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PROCESS FOR THE PRODUCTION OF ANHYDROUS MAGNESIUM CHLORIDE

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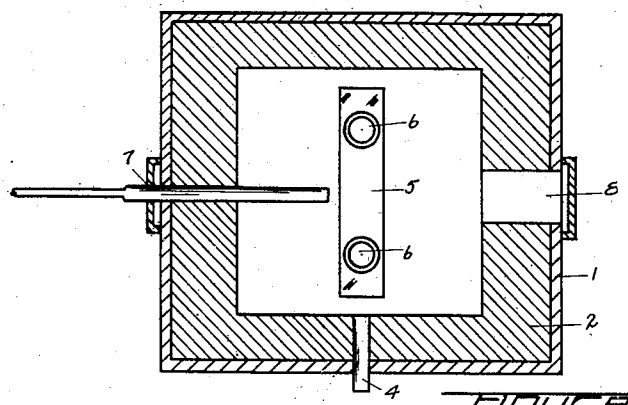


Figure 2

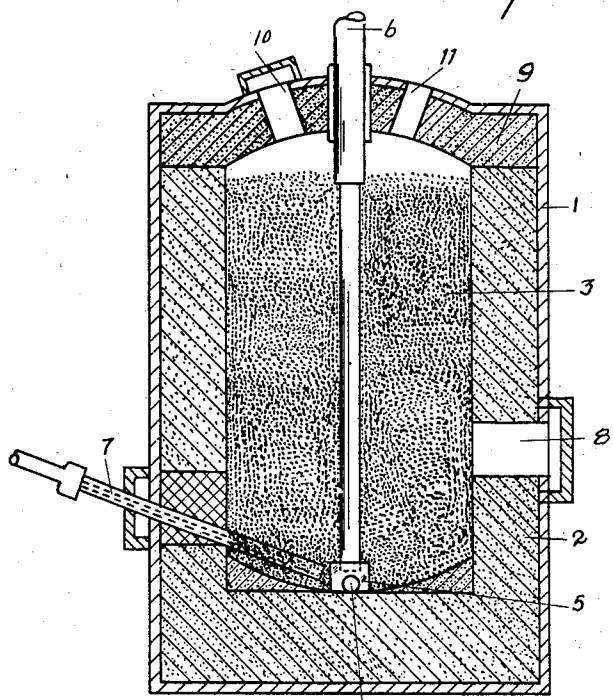


Figure 1

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PROCESS FOR THE PRODUCTION OF ANHYDROUS MAGNESIUM CHLORIDE

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7 Claims. (Cl. 23—91)

This invention relates to a process for the production of magnesium chloride and is particularly directed to the production of substantially pure, anhydrous magnesium chloride.

Processes for the electrolysis of fused magnesium chloride to obtain metallic magnesium therefrom have been in relatively widespread use for many years past. It is also well known that magnesite (magnesium carbonate) which occurs in nature alone or in compounds, such as dolomite ($MgCO_3.CaCO_3$), can be converted into chloride form and the resulting magnesium chloride electrolyzed to recover the metallic magnesium in substantially pure form.

For the successful electrolytic separation of magnesium from molten magnesium chloride, the magnesium chloride must be substantially free from solid impurities and preferably should contain no water and not more than 1% solids. Magnesium chloride of this quality is suitable for direct addition to the electrolytic cell, and recoveries of the order of 90% of the magnesium with high current efficiency can be obtained. To illustrate the importance of using a chloride substantially free from solid impurities and water, which latter would be decomposed to form magnesium oxide, it is found that a recovery of only about 80% of the magnesium can be obtained from magnesium chloride containing 2% solids, while a recovery of only 60% can be obtained from magnesium chloride containing 4% solids. These lower recoveries are due to the retention of electrolyte and of metallic magnesium by magnesium oxide and other insoluble impurities which it is necessary to remove from the cell during operation.

