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"IMPROVEMENTS IN OR RELATING TO THE ELECTROLYTIC  
PREPARATION OF POTASSIUM CHLORATE"

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

This is invention by HANDADY VENKATAKRISHNA UDUPA, Scientist, SRINIVASA SAMPATH, (Western India Match Company Ltd., Ambaruath), RAMANATHAN VISWANATHAN, Scientist, NARAYANASWAMY SARMA THIAGARAJAN, Senior Scientific Assistant, KAPISTHALAM CHETLUR NARASIMHAM, Scientist, MUTHIA NAGALINGAM, Senior Scientific Assistant, GANESHA GANAPADIGAL SUBRAMANIAN, Junior Scientific Assistant, PERUMAL SUBBIAH, J.S.A., RASAPPA GOUNDER PALANISAMY, J.S.A., SAVARIMUTHU JOHN PETER (Space Science and Technology Centre, Trivandrum), and SUBRAMANIAN PUSHPAVANAM, S.L.A., all except 2nd and 10th Inventor, of the Central Electrochemical Research Institute, Karaikudi, Tamil Nadu, India, all Indian citizens.

PROVISIONAL

The following Specification describes the nature of this invention.

This invention relates to the improvements in or relating to the electrolytic preparation of potassium chlorate making use of a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate.

Hitherto it has been the practice to prepare pure potassium chlorate by the following two methods:

1. Direct electrolytic oxidation of potassium chloride to potassium chlorate using graphite or magnetite anodes, where the cell effluent is cooled to crystallise out the potassium chlorate and the mother liquor is resaturated with potassium chloride before recirculation in the cell.
2. Electrolytic preparation of sodium chlorate from sodium chloride using magnetite anodes, at 90°C followed by the double decomposition of sodium chlorate with potassium chloride. The mother liquor after separating the potassium chlorate contains sodium chloride, potassium chloride and potassium chlorate. The solution is brought to the original concentration by addition of the required quantity of sodium chloride.

The objections to the current practices are as follows:

In the direct oxidation of potassium chloride to potassium chlorate operational difficulties are encountered due to the poor solubility of potassium chlorate which causes incrustation of the salt on the electrodes in the cell thereby resulting in increased voltage. In addition, large volumes of liquor are required to be handled.

The second process mentioned above may not be preferred under Indian conditions since magnetite anodes have to be imported.

The object of this invention is to obviate these disadvantages by adopting the following modified procedure.

The electrolysis is carried out using graphite or lead dioxide anodes and iron or stainless steel cathodes at a temperature ranging between 30°C to 60°C. An anode current density of 1 to 15 amp/dm<sup>2</sup> is used with lead dioxide anode. The starting electrolyte is a mixture of sodium chloride and sodium chlorate mixed in a specific proportion. The electrolyte is circulated at a predetermined rate based on the quantity of electricity passed through the cell. The cell effluent containing a higher concentration of sodium chlorate is double decomposed externally at elevated temperature with a calculated quantity of solid potassium chloride to precipitate out potassium chlorate. The quantity of potassium chloride

added is such that the resulting solution after precipitation of potassium chlorate is brought back to the original composition of cell-feed. This solution which is also saturated with respect to potassium chlorate is recirculated into the cell. The chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the cell effluent. The separated potassium chlorate is washed and dried.

The following typical examples are given to illustrate the invention:

Concentration of electrolyte:	Example I	Example II
Chloride (Na) mol/l	4	4.13
Chlorates (Na + K) mol/l	2	1.97
Addition agent (alkali chromates) g/l	2	2
Anode	Lead dioxide coated graphite carrying 0.5-5 mm thick PbO <sub>2</sub>	Lead dioxide coated graphite.
Cathode	Stainless steel	Mild Steel
Current density (amp/dm <sup>2</sup> )	5	15
Temperature	40°-45°C	60-65°C
pH	6-7	6-7
Current efficiency (%)	75-85%	80%
Cell voltage (V)	3.4	3.5
Energy consumption (kwh/kg of KClO <sub>3</sub> )	6.0-6.5	6.5

The following are among the main advantages of the invention:

1. Sodium chloride instead of potassium chloride is used as the starting material for the process of making potassium chlorate. This enables building up of higher concentration of chlorate in the cell as sodium chlorate and avoids handling of large volumes of solution. The KClO<sub>3</sub> is precipitated by double decomposition with KCl.
2. The new process eliminates incrustation of salt on the electrodes inside the cell.
3. The cell could be operated at temperatures of 30° to 60°C instead of 90°C as in the case of magnetite anodes.
4. The double decomposition is carried out directly with solid potassium chloride.

