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### PROVISIONAL SPECIFICATION

#### "IMPROVEMENTS IN OR RELATING TO ALKALINE MERCURIC OXIDE CELLS"

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAJ MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED  
BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

*The following Specification describes the nature of this invention*

This is an invention by Dr. Michael Angelo Vincent Devanathan formerly Scientist, Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, (Citizen of Ceylon), Address : Head, Physical Chemistry Division, CISIR, Colombo-7, Ceylon, and Shri Narayanan Ramasamy, Senior Scientific Assistant, Shri Srinivasan Venkatesan, Junior Scientific Assistant, Shri Veeraraghava Aravamuthan, Scientist, Shri Paul Nityanandan John, Senior Scientific Assistant, and Shri Srinivasan Sarangapani, Junior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, all Indian citizens.

The specification relates to improvements in or relating to the fabrication of alkaline mercuric oxide cells.

Hitherto it has been customary to prepare mercuric oxide cells where (a) mercuric oxide is obtained from mercury through the pyrolytic treatment of mercuric nitrate, a product of the reaction between mercury and excess nitric acid, (b) zinc powder of high purity of suitable mesh size mostly the condensation product in the retorts and (c) cylindrical cans of zinc obtained by extrusion of the metal to desirable wall thickness and desirable sizes which functions only as container, cathode bobbins of varying dimensions, zinc powder mounted on paper/sintered zinc functioning as anode arranged in a concentric pattern.

These suffer from the disadvantages that (a) such process involves the danger of exposing the personnel too much to the vapours of mercuric compounds. In addition to that, time and temperature are to be carefully controlled or else over heat may lead to loss of material and in cases of incomplete reaction, it leads to formation of mercurous or lower oxide or compounds. The finer dry particles are liable to be lost as dust during roasting. The pyrolytic product is a hard sintered mass which has to be first pulverised to the necessary mesh size and again ball-milled with acetylene black for mixing involving more labour and dangers of mercurial poisoning.

(b) Although there are several procedures for obtaining pure zinc such as by distillation followed by condensation or by electrolysis of compounds of zinc the most simple and reproducible procedure for the preparation of zinc in desired particle size maintaining at the same time purity and performance guaranteed both for intermittent as well as continuous current drains in cells has not been known. Since the success of the batteries depends on the quality of the zinc

anode, an economical method of producing the same is essential.

(c) Zinc cans produced by extrusion require the use of intricate machinery, involve waste of zinc in the process and have limited utility in assembling numerous and odd sized batteries.

To obviate the above mentioned disadvantages, the invention has the following features :

(a) The present method entails a wet process involving the precipitation of fine particles from mercuric nitrate solution by adding just the required amount of alkali or carbonate or bicarbonate of sodium or potassium or mixtures thereof.

(b) Preparation of zinc of analar grade and purity by aqueous electrolysis of chloride bath under conditions of powder deposit which can be scraped and sieved to required mesh size. The amalgamation is done by treatment with mercury under controlled condition of acidity.

(c) Design of the electrodes in the manner that makes possible the development of flat cells embodying the principle of the matrix electrode covered by the Patent Specification No. 98157.

The following briefly describes the various steps of the invention :

(a) The mercuric oxide powder that is obtained by precipitation is heated till vapours of water are completely expelled. The dry powder is cooled and mixed with 2 to 10 percent acetylene black/graphite/any conducting variety of carbon and used as cathode material to be pressed on the Ni/tin plated metallic grid such as mild steel with a suitable binder such as polyvinyl alcohol, starch and the like (2 to 5 percent).

(b) The electrolytic zinc powder that is obtained is triturated to required particle size (mesh—401—00) and amalgamated by treatment with required quantity of mercury (2 to 18 percent) in the presence of strong mineral acids. The product is kept at a temperature of 40 to 80°C for two to four hours, then washed thoroughly to free from mineral acid and dried at room temperature. Necessary quantity of binder is added and the paste pressed on to the zinc plated metal grid.

(c) The alkali treated cellulose separator which functions also as the electrolyte reservoir is wound round the cathode.