It is manifest from the published literature relating to processes for the chlorination of magnesium oxide, such as exemplified on page 175, vol. III of "Handbuch der Technischen Elektrochemie," by Engelhardt, 1934, that considerable difficulty has been encountered in the development of processes for the production of anhydrous magnesium chloride from magnesite.

Among the principal difficulties encountered in such processes is that of effecting the substantially complete combination of chlorine with the magnesium of the magnesium oxide.

A further difficulty arises from the tendency of the magnesium oxide material to disintegrate during chlorination, whether the material is in the form of lumps of magnesite or in the form of briquettes of finely ground magnesia, bonded by known methods involving the use of magnesium chloride, carbonaceous materials and the like. These magnesium oxide materials tend to disintegrate into particles which are carried away with the molten magnesium chloride, with the result that the whole charge is converted into an extremely viscous mass which retards, if it does not completely prevent, the further chlorination of such of the unchlorinated pieces of the charge as are covered with this viscous material. The magnesium oxide thus entrained impairs the purity of any product obtained and necessitates its further treatment to make it suitable for electrolysis. Similar difficulties are caused by contamination of the molten magnesium chloride with finely ground coke or other forms of carbon whether bonded or not.

A still further difficulty arises from the development of high temperature zones in the electrically heated chlorinating furnaces of the prior art, which tend to volatilize the magnesium chloride as it is formed. The volatilized magnesium chloride may be carried off in the exit gases from the furnace or condensed to an appreciable extent in colder sections of the charge or furnace to form viscous zones, thus decreasing the efficiency of the operation. Under such circumstances, very little, if any, magnesium chloride is recovered in molten form at the tap-hole.

Reference is made in the prior art to the exothermic reactions:

1. $MgO+C+Cl_2=MgCl_2+CO$
2. $2MgO+C+2Cl_2=2MgCl_2+CO_2$
3. $MgO+CO+Cl_2=MgCl_2+CO_2$

It appears that the above reactions have been the subject of detailed investigation for a number of years past but due to the above mentioned difficulties in operation, processes based on these reactions have not appeared in practice until recently.

In these processes, expedients have been suggested to obviate the disadvantages arising out of the entrainment of particles of magnesium oxide by the molten magnesium chloride in its downward course through the charge. Filtering or irrigation beds have been suggested to serve the purpose of promoting the chlorination of the entrained magnesium oxide particles suspended in the molten magnesium chloride and of separating out the impurities and any unchlorinated magnesium oxide particles. Alternatively, it has been suggested, in order to avoid the above disadvantages, that chlorination should be carried out at a temperature below the melting point of the magnesium chloride, conditions being arranged so that the chloride will fuse and run

quickly off the unreacted material of the charge at the zone of contact with the irrigation bed, which is maintained at a temperature above the melting point of the chloride. The quick separation of the molten chloride, immediately prior to entering the irrigation bed, is recommended, presumably to prevent the magnesium chloride from picking up particles of magnesium oxide. However, operating under these restricted conditions involves considerable difficulties and the disadvantage that residue, resulting from the use of excess magnesium oxide, excess coke or impure magnesium oxide, would accumulate above the electrically heated irrigation bed, making continued operation impracticable and, furthermore, chlorinating at such low temperatures entails the disadvantage that a slower rate of chlorination is inevitable.

We have found that it is possible to effect the chlorination of magnesium oxide in such a manner that the magnesium chloride is recovered in substantially pure and highly fluid form, while obtaining a high yield and avoiding the difficulties hitherto associated with the chlorination of magnesium oxide.

One of the principal features of our invention resides in the preliminary treatment to which the magnesium oxide is subjected before it is charged into the chlorinating furnace. The magnesium oxide is given a preliminary treatment to prepare it in a form in which it is porous and in which it has sufficient physical strength to enable it to withstand disintegration during chlorination. A piece of magnesium oxide prepared in the manner disclosed hereinafter remains, throughout the chlorinating operation, as a single piece which gradually decreases in size as its chlorination progresses. Molten magnesium chloride formed in a charge of this material is pure and, furthermore, does not become contaminated during its passage through the charge. By employing a charge of this nature, the expedients resorted to in the prior art, in order to obtain a product of pure, anhydrous magnesium chloride, are avoided. As will be apparent hereinafter, the customary preparation and use of carbonaceous briquettes from finely ground magnesia and carbon are also avoided by using the methods of our invention.

