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IMPROVEMENTS IN OR RELATING TO THE ELECTROLYTIC PREPARATION OF POTASSIUM PERCHLORATE.
 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 HANDADY VENKATAKRISHNA UDUPA, Scientist, SRINIVASA SAMPATH,
 Scientist, RAMANATHAN VISWANATHAN, Scientist and MUTHIAH NAGALINGAM, Senior Scientific
 Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, India, all Indian citizens.

This invention relates to improvements in or relating to the electrolytic preparation of potassium perchlorate.

This invention relates particularly to improvements in or relating to the electrolytic preparation of potassium perchlorate making use of potassium chlorate as the starting material.

Hitherto it has been the practice to prepare pure potassium perchlorate by the electrolytic oxidation of sodium chlorate using platinum or lead dioxide anodes followed by the chemical interaction of sodium perchlorate so prepared with potassium chloride. Besides, potassium perchlorate, sodium chloride is obtained as a byproduct. The sodium chloride is recovered by evaporation and recycled to the chlorate cell for electrolytic oxidation to sodium chlorate at a graphite anode.

Attempt at direct electrolytic oxidation of potassium chlorate to potassium perchlorate has been reported in published literature.

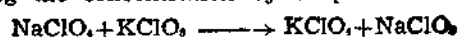
The objections to the current practices are that the conventional process adopted is complicated due to the processing steps involved and the energy consumption for the oxidation of sodium chlorate to perchlorate is higher (3.8 kw hr/kg with platinum anode and 3.2 kw hr/kg at a lead dioxide anode) in view of the fact that sodium chlorate in the bath has to be almost completely depleted with consequent drop in current efficiency (75% at a platinum anode and 60% at a lead dioxide anode).

The direct oxidation of potassium chlorate to perchlorate is unlikely to become a commercial success as the process is beset with operational difficulties viz., the incrustation of the anode with perchlorate and handling of large volumes of dilute solutions due to the low solubility of potassium chlorate at normal temperatures of operation and the almost insoluble nature of potassium perchlorate.

The object of this invention is to obviate the above-mentioned drawbacks by preparing potassium perchlorate making use of potassium chlorate as the starting material instead of potassium chloride to provide the potassium ions as in the conventional process and by carrying out the oxidation of potassium chlorate indirectly employing a mixed electrolyte containing sodium and potassium chlorates and perchlorates with platinum or lead dioxide anodes and iron or stainless steel cathodes.

The main finding pertaining to the invention is the use of a mixed electrolyte in the appropriate concentrations of the various constituents in order to bring above the oxidation in an efficient way.

Such use of a mixed sodium-potassium chlorate-perchlorate electrolyte has not been attempted before, as evidenced by a survey of published literature on the subject. The invention results in the following advantages: (i) The use of potassium chlorate as a starting material (instead of potassium chloride and sodium chlorate) does not involve the intermediate processing to isolate a solid product (like sodium chloride) after separating potassium perchlorate. The solution remaining after the separation of the precipitated potassium perchlorate contains sodium chlorate and this can be recycled into the electrolytic cell after adjusting the concentration by evaporation.



(ii) Sodium chlorate and perchlorate are highly soluble and hence high concentrations are used in the process and as a consequence the volumes of liquors handled are small. (iii) As the chlorate concentration in the cell is maintained at a high level (1 to 4 mols/litre) the current efficiency remains very high (about 70 to 75% with lead dioxide anodes and 90-95% with platinum anodes) and the energy consumption for the product low (2.4-3.0 kw hr/kg with lead dioxide anodes and 2.5-3.5 kw hr/kg with platinum anodes). (iv) The product made by the process is free from chloride and low in chlorate (less than 0.05%).