Price : TWO RUPEES.

5. The potassium chlorate obtained is found to conform to the required specifications.
6. The solution after separating potassium chlorate can be directly fed into the cell since its composition gets adjusted automatically to the cell feed concentration.
7. The current efficiency of the process is high and the energy consumption is also comparable.
8. Graphite or lead dioxide on graphite is indigenously available.
9. The use of lead-dioxide electrode will eliminate recurring consumption of graphite and helps also to operate the cell over a wide range of current density.

### COMPLETE

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

This invention relates to the improvements in or relating to the electrolytic preparation of potassium chlorate making use of a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate and employing either graphite or lead dioxide coated graphite anodes.

Hitherto, it has been the practice to prepare pure potassium chlorate by the following two methods:

- (i) Direct electrolytic oxidation of potassium chloride to potassium chlorate using graphite or magnetite anodes, where the cell effluent is cooled to crystallise out the potassium chlorate and the mother liquor is resaturated with potassium chloride before circulation in the cell.
- (ii) Electrolytic preparation of sodium chlorate from sodium chloride using magnetite anodes at 90°C followed by the double decomposition of sodium chlorate with potassium chloride. The mother liquor after separating the potassium chlorate contains sodium chloride, potassium chloride and potassium chlorate. The solution is brought to the original concentration by addition of the required quantity of sodium chloride.

The objections to the current practices are as follows:

In the direct oxidation of potassium chloride to potassium chlorate operational difficulties are encountered due to the poor solubility of potassium chlorate which causes incrustation of the salt on the electrodes in the cell thereby resulting in increased voltage. In addition, large volumes of liquor are required to be handled.

The second procedure mentioned above may not be preferred under Indian conditions since magnetite anodes have to be imported.

The object of this invention is to obviate these disadvantages by adopting the following modified procedure.

According to the present invention, the process consists of the electrolytic preparation of potassium chlorate from a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate (the chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the effluent) in a cell having graphite or lead dioxide (electrodeposited on graphite substrate) anode and iron or stainless steel cathode, using an anode current density range of 1 to 15 amp/dm<sup>2</sup> at a temperature of 30° to 60°C and alkali chromates as addition agent.

The electrolysis is carried out using graphite or lead dioxide anodes and iron or stainless steel cathodes at a temperature ranging between 30°C and 60°C. An anode current density of 1 to 15 amp/dm<sup>2</sup> is used with lead dioxide anode. The starting electrolyte is a mixture of sodium chloride and sodium chlorate mixed in a specific proportion.

The electrolyte is circulated at a predetermined rate based on the quantity of electricity passed through the cell. The cell effluent containing a higher concentration of sodium chlorate is double decomposed externally at elevated temperature with a calculated quantity of solid potassium chloride to precipitate out potassium chlorate. The quantity of potassium chloride added is such that the resulting solution after precipitation of potassium chlorate is brought back to the original composition of cell feed. This solution which is also saturated with respect to potassium chlorate is recirculated into the cell. The chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the effluent. The separated potassium chlorate is washed and dried.

The modifications introduced, forming part of the present invention, have simplified the process without affecting the current efficiency. The graphite substrate lead dioxide anodes have the advantage of a longer life by 3 to 4 times those of the conventional graphite or magnetite anodes used in potassium chlorate production. High current densities upto nearly 8 times the conventional current densities can be employed leading to less floor space and giving more compact cells with graphite substrate lead dioxide anodes. The processing of the effluent can be carried out without filtration since the cell effluent is clear and free from suspended impurities when graphite substrate lead dioxide anodes are used. The longer life of the lead dioxide anode coupled with the higher current efficiency will have an edge over the conventional processes.