(d) Through the proper design of the electrodes, standard units of the cathode and anode can be made (typical size 5.0 cm. + 2.0 cm.). These are arranged alternately, the number of such electrodes depending

upon the ampere hour capacity of the current drain expected from the cell. The assembly is then inserted into a suitable alkali resistant plastic container, allowed to soak with electrolyte in the normal manner and then sealed.

The following examples typically illustrate the invention :

#### Example 1

For low capacity high tension batteries, cells with one cathode, two anode assembly of overall dimensions 5.0 cm.  $\times$  2 cm.  $\times$  1 cm. can be stacked and connected in series. These can be fitted into plastic containers leading to reduction in weight and saving in zinc. In the case of conventional design, the cells are cylindrical zinc can type.

#### Example 2

Increase in capacity of cells can be easily achieved by stacking more electrodes of the basic size and have a multiple electrode set-up. Then it avoids the necessity to resort to different sized cans, bobbin making for different capacity cells, i.e. 2 AH and 10 AH cells.

The following are the among the main advantages of the invention :

1. The dangerous and involved process of mercuric oxide production is made simpler and economical.
2. A uniform, gray, amalgamated pure zinc powder is available for anode fabrication.
3. Method of fabrication is greatly simplified due to design of the electrodes.
4. By using a container of plastic, the wastage of zinc in the form of conventional extruded zinc container is eliminated.

5. The ampere hour capacity can be stepped up by simply stacking more number of electrodes without resorting to different machinery and tools for bobbin making and container manufacture etc.

The plate type assembly is ideally suitable for small flat cells for high tension purposes.

R. BHASKAR PAI  
PATENTS OFFICER,

Council of Scientific and Industrial Research.

Dated this 20th day of June, 1968.

## COMPLETE SPECIFICATION

"IMPROVEMENTS IN OR RELATING TO ALKALINE MERCURIC OXIDE CELLS"  
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT  
(ACT XXI OF 1860)

*The following Specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.*

This is an invention by Dr. Michael Angelo Vincent Devanathan, formerly Scientist, Central Electrochemical Research Institute, Karaikudi-3, (Tamil Nadu, India) Citizen of Ceylon : Address : Head, Physical Chemistry Division, CISIR, Colombo-7, Ceylon, and Shri Narayanan Ramasamy, Senior Scientific Assistant, Shri Srinivasan Venkatesan, Junior Scientific Assistant, Shri Veeraraghava Aravamuthan, Scientist, Shri Paul Nityanandan John, Senior Scientific Assistant and Shri Srinivasa Sarangapani, Junior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, Tamil Nadu, India, all Indian Citizens.

This invention relates to improvements in or relating to the fabrication of alkaline mercuric oxide cells.

Hitherto it has been customary to prepare mercuric oxide cells where (a) mercuric oxide is obtained from mercury through the pyrolytic treatment of mercury nitrate, a product of the reaction between mercury and excess nitric acid, (b) zinc powder of high purity of suitable mesh size mostly the condensation product in the retorts and (c) cylindrical cans of zinc obtained by extrusion of the metal to desirable wall thickness and desirable sizes which functions only as container, cathode bobbins of varying dimensions, zinc powder mounted on paper/sintered zinc functioning as anode arranged in a concentric pattern.

These suffer from the disadvantages that (a)-such a process involves the danger of exposing the personnel too much to the vapours of mercury compounds. The consumption of nitric acid often far exceeds the theoretical quantity, the excess being wasted away as nitric fumes. The drying and decomposition of nitrate formed to oxide without sintering of the particles is not possible thereby involving much time and labour for grinding and sizing the product.

(b) Although there are several procedures for obtaining pure zinc such as by distillation followed by condensation or by electrolysis of compounds of zinc, the most simple and reproducible procedure for the preparation of zinc in desired particle size maintaining at the same time purity and performance satisfactory for intermittent as well as continuous current drains in cells has not been known. Since the success of the batteries depends on the quality of the zinc anode, an economical method of producing the same is essential.

(c) Zinc cans produced by extrusion require the use of intricate machinery, involve waste of zinc in the process and have limited utility in assembling numerous and odd sized batteries.

The object of the present invention is to obviate the above mentioned disadvantages.