According to the present invention, the process for the electrolytic preparation of potassium perchlorate consists in electrolysis of a solution containing sodium and potassium chlorates and perchlorates in a cell having lead dioxide (massive or electrodeposited on substrates like graphite, carbon or other suitable material) or platinum as anode and iron or stainless steel as cathode, using an anodic current density of 5 to 40 amp/dm² at a temperature of 30° to 60°C (using sodium fluoride as addition agent with lead dioxide anode), the said electrolysis being followed by a reaction with saturated potassium chlorate solution to precipitate potassium perchlorate.

In the case of lead dioxide anode, addition of sodium fluoride (0.5 to 5 g/l) is necessary. A subsidiary advantage of the process developed is that intermediate isolation of a solid product from the mother liquor after separating potassium perchlorate is avoided.

A flow sheet of the process is given in Fig. I of the accompanying drawings.

The following typical examples are given to illustrate the invention:

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Concentration of Electrolyte :	Example I	Example II	Example III
Chlorates (Na + K) (mol/l) :			
Cell feed	2.7	3.1	3.3
Cell effluent	1.1	0.9	0.7
Perchlorates (Na + K) (Mol/l) :			
Cell feed	1.5	1.3	0.9
Cell effluent	3.1	3.5	3.5
Addition agent (NaF) (g/l)	2.0
Volume of electrolyte used (l).	0.9	15.0	536
Anode material	Lead dioxide coated graphite	Platinum	Platinum
Cathode material	Stainless steel	Stainless steel	Stainless steel
Anode current density (amp/dm ²)	20	25	33.3
Temperature (°C)	35°-40°	35°-40°	50°-55°
Current (amp)	35	150	800
Duration of the experiment (hrs)	3	12	120
Cell voltage (volts)	4.6	6.1	6.5
Current efficiency (%)	74	90	94
Energy consumption (kw hr/kg)	2.4	2.6	3.5

Based on the conditions arrived at in the laboratory (Examples I and II are typical of the laboratory runs), one 800 amp cell was set up and operated. Example III above illustrates results obtained from this larger cell.

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Advantages—The present invention is an improvement over the conventional process for the production of potassium perchlorate in that the current efficiency and energy consumption are more favourable and also it is more convenient and simpler to operate involving less equipment.

Summary—The present invention makes use of potassium chlorate as the starting material for the production of potassium perchlorate. Electrolysis using a mixed electrolyte (containing sodium, potassium chlorates and perchlorates) effects an indirect oxidation of potassium chlorate to potassium perchlorate. With the concentrations of the electrolyte remaining high, volumes handled are less and energy consumption lower.

We claim :

1. A process for the electrolytic preparation of potassium perchlorate, which consists in electrolysing a solution containing sodium and potassium chlorates and perchlorates in a cell having lead dioxide (massive or electrodeposited on substrates like graphite, carbon or other suitable material) or platinum as anode and iron or stainless steel as cathode using an anodic current density of 5 to 40 amp/dm² at a temperature of 30-60°C (using sodium fluoride as addition agent with lead dioxide anode), the said electrolysis being followed by a reaction with saturated potassium chlorate solution to precipitate potassium perchlorate.

2. A process as claimed in Claim 1 wherein the electrolyte consists of 1 to 4 mol/l sodium and potas-

sium chlorates in solution and 1 to 4 mol/l sodium and potassium perchlorates in solution.

3. A process as claimed in Claim 1 or 2 wherein the anode for the electrolytic oxidation is platinum or lead dioxide (massive or electrodeposited on suitable substrate as graphite, carbon etc.) and the cathode is iron or stainless steel in the case of lead dioxide anode, addition of sodium fluoride (0.5 to 5 g/l) is necessary).

4. A process as claimed in any of the above claims wherein the liquor obtained after electrolysis is continuously withdrawn and treated with saturated potassium chlorate solution to separate out the potassium perchlorate and the mother liquor recycled into the electrolytic cell after concentration to the original level.

5. A process for the preparation of potassium perchlorate carried out by a combination of the steps as substantially described hereinbefore.

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RESEARCH.

Dated this 3rd day of August 1966.