A further feature of our process resides in the manner in which the chlorinating zone of the furnace is heated uniformly throughout and maintained at a temperature above the melting point of magnesium chloride, but below the boiling point and preferably below the temperature at which any appreciable volatilization of magnesium chloride occurs.

A further feature of the invention resides in the manner in which the chlorinating process is operated to obtain a high reaction velocity and efficiency with the result that at the same time economies are effected in the operation of the process which were hitherto considered impossible to realize.

A complete understanding of the objects of the present invention and the manner in which they are attained may be had from the following description and accompanying drawing in which:

Figure 1 is a cross-sectional side elevation of an electrical resistance furnace suitable for carrying out the process herein described; and

Figure 2 is a top plan view of the furnace illustrated in Figure 1.

Like characters of reference refer to like parts throughout the specification and drawing.

In general, the present process consists of two steps:

(*a*) The preparation of the charge containing strong coherent porous pieces of sintered magnesium oxide and pieces of coke; and

(*b*) The chlorination of the charge during which the magnesium oxide is converted into substantially pure and highly fluid molten magnesium chloride.

We have found that magnesium bearing ores such as dolomite, brucite and magnesite, in the form of finely ground concentrates or of ore in lump or finely ground form, all of which forms may contain impurities such as the oxides of silicon, iron, aluminum and calcium, may be used satisfactorily as raw materials for our process.

In the case of a finely ground concentrate, containing magnesium in the form of carbonate, we prefer first to calcine the concentrate at about 700° C. to produce a lightly calcined or caustic magnesium oxide. Water or a dilute acid solution, such as, for example, would contain 3% sulphuric acid, is added to form a plastic mixture suitable for moulding into brick shapes. The use of sulphuric acid solution increases the strength of the bricks after drying and in the first stages of firing, the firing preferably being conducted in the manner described hereinafter. The use of sodium carbonate, Goulac or similar binding agents gives similar characteristics. The concentrate may, if desired, be first moulded, then calcined and fired. Also, magnesium bearing ores may be calcined, sized, moulded and fired, or they may be sized, moulded, calcined and fired at the requisite temperatures. In the latter case, the calcination and firing may take place in one operation. Alternatively, if the magnesium bearing ores are in lump form, they may be calcined and fired with the omission of the moulding step.

The temperature at which the finely ground material is given its calcination has an important bearing on the subsequent behaviour of the calcined material. The lower the temperature of calcination, the lighter, more chemically reactive and water absorbent is the resulting calcined material and we, therefore, prefer to calcine the magnesium bearing material at a comparatively low temperature, about 700° C., which, in the next stage of the firing, results in a very strong and relatively porous brick which is readily amenable to chlorination.

The bricks, after they have been moulded, are dried slowly and then fired at the requisite temperature which, as described hereinafter, causes sufficient sintering of the material to produce a strong, porous, coherent product.

The bricks are broken and sized, the pieces within the range of from ¼" to 1½" being preferred as feed for the chlorination furnace, the minus ¼" material being returned to the moulding step.

We have found that the satisfactory behaviour of the magnesium oxide in the subsequent chlorinating step depends to a great extent upon the temperature at which the bricks are fired. Pure magnesium oxide has a melting point of about 2800° C. but, if impure, it softens and partially or completely fuses at considerably lower temperatures, the actual temperatures of such physical change depending upon the composition of the material being used. Magnesium silicates form at temperatures of 1400° C. and above.