According to the present invention, the alkaline mercuric oxide cell is characterised in that the cathode

and anode consists of matrix of flat electrodes packed with mercuric oxide and zinc powder respectively and are separated by the electrolyte absorbant medium/separator consisting of pre-shrunk cellulosic electrolyte retention medium.

Thus, the anode consists of a matrix electrode of flat type packed with zinc powder and the cathode consists of a matrix of flat electrodes packed with mercuric oxide.

The electrolyte absorbant medium/separator may consist of pre-shrunk cellulosic electrolyte retention medium (separator papers).

The electrodes may be made by pressing the material on to the metallic grid or metallic matrix electrode described in prior Indian Patent Specification No. 98157.

The cathode may be fabricated using mercuric oxide powder prepared by reacting mercury with stoichiometric quantity of nitric acid followed by drying and roasting.

Zinc powder prepared electrolytically, ground and amalgamated, is used for making the anode.

The invention includes within its scope an alkaline mercuric oxide cell which comprises laminar matrix zinc powder packed anode and laminar matrix mercuric oxide packed flat cathode separated by separator paper. The alkaline mercuric oxide cell is laminar and has a rectangular cross section and the anodes and the cathodes are alternately separated by the separator paper in between.

The alkaline mercuric oxide cell is suitable for use with transistorised electronic equipment, walkie talkie equipment, hearing aids and the like for providing a steady voltage of 1.35V—0.95V while giving a constant current of upto 6—8 mA/cm<sup>2</sup>.

Thus, in the invented mercuric oxide cells, the electrodes use the materials mercuric oxide, zinc powder, pre-shrunk cellulosic electrolyte retention medium prepared by special procedures and they are of the flat type and the cell is fabricated by an assembly of the flat electrodes. The electrodes are made by pressing the material on to a metallic grid covered by Patent No. 98157. The anode and cathode materials are also prepared processes described hereinbelow :

(a) The present method entails a wet process involving the precipitation of fine particles from mercuric nitrate solutions by adding the stoichiometric quantity of alkali or carbonate or bicarbonate of sodium or potassium or mixtures thereof. The mercuric oxide powder that is obtained by precipitation is heated till vapours of water are completely expelled. Alternatively, the mercuric oxide may also be obtained in a much simpler manner using nearly theoretical quantity of nitric acid for mercury in the following way. The mercury is reacted with the stoichiometric quantity of concentrated nitric acid in a reaction vessel which favours the cathodic reaction accelerating the dissolution of mercury. The product is dried at a temperature of 30-50° where drying without sintering/agglomeration is favoured. The dry powder is then ignited to get the mercuric oxide of a range mesh size—200.

(b) Preparation of zinc by aqueous electrolysis of chloride bath at a pH less than one using a current

density of 40—60 ASF at a temperature of 45—55°C giving a powder deposit which can be scraped and sieved to required mesh size is done. The amalgamation is done by treatment with 2—15 percent mercury under highly acidic conditions (pH 1).

(c) Design of the electrodes embodying the principle of the matrix electrode covered by the patent specification No. 98157.

The following briefly describes the various steps of the invention :

(a) The dry mercuric oxide powder prepared by the procedures described hereinabove is mixed with 2 to 10 percent acetylene black/graphite/any conducting variety of carbon and used as cathode material to be pressed on to Ni/Sn plated metallic grid such as mild steel with a suitable binder such as polyvinyl alcohol, starch, polytetrafluoroethylene, polystyrene, methyl methacrylate, polyvinyl chloride in suitable dispersion media and the like (2 to 5 percent).

(b) The electrolytic zinc powder that is obtained is triturated to required particle size (mesh—40 +100) and amalgamated by treatment with required quantity of mercury (2 to 18 percent) in the presence of strong mineral acids. The product is kept at a temperature of 40 to 80 C for two to four hours, then washed thoroughly to free from mineral acid and dried at temperature of 25—30 C. The amalgamated powder is mixed with an inert absorbent and non-swelling filler (1—10 percent). The filler can be organic/inorganic. Typical examples include polyvinyl alcohol, carboxymethyl cellulose, alkali treated cellulose fibre, barium sulphite, calcium carbonate, magnesium carbonate, zinc silicate, zinc oxide, zinc carbonate, calcium silicate. The mix is pressed on to the grids in the form of a paste, the paste being made with a binder or plain water.

