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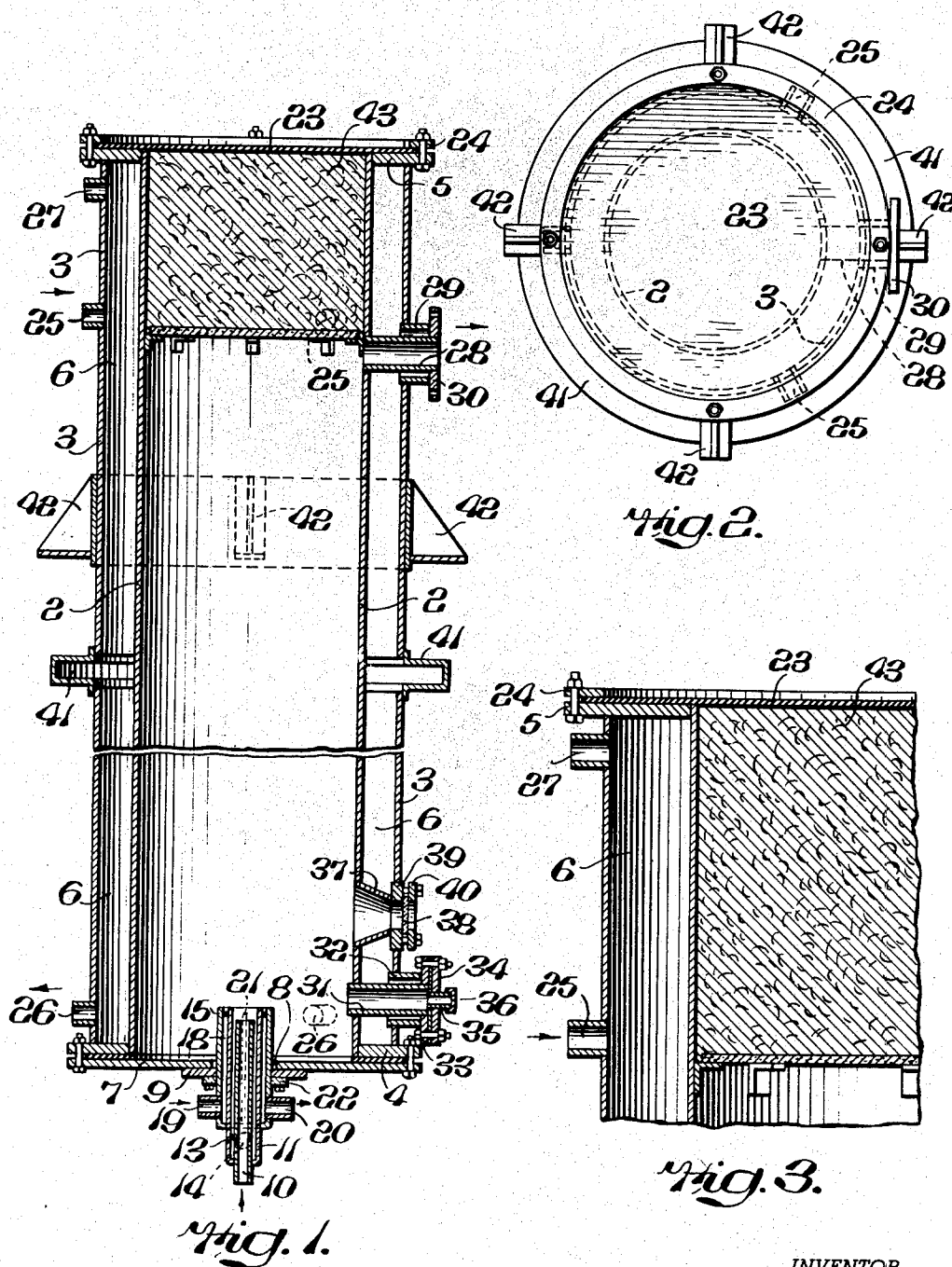
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METHOD FOR SYNTHESIZING HYDROGEN CHLORIDES