Magnesium oxide in the fused condition is difficult to chlorinate and magnesium silicate is practically inert with respect to chlorination. Both these materials, and also other silicates which may form, have the effect of coating the magnesium oxide particles and filling the pores in the brick. Therefore, at too high a temperature of firing, apart from the increased cost entailed, a product is obtained which is not as easily chlorinated and the higher the temperature of firing, the greater is the proportion of fused products. Conversely, when too low a temperature of firing is employed, a product is obtained which lacks strength due to an insufficient proportion of fused materials being present in the mass. This product tends to crumble during chlorination to form fines which contaminate the magnesium chloride and cause the operating difficulties which it is the object of this invention to avoid.

We have found that the most suitable temperature range of firing will vary with different types of magnesium oxide bearing materials. The product from such firing should preferably be not glassy or vitreous in appearance, though a partially vitrified product, such as commercial magnesite brick, may be used, but sufficient fusion of some of the constituents should have taken place to bind all the grains together into a strong, porous, coherent mass and only sufficient temperature to produce this result should be used for reasons mentioned hereinabove. We have found that the preferred firing temperature for certain magnesium bearing concentrates, which had been caustic calcined and bonded as described hereinabove and for certain magnesium bearing ores, lies within the range of from 1250° C. to 1400° C.

The size of the pieces of magnesium oxide charged into the chlorinating furnace also has an important bearing on the successful chlorination of the charge, inasmuch as the speed of the reaction between the chlorine and the magnesium oxide is largely determined by the surface area of the pieces exposed to the action of the chlorine. While this factor governs the maximum size, it is particularly desirable to limit the minimum size, since too large a proportion of fine material in the charge tends to choke the furnace and, moreover, such fine material has a tendency to be washed down with the magnesium chloride, forming therewith the thick pasty mass, which makes the continued operation of the process extremely difficult and contaminates the product.

We have found that very satisfactory operating results are obtained when the size of the magnesia brick fed to the chlorinating furnace is between ¼" and 1½". It will be understood, of course, that the scope of the invention is not to be limited to these sizes. It is pointed out that even a ¼" piece, after it has been 90% chlorinated, has a diameter of approximately 3 millimeters and no difficulties are encountered with pieces of such size.

The size of the coke, which preferably comprises about 20% of the weight of the charge, is of the same order as that of the pieces of magnesia brick feed which comprise the remainder of the charge. We prefer to use petroleum coke but have found that other types of coke react well, although they may have a higher ash content, containing in particular silica, iron oxide and alumina, which are chlorine consuming and/or residue producing impurities.

The coke, if containing appreciable amounts of hydrocarbons and water, preferably should first be calcined in order to avoid the possible formation of soot and hydrochloric acid.

We have found that very satisfactory operating results are obtained when an excess of coke is mixed with the magnesium oxide over the amount actually required to effect the reaction.

$$2MgO+C+2Cl_2=2MgCl_2+CO_2$$

In operating with a charge containing pieces of magnesium oxide and coke in the proportion of about 80 to 20 by weight, the chlorination proceeds at a very rapid rate.

The sized pieces of magnesium oxide and coke are charged into a suitable furnace, such as that described hereinafter, and the temperature of the lower or chlorinating zone of the furnace is raised by means of internal or external heating to the desired temperature above the melting point of magnesium chloride, but below the point at which appreciable volatilization of the magnesium chloride will occur. Having regard to the above limitations, we have found that the higher the temperature above the melting point in this zone, the more rapid is the rate of chlorination. We have further found that the most satisfactory results are obtained when the chlorinating zone is maintained at about 1100° C., as the use of this temperature serves to increase the rate of chlorination many times over the rates prevailing in processes operated at temperatures below or near the melting point of magnesium chloride. The temperature of 1100° C. is well above the melting point of magnesium chloride, 712° C., yet not sufficiently high to cause any appreciable volatilization of the magnesium chloride, attack upon the furnace lining or the formation of silicates or other fusible materials which, if present, would increase operating difficulties, decrease efficiencies and impair the purity of the product. The top zone of the furnace, which is much shallower than the chlorinating zone beneath it, need only be maintained at a temperature sufficiently high to prevent, therein, any appreciable condensation of volatile impurities such as the chlorides of iron and aluminum. When the furnace has been heated to within the predetermined temperature ranges, chlorine gas is admitted to the chlorinating zone and passed up through the charge at the desired rate.