(c) The alkali treated cellulose paper which functions also as the electrolyte reservoir is wound round the anode and a separator is also employed. The separator must be non-reactive and non-gelating. Separators used may be microporous PVC, nylon, cellophane, treated cotton and the like.

(d) Standard units of the cathode and anode can be made (typical size 5.0 cm. × 2.0 cm.). These are arranged alternately, the number of such electrodes depending upon the ampere hour capacity and the current drain expected from the cell. The assembly is then inserted into an alkali resistant plastic container such as high density polythene, high impact polystyrene, teflon and nylon, allowed to soak with electrolyte in the normal manner and then sealed.

The following is a brief description of how the invention is carried out. The accompanying drawings indicate the various parts that comprise the device and the manner in which it is constructed.

Fig. 1 shows the front view of metal matrix electrode;

Fig. 2 is a side view of the metal matrix electrode ;

Fig. 3 represents the side view of metal matrix packed with mercuric oxide ;

Fig. 4 indicates the side view of (3) with separator paper layer over the mercuric oxide ;

Fig. 5 is a side view of one anode and one cathode;

Fig. 6 shows the alternate adjacent placing of the anode and cathode electrodes (front view);

Fig. 7 shows cut away section of anode ;

and Fig. 8 is a cut away section of a cathode

and Fig. 9 is a view of the pile type arrangement anode and cathode alternatively placed, the total number of which decide the type and capacity of the cell.

The anode A fabricated from a laminar metal matrix 4 packed with zinc powder 5, is covered with a treated separator paper 6. The cathode C is fabricated from laminar metal matrix 1 packed with mercuric oxide 2 and covered with separator paper 3. The anode and cathode electrodes are alternately placed adjacently (Fig. 6) and the device as a whole comprises two cathodes C and three anodes A. The device jointly comprising these parts enclosed in a plastic container of dimension  $2.4 \times 2.6 \times 5$  cm. and containing 5 c.c. of strong 85 percent KOH solution services as the source of electrochemical energy giving a steady voltage of 1.3 V at a high and constant current rating without getting polarised quickly.

#### Example 1

For low capacity high tension batteries, cells with one cathode, two anode assembly of overall dimensions 5.0 cm.  $\times$  2 cm.  $\times$  1 cm can be stacked and connected in series. These can be fitted into plastic containers leading to reduction in weight and saving in zinc. In the case of conventional design, the cells are cylindrical zinc can type.

#### Example 2

Increase in capacity of cells can be easily achieved by stacking more electrodes of the basic size and have a multiple electrode set-up. Then it avoids the necessity to resort to different sized cans, bobbin making for different capacity cells, i.e. 2 AH and 10 AH cells.

The following are among the main advantages of the invention :

1. The involved and costly process of mercuric oxide production is made simpler and economical.
2. A uniform, amalgamated pure zinc powder is available for anode fabrication.
3. Method of fabrication is greatly simplified due to design of the electrodes.
4. By using a container of plastic, the wastage of zinc in the form of conventional extruded zinc container is eliminated.
5. The ampere hour capacity can be stepped up by simply stacking more number of electrodes without

resorting to different machinery and tools for bobbin making and container manufacture.

The plate type assembly suitable flat cells for high voltage battery packs, viz. 90V, 150V.

#### NOTEWORTHY FEATURES

1. A process for the preparation of mercuric oxide cells wherein the electrodes use the materials mercuric oxide zinc powder, pre-shrunk cellulosic electrolyte retention medium and they are of the flat type and the cell is fabricated by an assembly of the flat electrodes. The electrode are made by pressing the material on to a metallic grid covered by Patent No. 98157.

2. The electrodes are fabricated by pressing the material on to a metallic matrix electrode covered by the Indian Patent Specification No. 98157.

3. The cathode is fabricated using mercuric oxide powder prepared by reacting mercury with stoichiometric quantity of nitric acid followed by drying and roasting.

4. The anode uses zinc powder prepared electrolytically, ground and amalgamated.

We claim :

1. An alkaline mercuric oxide cell characterized in that the cathode and anode consist of matrix of flat electrodes packed with mercuric oxide and zinc powder respectively and are separated by the electrolyte absorbant medium/separator consisting of pre-shrunk cellulosic electrolyte retention medium.