Filed Nov. 10, 1941

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

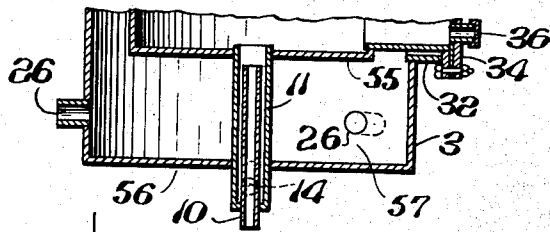


Fig. 7.

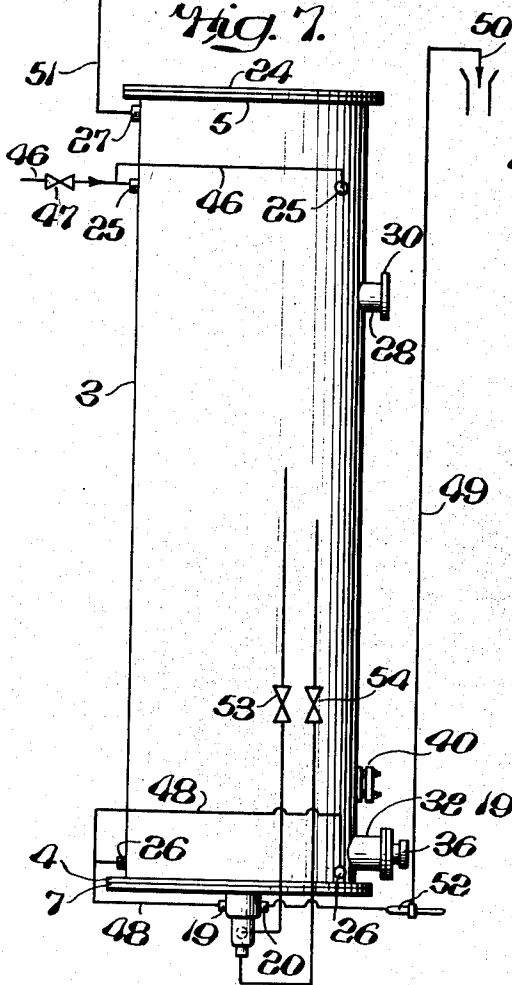


Fig. 6

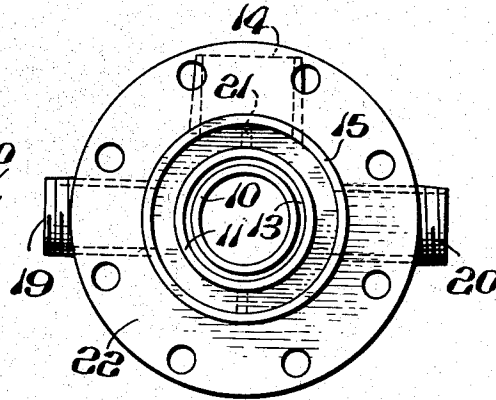


Fig. 5.

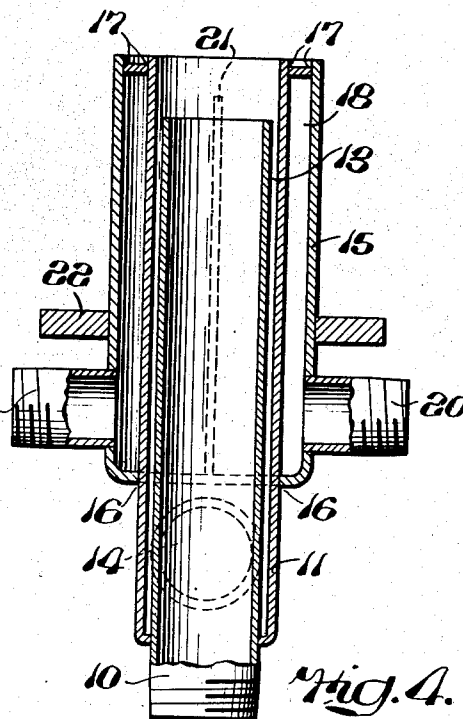


Fig. 4.