As the fluid, molten magnesium chloride, uncontaminated by entrained impurities, is formed, it passes downwardly through the charge and is collected at the base of the furnace in a reservoir of coarse lumps of suitable material such as coke, graphite, silica, magnesium silicate, fused magnesium oxide, charge mixture, or spent charge. The fluid molten magnesium chloride is removed periodically from the base of the furnace in a manner similar to that used in the operation of blast furnaces for the production of various metals.

Fresh charge is added to the top zone of the furnace, as the charge below is consumed and sinks downwardly as the reaction between the magnesium oxide and chlorine proceeds. This procedure is repeated until the furnace becomes full of residue, which is then removed and the operation is repeated. Alternatively, the operation may be conducted continuously, the residue being removed periodically through a furnace clean-out door provided for that purpose.

Recoveries of magnesium as magnesium chloride of the order of 90% or more can be obtained in our process with substantially no chlorine loss

by conducting the reaction in a shaft furnace of a height sufficient such that all the chlorine is consumed and such that the residue removed from the furnace contains very little unreacted material. In the event that the operation is carried out in a manner such that the exit gases contain chlorine, this chlorine may be recovered and passed into a second furnace or returned to the first furnace.

Referring to the accompanying drawing, the numeral 1 indicates an electrically heated shaft furnace lined with heat and chlorine resistant material 2 such as silica bricks, into which is charged the sized pieces of magnesium oxide and coke 3. Molten magnesium chloride collects in the reservoir formed in the base of the furnace from which it is periodically withdrawn through tap-hole 4.

The furnace is heated by means of two vertical carbon or graphite electrodes 6, used as resistors, which are solidly connected to the horizontal carbon or graphite connection 5 of relatively large cross-sectional area. It will be apparent that the furnace may be fitted with any number of vertical resistors connected together by means of one or more horizontal carbon connections or blocks in single-phase or three-phase circuits. The cross-section, current density and spacing of these resistors are such as to establish and maintain the desired temperatures through the furnace.

Chlorine is admitted into the lower portion of the charge 3 through the nozzle 7.

As the reaction between the chlorine and magnesium oxide progresses, the residue which collects at the bottom may be removed through the clean-out door 8 located at the side of the furnace.

A removable brick cover 9 is fitted over the top and provided with openings 10 and 11 which serve respectively for barring the charge, if necessary, and for evacuation of the exit gases. The resistors are shielded and water-jacketed to prevent their corrosion where they enter the furnace.

In the event that it is desired to conduct the chlorinating process in an externally heated furnace, a retort type furnace, similar to that used in coke oven practice, is required.

In the operation of the furnace, it is necessary to ensure that the charge be heated and maintained within the highest possible temperature range having regard to the limitations set out hereinbefore in order to conduct the operation of the furnace in the most efficient manner. We have found that temperature control within this range is necessary for the reasons also set out hereinabove. In addition, local high temperature zones, such as would be developed by arcing, by points of high electrical resistance or by other causes, must be avoided to prevent high electrode consumption and the formation of undesirable materials such as secondarily formed, finely divided magnesium oxide and soot.

The composition of the residue is dependent upon the analysis of the charge fed into the furnace and the degree of chlorination of the charge. A typical residue where the process has been operated in a manner described hereinbefore, consists of unused coke, saturated with magnesium chloride, and mixed with considerable amounts of impurities such as silica and incompletely chlorinated magnesium oxide.