The present invention relates to a process for the electrolytic preparation of potassium chlorate which comprises of the oxidation of sodium chloride to sodium chlorate using a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate and precipitating the chlorate as potassium chlorate by adding solid potassium chloride, wherein graphite substrate lead dioxide anode or graphite anode and stainless steel cathode or iron cathode are used. The chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the effluent. A current density range of 1 to 15 amp/dm<sup>2</sup>, a temperature of 30°C to 60°C are employed with lead dioxide anode.

The use of lead dioxide electrode will eliminate recurring consumption of graphite. The processing of the effluent can be carried out without filtration since the cell effluent is clear and free from suspended impurities when lead dioxide anode is used.

Flow sheet is given in the accompanying drawings.

Typical examples:

	Example I	Example II	Example III	Example IV
Concentration of electrolyte:				
Chloride ( $\text{Na}^+$ ) (mol/l)	4.9	4.1	4.13	4
Chlorates ( $\text{Na}^+ + \text{K}^+$ ) (mol/l)	1.8	2.2	1.97	2
Addition agent (alkali chromates) (g/l)	2	2	2	2
Anode	Graphite	Lead dioxide coated graphite	Lead di- oxide coated graphite (carrying 0.5-5 mm/ thick deposit)	Lead dioxide coated graphite
Cathode	Mild Steel	Stainless steel	Mild steel	Stainless steel
Current amps	5	18	300	800
Anode current density (amp/dm <sup>2</sup> )	2.4	15	15	5
Temperature (°C)	37-40	32-35	60-65	40-45
pH	6-7	6-7	6-7	6-7
Current efficiency (%)	61	71.5	80	75-85
Cell voltage (V)	3.3-3.6	3.8	3.5	3.4
Energy consump- tion (kwh/kg of $\text{KClO}_3$ )	8.1-8.8	8.0	6.5	6.0-6.5

The main advantages of the invention are that (i) sodium chloride instead of potassium chloride is used as the starting material for the process of making potassium chlorate, which enables building up of higher concentration of chlorate in the cell as sodium chlorate, avoids handling of large volumes of solution and eliminates incrustation of salt on the electrodes inside the cell, (ii) the double decomposition is carried out directly with solid potassium chloride, (iii) the current efficiency of the process is high, (iv) the solution after separating potassium chlorate can be directly fed into the cell since its composition gets adjusted automatically to the cell feed concentration, and (v) the use of lead dioxide anode will increase the life of the anode and helps also to operate the cell over a wide range of current density,

The present invention consists in producing potassium chlorate from a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate, using graphite or lead dioxide anode and iron or stainless steel cathode and employing alkali chromates as addition agent and precipitating the chlorate from the cell effluent as potassium chlorate by the addition of solid potassium chloride.

#### We Claim:

1. A process for the electrolytic preparation of potassium chlorate from a mixed electrolyte consisting of sodium chloride, sodium chlorate and potassium chlorate (the chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the effluent) in a cell having graphite or lead dioxide (electrodeposited on graphite substrate) anode and iron or stainless steel cathode, using an anode current density range of 1 to 15 amp/dm<sup>2</sup> at a temperature of 30° to 60°C and alkali chromates as addition agent.
2. A process as claimed in claim 1 wherein the chloride and chlorate concentrations in the electrolyte are maintained between 5 mols and 1 mol per litre respectively in the feed electrolyte and vice-versa in the effluent.
3. A process as claimed in claim 1 wherein graphite or lead dioxide (electrodeposited over graphite) are used as anode and iron or stainless steel as cathode.
4. A process as claimed in claim 1 wherein the electrolytic cell can be operated using wide range of

anode current densities from 1 amp/dm<sup>2</sup> to 15 amp/dm<sup>2</sup> and preferably 5 amp/dm<sup>2</sup> with lead dioxide anode.

5. A process as claimed in claim 1 wherein the electrolytic cell can be operated using a wide range of temperatures from 30°C to 60°C and preferably at 45°C.
6. A process as claimed in claim 1 wherein alkali chromate (2 g/l) is added at the commencement of the electrolysis.
7. A process as claimed in claims 1 to 7 wherein the double decomposition is carried out directly with solid potassium chloride.
8. A process as claimed in claims 1 to 7 wherein the solution after separating potassium chlorate can

be directly fed into the cell as its composition gets adjusted automatically to the cell feed concentration.

