall disposed in spaced apart relationship to the front wall of the cell and above said cell bottom to form an electrolytic chamber having a rear transverse wall having a passageway therebeneath, a second transverse wall disposed in spaced relationship to said first transverse wall to form a dross removal chamber, said second transverse wall being disposed in space-relationship to the rear wall of said cell to form a liquid metal removal chamber and above said bottom to form a passageway, an electrically conducting air-tight cover plate for said cell disposed over said three chambers in air-tight relationship thereto, means for insulating electrically said cover plate from the cell walls, and means for securing hermetically said cover plate to said cell walls.

2. The cell of claim 1 comprising anodes removably suspended from the under surface of said cover plate.

3. The cell of claim 2 wherein the bottom has a sloped portion disposed beneath said first transverse wall to form a reservoir in said dross removal chamber, a side wall of said dross removal chamber having an aperture for selective removal of dross.

4. The cell of claim 3 comprising means for continuously introducing air free and moisture free molten cathode metal to one end of the bottom of the electrolytic chamber and means for introducing intermittently air free and moisture free molten alkali metal salt to said one of the bottom of said electrolytic chamber.

5. An air-tight electrolytic cell for air free and moisture free electrolysis of molten alkali metal salts comprising a substantially rectangular three chambered cell having a front wall and a rear wall and a bottom, a first transverse wall disposed in spaced apart relationship to the front wall of the cell and above said cell bottom to form an electrolytic chamber having a rear transverse wall having a passageway therebeneath, a second transverse wall disposed in spaced relationship to said first transverse wall to form a dross removal chamber, said second transverse wall being disposed in space-relationship to the rear wall of said cell to form a liquid metal removal chamber and above said bottom to form a passageway, an electrically conducting air-tight cover plate for said cell disposed over said three chambers in air-tight relationship thereto, means for insulating electrically said cover plate from the cell walls, and means for securing hermetically said cover plate to said cell walls, said anodes being removably suspended from the undersurface of said cover plate, said bottom having a sloped

portion disposed beneath said first transverse wall to form a reservoir in said dross removal chamber, a side wall of said dross removal chamber having an aperture for selective removal of dross, means for continuously introducing air free and moisture free molten cathode metal to one end of the bottom of the electrolytic chamber and means for introducing intermittently air free and moisture free molten alkali metal salt to said one end of the bottom of said electrolytic chamber, electromagnetic gate means disposed in the outlet orifice of the third or molten metal removal chamber for selective electromagnetic removal of molten metal whereby the operating faces of said anodes are washed free of deleterious occluded material.

6. The cell of claim 5 comprising enclosed means for evaporating alkali metal from said molten cathode metal, and means for re-cycle pumping of alkali metal free molten cathode metal to said electrolytic chamber.

7. An air-tight electrolytic cell for air free and moisture free electrolysis of molten alkali metal salts comprising a substantially rectangular three chambered cell having a front wall and a rear wall and a bottom, a first transverse wall disposed in spaced apart relationship to the front wall of the cell and above said cell bottom to form an electrolytic chamber having a rear transverse wall having a passageway therebeneath, a second transverse wall disposed in spaced relationship to said first transverse wall to form a dross removal chamber, said second transverse wall being disposed in space-relationship to the rear wall of said cell to form a liquid metal removal chamber and above said bottom to form a passageway, an electrically conducting air-tight cover plate for said cell disposed over said three chambers in air-tight relationship thereto, means for insulating electrically said cover plate from the cell walls, and means for securing hermetically said cover plate to said cell walls, said anodes being removably suspended from the undersurface of said cover plate, said bottom having a sloped portion disposed beneath said first transverse wall to form a reservoir in said dross removal chamber, a side wall of said dross removal chamber having an aperture for selective removal of dross, means for continuously introducing air free and moisture free molten cathode metal to one end of the bottom of the electrolytic chamber and means for introducing intermittently air free and moisture free molten alkali metal salt to said one end of the bottom of said electrolytic chamber, the means for introducing intermittently said molten salt being a vertically disposed captively held float valve automatically operated by the level of the molten salt in the electrolytic chamber.

8. The cell of claim 7 wherein the cell cover plate insulating means are mica sheets and wherein the cell cover plate is a graphite plate having an external coating of air impervious and moisture impervious glass.

9. The method of electrolyzing alkali metal salts in the absence of air and moisture comprising replacing with an inert gas the air of a three chamber electrolytic cell with one chamber being the electrolysis chamber, the second chamber being the dross removal chamber and the third chamber being the liquid cathode removal chamber; removing dross from said chamber through a liquid seal; distilling off alkali metal from the molten cathode and recycling it back to the cell in the absence of air; introducing air free and moisture free molten alkali metal salt into the electrolytic chamber and intermittently washing the operating faces of the anodes by movement of said molten cathode in said cell in the absence of contact with air.



10. An electrolytic cell for the continuous electrolytic decomposition of a molten alkali metal salt in the presence of a molten lead cathode flowing through said cell to absorb the positive component of said salt released during electrolysis which comprises an air-tight cell body having a bottom to support the molten lead cathode, first air-tight inlet means for introduction of said salt in molten form, second air-tight inlet means for introducing said lead cathode in molten form, air-tight outlet means for removal of said molten lead cathode containing said positive component, means for removing said positive component by vaporization from said molten lead cathode and means for returning said molten lead cathode to said second inlet means.

11. An electrolysis cell for electrolyzing a molten alkali metal salt in the presence of a molten lead cathode, the combination of a cell body having a bottom to support said molten lead cathode, a cover for said body, and anodes depending from said cover, said anodes having support means engaging said cover and extending at most only partially through said cover, and electrical current supply means connected to said cover, whereby electrical current is supplied to said anodes through said cover.

12. An electrolysis cell as defined in claim 11, wherein said cover is formed from graphite.

13. An electrolysis cell as defined in claim 11, wherein said support means are removably connected to said cover.

14. An electrolysis cell as defined in claim 11, wherein said support means are removably connected to said cover and are removably connected to said anodes.

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