In the operation of the process we have found that we are primarily interested in the following consecutive reactions:

A. $MgO+Cl_2 \rightleftarrows MgCl_2+\frac{1}{2}O_2+7{,}310$ cals.

B. $\frac{1}{2}C+\frac{1}{2}O_2=\frac{1}{2}CO_2+47{,}200$ cals.

C. $\frac{1}{2}C+\frac{1}{2}CO_2 \rightleftarrows CO-20{,}770$ cals.

D. $\frac{1}{2}O_2+CO \rightleftarrows CO_2+68{,}000$ cals.

With respect to rate constant k of reaction A, Gmelin's Handbuch der Anorganischen Chemie, 8th edition, part A–B, page 111, part B magnesium 1937, states that

$$K_{550^\circ\,C.}=\frac{[Cl_2]}{\sqrt{[O_2]}}=17.4;\ \text{and}\ K_{700^\circ\,C.}=29.5$$

From these values we have determined by calculation that $K_{1000^\circ\,C.}=60.3$ and that at equilibrium at 1000° C. the theoretical gas analysis would, therefore, show 2.6% O_2 and 97.4% Cl_2. The rate of reaction of a reversible reaction being proportional to the extent to which the system is removed from the state of equilibrium, it follows that the rate of chlorination in the case of reaction A must depend upon the difference in oxygen concentrations at equilibrium and at any given state of the system. Where reactions A and B only are concerned, the rate of chlorination, being promoted by the removal of oxygen from the system and by the intimate distribution of coke in the charge, is thus governed by the diffusion rate of oxygen. Where reactions C and D are, however, also involved, the diffusion rate is of much less importance. Of these secondary reactions at 1000° C., C and D are sufficiently fast to serve to depress the oxygen concentration and, provided there is sufficient coke available, it is not essential that the coke be intimately mixed in the charge.

This theory is confirmed by chlorinating pieces of magnesium oxide brick feed alone in an externally heated silica-lined furnace at a temperature of about 1000° C., no reducing agent being present. Oxygen was found in the exit gas, while magnesium chloride, free from magnesium oxide, was collected in the well of the apparatus.

In a second experiment, a column of alternate pieces of magnesium oxide brick feed and coke, the pieces being about an inch and a half in size and touching only at their upper and lower extremities, were exposed to reaction with chlorine and the chlorinating rate was much faster due to the secondary reactions than in the first experiment, where no secondary reactions were possible.

In a third experiment, an intimate mixture of pieces of coke and of similarly sized pieces of magnesium oxide brick feed was chlorinated, with the result that the secondary reactions were favoured and the chlorinating rate was increased considerably. In this experiment, in which the coke was distributed throughout the charge, conditions were similar to those prevailing in our normal furnace operation.

It will be apparent from the above discussion that, for any given temperature, the rate of chlorination would be favoured to some extent by the intimacy of the magnesium oxide and carbon mixture in the charge. Briquetted mixtures of finely ground magnesium oxide and carbon might thus be more reactive than a mixture of coarser pieces of magnesium oxide and carbon, if the latter mixture had not sufficient porosity. However, temperatures below the melting point of magnesium chloride have been recommended for the chlorination of these briquetted materials, since these disintegrate above the melting point of magnesium chloride, and this

disintegration seriously retards the rate of chlorination, impairs the purity of any product formed, and causes difficulties which finally make continued operation impossible. The lower temperatures greatly decrease the rate of chlorination. In operating with the type of charge described hereinbefore and at a relatively high temperature, the chlorination is carried out considerably above the melting point of magnesium chloride, such as for example at about 1100° C., thereby gaining the advantage of the greatly increased rate of chlorination without any of the above disadvantages associated with the use of briquettes at such high temperatures.

Carbon monoxide, as is shown in the prior art, may be used as a reducing agent in place of coke, but we have found that the chlorinating rate is, however, only about three-quarters that when coke is used, due to dilution of the chlorine with carbon monoxide, both the driving force

$$\frac{d[MgCl_2]}{dt} = k[Cl_2]$$

and the time of contact being reduced. The carbon monoxide mentioned here is not to be confused with the carbon monoxide of reaction C above, which is only an intermediate reaction product. In this case also, when using a magnesium oxide charge, as prepared above, we have found that a pure magnesium chloride product is obtained directly from the charge.