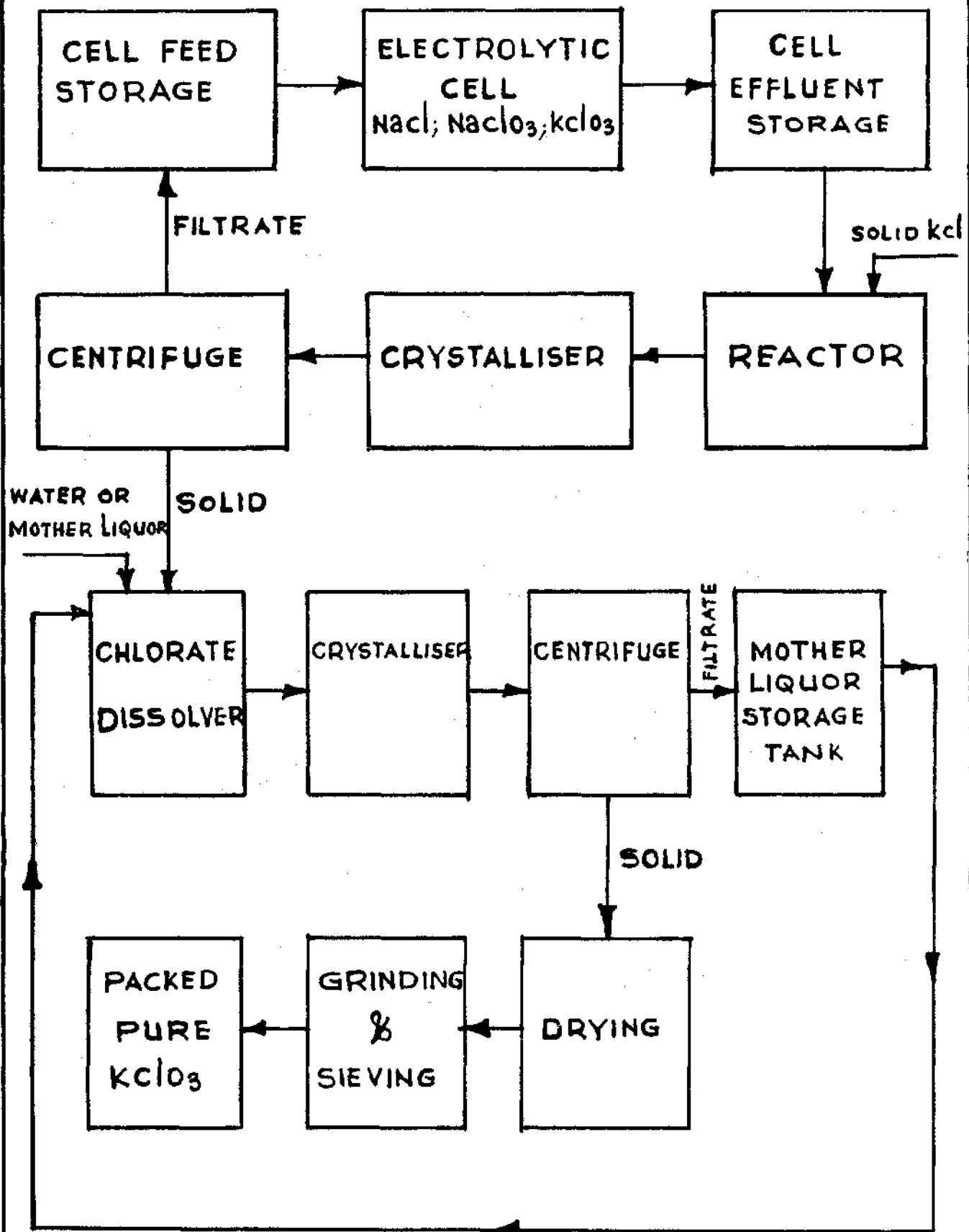
9. A process for the preparation of potassium chlorate substantially as described hereinbefore.

*Dated this 29th day of September 1969.*

Sd.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH  
PATENTS OFFICER

No. 119189.

*R. Bhaskar Pai*( R.B. PAI.)  
PATENTS OFFICER,  
C.S.I.R.