2. An alkaline mercuric oxide cell as claimed in Claim 1 wherein the electrodes are made by pressing the material onto the metallic grid or metallic matrix electrode described in prior Indian Patent Specification No. 98157.

3. An alkaline mercuric oxide cell as claimed in Claim 1 wherein the cathode is fabricated using mercuric oxide powder prepared by reacting mercury with stoichiometric quantity of nitric acid followed by drying and roasting.

4. An alkaline mercuric oxide cell as claimed in Claim 1 where zinc powder used is prepared electrolytically which is grounded and amalgamated and is used for making anodes

5. An alkaline mercuric oxide cell substantially as herein described.

R. BHASKAR PAI,  
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Dated this 24th day of February, 1969.

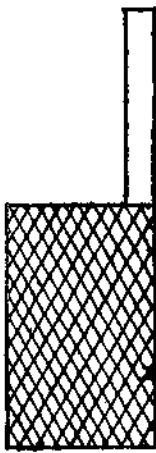


FIG-1



FIG-2

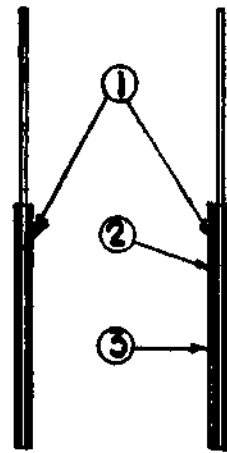


FIG-3



FIG-4

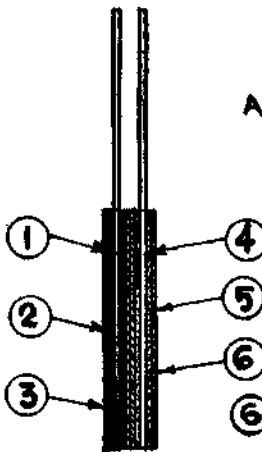


FIG-5

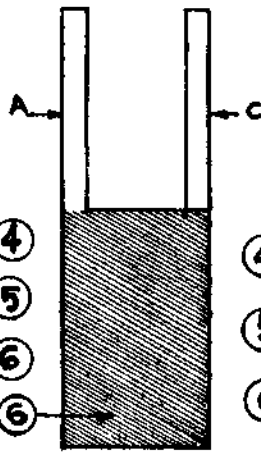


FIG-6

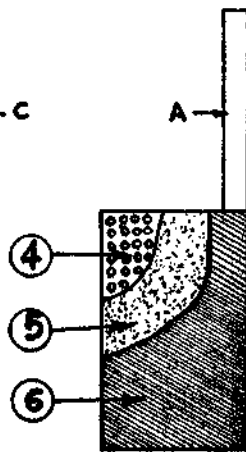


FIG-7

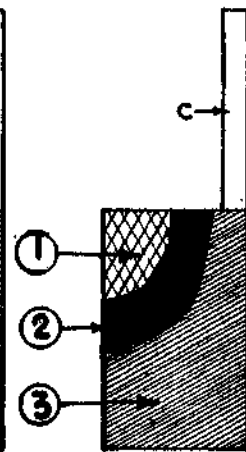


FIG-8

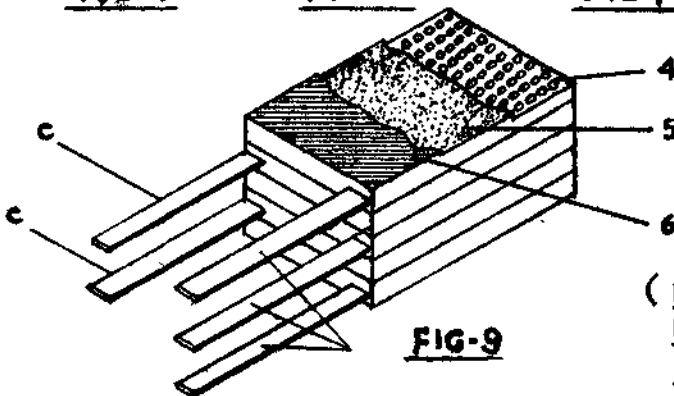


FIG-9

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