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METHOD FOR SYNTHESIZING HYDROGEN CHLORIDE

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Application November 10, 1941, Serial No. 418,586

5 Claims. (Cl. 23—156)

My invention relates to an improved method for burning hydrogen and chlorine together in a metal burner and combustion chamber, to produce hydrogen chloride, and cooling the product while protecting the metal in contact with the hot chlorine, which is very corrosive to metals even when anhydrous, and against the hydrogen chloride, which when moist is very corrosive to metals even at moderate temperatures.

Heretofore it has been customary to burn hydrogen and chlorine together in burners of refractory material such as silicon dioxide or "Vitreosil," and cool the products in contact with refractory ceramic surfaces. The use of such apparatus involves not only considerable expense for renewals, but also frequent interruption of production, because of its fragile nature. Moreover, such materials are not well adapted to formation of air tight joints in and around the burner and burner connections. If such burners were to be operated at pressures above atmospheric there would be a leakage of HCl gas which would be highly objectionable. It is practically necessary, therefore, to operate under a slight negative pressure. This results in infiltration of air which causes a part of the hydrogen to burn to H₂O, reducing the yield of HCl. In the course of cooling, this moisture may condense while the gas is still quite hot. This necessitates dealing with hot moist hydrogen chloride, which is chemically very active. The product therefore becomes contaminated with iron if any be present in the ceramic material used in the apparatus, as is usually the case. Furthermore, since refractory materials are in general very poor conductors of heat the apparatus which must be installed for cooling the HCl gas, when constructed of such material, is necessarily quite bulky and occupies much space.

I am aware that it has been proposed to burn hydrogen and chlorine together in a metal burner, by supplying the gases to the burner under such pressure that the resulting velocity is greater than the velocity of flame propagation, so that the flame is prevented from actual contact with the burner tip. This practice is objectionable for the following reasons: In the first place, if the hydrogen and chlorine are of electrolytic origin, as is very likely to be the case, it becomes necessary to compress both these gases to a substantial pressure, which is difficult and expensive. In the second place, the velocity necessary to maintain the flame without contact with the burner tip, is quite critical and even with good control the flame is liable to become extinguished.

This, of course, causes the combustion chamber to become filled with a highly explosive mixture within a few seconds.

My invention accordingly has for its object to provide an air tight water-cooled burner and combustion chamber, constructed of ordinary metals, not resistant to moist hydrogen chloride, in which hydrogen and chlorine may be burned together at ordinary flame velocities without destroying the burner, by regulating the temperature of the metal surfaces in contact with the gases so that any moisture that may be present, due to moisture in the chlorine or hydrogen or combustion of hydrogen with traces of oxygen in these gases, will not be condensed. The remaining heat of reaction can then be extracted in relatively small coolers constructed of special metals, such as tantalum, which is resistant to moist HCl.

In practice, some slight oxidation of the metal may give rise to formation of its chloride. If a liquid film saturated with metal chloride were allowed to form on the metal surface, it would have a vapor pressure lower than that of the uncontaminated condensate. This film would therefore dry with difficulty. To avoid this, it is necessary to operate at a temperature a few degrees higher than the dew point of the gases. If the metal forms a deliquescent chloride this differential must be greater than otherwise. Thus, if the metal be iron, which forms a deliquescent chloride, the surface must be kept at least 15° C., above the dew point of the gases, whereas if the metal be copper, which forms a non-deliquescent chloride, a differential of 3° C. is sufficient.