What we claim as new and desire to protect by Letters Patent of the United States is:

1. The method of producing anhydrous magnesium chloride which comprises reacting magnesium oxide, in the form of strong, porous, coherent pieces formed by heating magnesium concentrates to incipient fusion of the impurities therein, with chlorine gas in the presence of coke while maintaining a reaction temperature above the melting temperature of magnesium chloride.

2. The method of producing anhydrous magnesium chloride which comprises reacting magnesium oxide, in the form of strong, porous, coherent pieces formed by heating to incipient fusion magnesium oxide bearing substances which fuse substantially above the melting temperature of magnesium chloride, with chlorine gas in the presence of coke while maintaining a reaction temperature above the melting temperature of magnesium chloride.

3. A process for the production of anyhydrous magnesium chloride which comprises charging pieces of impure magnesium oxide into a chlorinating furnace, said pieces of magnesium oxide having been formed by heating to a temperature substantially above the melting temperature of magnesium chloride whereby incipient fusion of the impurities occurs to form pieces which are strong, porous and coherent at temperatures above the melting temperature of magnesium chloride, reacting the magnesium oxide with chlorine gas in the presence of coke at a temperature above the melting temperature of magnesium chloride and separately withdrawing substantially pure magnesium chloride from the chlorinating furnace.

4. A process for the production of anhydrous magnesium chloride which comprises charging into a chlorinating furnace pieces of sintered magnesium oxide and pieces of coke, said pieces of sintered magnesium oxide having been prepared from magnesium oxide sintered at a temperature within the range of from 1250° C. to 1400° C.; maintaining in the chlorinating zone of the furnace a uniform temperature above the melting point of magnesium chloride and in the upper zone a temperature sufficiently high to prevent condensation of volatile metallic chloride impurities from the exit gases; passing chlorine gas through the charge and separately withdrawing substantially pure magnesium chloride.

5. A process for the production of anhydrous magnesium chloride which comprises charging pieces of impure magnesium oxide, sized within a particle size within the range of from ¼ inch to 1½ inches, into a chlorinating furnace, said pieces of magnesium oxide having been formed by heating to a temperature substantially above the melting temperature of magnesium chloride whereby incipient fusion of the impurities occurs to form pieces which are strong, porous and coherent at temperatures above the melting temperature of magnesium chloride, reacting the magnesium oxide with chlorine gas in the presence of coke at a temperature above the melting temperature of magnesium chloride, and separately withdrawing substantially pure magnesium chloride from the chlorinating furnace.

6. A process for the production of anhydrous magnesium chloride which comprises heating to incipient fusion magnesium oxide bearing substances which fuse at a temperature substantially above the melting temperature of magnesium chloride, thereby forming pieces of impure magnesium oxide which are strong, porous and coherent at temperatures above the melting temperature of magnesium chloride, charging the pieces of impure magnesium oxide into a chlorinating furnace and reacting the magnesium oxide with chlorine gas in the presence of coke while maintaining a reaction temperature above the melting point of magnesium chloride and separately withdrawing substantially pure magnesium chloride from the chlorinating furnace.

7. A process for the production of anhydrous magnesium chloride which comprises sintering magnesium oxide at a temperature within the range of from 1250° C. to 1400° C.; breaking the sintered magnesium oxide into pieces of from ¼'' to 1½'' size; charging into a chlorinating furnace the sized pieces of sintered magnesium oxide and similarly sized pieces of coke, maintaining in the chlorinating zone of the furnace a uniform temperature of about 1100° C., and in the upper zone a temperature sufficiently high to prevent condensation of volatile metallic chloride impurities from the exit gases; passing chlorine gas through the charge and separately withdrawing substantially pure magnesium chloride.

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