COMPLETE SPECIFICATION

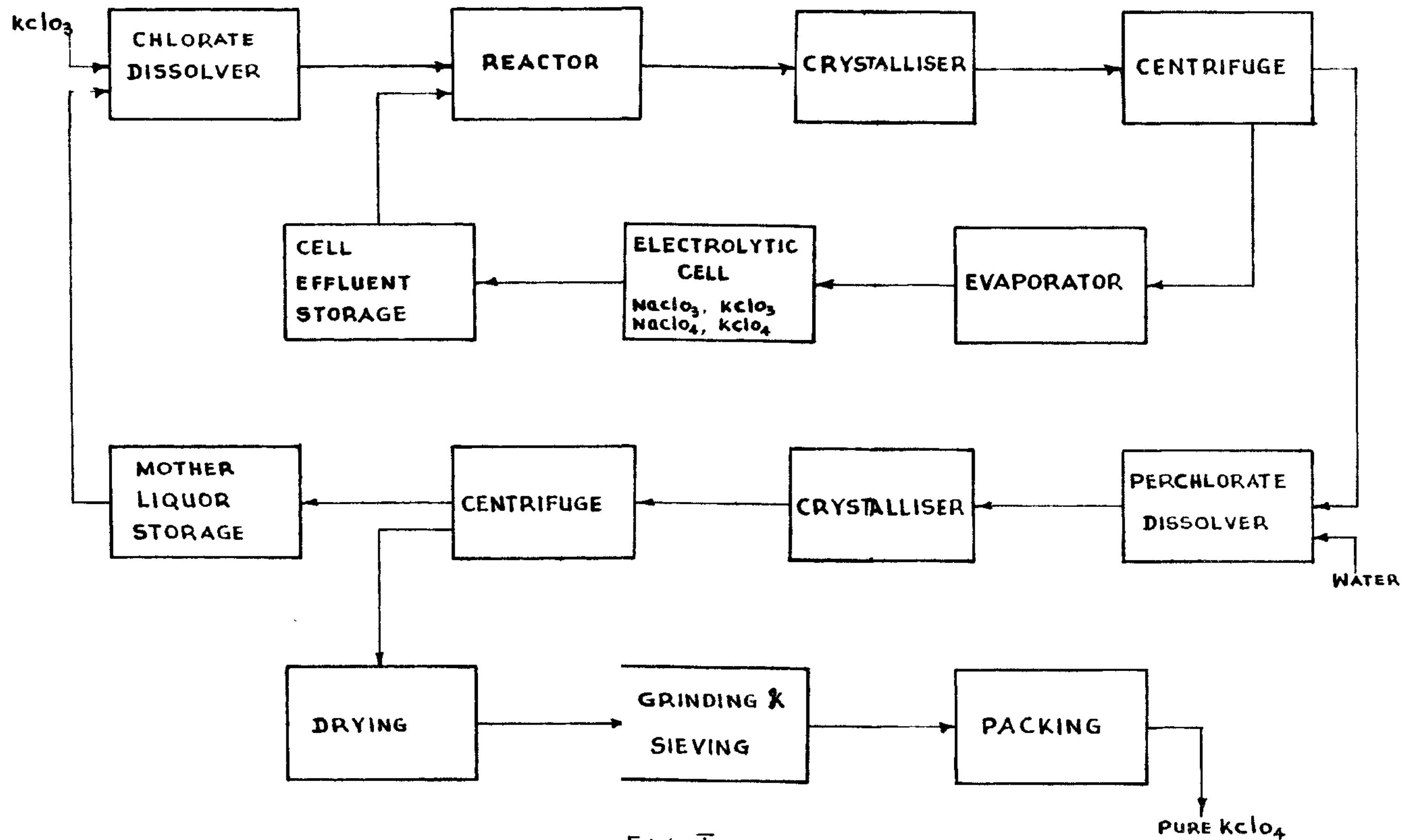


Fig. I

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