I am aware that it has been proposed to cool humid SO₂ and SO₃, which are corrosive to metals when wet, by contact with metal surfaces maintained at temperatures slightly above the dew point of the gases. However, in this case the cooling is by means of water circulating in a long cooling coil of relatively small diameter, by convection only, therefore at relatively low velocity. Hence there is no safeguard against local overcooling at the entrance to the coil, nor to the outer wall of the chamber when starting the process, nor against local overheating of the outer wall. Moreover, the water is allowed to boil, hence to create zones where the steam may be superheated. When one of the gases is chlorine, this is not permissible, as chlorine is corrosive to metals at the temperature of water boiling at ordinary pressure.

In copending application Serial No. 321,600, filed Feb. 29, 1941, there is disclosed a method

and apparatus for accomplishing my purpose, in which the burner is directed longitudinally with respect to the combustion chamber and the water is introduced into the cooling system at an initial temperature maintained above the dew point of the gas, so that local overcooling is impossible. The water is then mechanically circulated at such a velocity that it cannot become heated to its boiling point.

I have now discovered that if the combustion chamber be cooled by water in an ample jacket, formed without pockets or recesses, so that convection currents may circulate freely in every part of the jacket, and the cooling water be introduced at or near the top of the jacket, the water may be introduced at a temperature below the dew point of the gas. The rate of admission of cooling water is then regulated to maintain the desired temperature in the jacket, which, because of the convection currents, is everywhere substantially the same. The temperature maintained in the jacket is of course safely above the temperature at which moisture would be condensed from the gases in presence of the chloride of the metal of which the burner and combustion chamber are constructed. The burner may be let in preferably through the bottom of the combustion chamber, which may be jacketed, or the burner may be provided with its own jacket. In any case, water is drawn from the main jacket around the walls or through the jacket of the burner in amount equivalent to the fresh cooling water introduced and either allowed to go to waste or cooled and recycled. A thermostat or thermometer may be inserted at any convenient point in the cooling system, such as in the jacket or stream of water leaving it, and used to regulate the temperature of the water in the jacket by control of the rate of admission of cooling water, either manually or automatically.

Referring to the drawings:

Fig. 1 is a vertical elevation of my apparatus, in section through the axis of the combustion chamber and burner.

Fig. 2 is a plan view of the combustion chamber.

Fig. 3 is an elevational view, in section, of a portion of the combustion chamber, showing the frangible diaphragm of Fig. 1, to an enlarged scale.

Fig. 4 is a sectional elevation of the burner, to an enlarged scale.

Fig. 5 is a plan view of the burner.

Fig. 6 is a diagrammatic elevational view of the apparatus to a reduced scale, showing the pipe connections to and between its various parts.

Fig. 7 is a sectional elevation through the lower portion of a combustion chamber and burner of modified construction.

Referring to the figures:

1 is the combustion chamber proper, formed by metal shell 2, preferably cylindrical, and closed by end plates to be described, as illustrated. Outside shell 2, and preferably concentric with it, is shell 3. Shell 2 is provided with flanges 4 and 5, which extend beyond shell 3, closing the annular space between shells 2 and 3 and forming with these inner and outer shells a water jacket 6.

Combustion chamber 1 is closed at its bottom by plate 7, which, in the construction illustrated, is provided with a central opening 8, preferably circular, through which the burner is let into the combustion chamber. The edges of opening 8 are reinforced by annular plate 9.

The burner consists of inner tube 10 for ad-

mission of chlorine and outer tube 11, concentric with tube 10, and closed in at its lower end and welded thereto, forming therewith an annular passage 13, for hydrogen admitted through pipe 14. Outside tube 11, concentric tube 15, closed in and welded to tube 11 at points 16 and 17, forms with tube 11 a burner jacket 18. Water is admitted to burner jacket 18 through inlet 19 and led away therefrom through outlet 20. Burner jacket 18 is provided with baffle 21 to force the water to pass from the inlet upwards and around the tip of the burner before it can find its way to outlet 20. Tube 15 is provided with flange 22, by means of which it is bolted to annular plate 9. Combustion chamber 1 is closed at its top by frangible disc 23, seated upon flange 5 and secured thereagainst by annular plate 24, and serving as a gasket between flange 5 and plate 24. Frangible disc 23 is described more fully and claimed in co-pending application Serial No. 418,585, filed November 10, 1941.

Water is admitted to jacket 6 through pipe couplings 25, of which there are preferably at least three, equally spaced around the circumference of shell 3 near its top. Water is conducted from jacket 6 through pipe couplings 26, which are similarly spaced around shell 3 near its bottom. Jacket 6 is provided with an air vent through pipe coupling 27, located preferably at its extreme top.

Combustion chamber 1 is provided with an exit for hydrogen chloride, comprising pipe 28 and flange 30. Pipe 28 extends through jacket 6 and is itself jacketed by pipe 29. Combustion chamber 1 is also provided with an opening near its bottom, similar in construction to the hydrogen chloride exit, for ignition of the burner, comprising pipes 31 and 32, flange 33 and cover 34, through which extends nipple 35. Nipple 35 is normally closed by cap 36 as shown. When it is desired to ignite the burner, cap 36 is removed and an oxy-hydrogen torch inserted through nipple 35. A sight glass window is also provided for observation of the burner flame, comprising conical wall 37, extending across jacket 6 to outer shell 3, and glass plate 38, which is clamped between flanges 39 and 40, with suitable gaskets between.

Outer shell 3 of the combustion chamber jacket 6 is provided with expansion joint 41, to allow for difference in expansion between the inner and outer walls of jacket 6.

The apparatus is supported upon lugs 42, having the bottom suspended above the floor and giving ready access to the burner.

Referring particularly to Fig. 6, it will be seen that the water is supplied to jacket 6 through pipe couplings 25 from pipe 46, under control of valve 47. Water is conducted away from jacket 6 through pipe couplings 26 by pipe 48, which is connected to inlet 19 of burner jacket 18. Exit 20 from burner jacket 18 is connected to pipe 49, from which the water may be allowed to discharge to waste at point 50, above top level of the combustion chamber; or if preferred the water may be cooled and recycled to pipe 46 in obvious manner. Air vent pipe 51 is connected to pipe coupling 27 and likewise carried above the top level of the combustion chamber, so that jacket 6 stands full of water under a slight head.

At any convenient point in pipe 49, such as the elbow, is inserted a thermometer well 52, by means of which a thermometer may be introduced for the purpose of observing the temperature of the water leaving jacket 6 through

burner jacket 18, and regulating the same by means of valve 47. If preferred, the thermometer may be replaced by a thermostat automatically controlling valve 47.

Admission of hydrogen and chlorine is controlled by calibrated orifices (not shown) but may be shut off by valves 53 and 54 respectively.

Referring to Fig. 7: In this figure there is illustrated a modification in which the bottom of the combustion chamber is closed by a bottom plate 55, welded thereto, and the bottom of the water jacket is likewise closed by a bottom plate 56, welded thereto, and forming a jacket 57, with respect to the bottom of the combustion chamber. With this construction, the burner jacket may be dispensed with. The burner is then let in through and welded to both bottom plates, the water jacket 57 serving to cool the burner.

It will be observed that in the figures the ratio of diameter of shells 3 and 2 is approximately as 7 to 5. This is an important point, since a jacket space too narrow to permit free circulation of the cooling water would cause temperature differences to develop between different parts of the jacket and might result in local overcooling near the points of inlet of the cooling water adjacent pipe coupling 25.

With apparatus constructed as just described, having the burner located at the bottom of a jacketed combustion chamber cold water admitted at the top of the jacket and hot water drawn off from the bottom of the jacket, as through a jacket enclosing the burner proper, it is possible to make use of cooling water from a source that may vary quite widely in temperature and may be below the dew point of the gases.

The following table will serve to show the dew point of gases of different moisture content:

Moisture per cent by volume	Dew point centigrade
0.5.....	Degrees 28
1.0.....	37
2.0.....	47
3.0.....	53
4.0.....	57
5.0.....	60
6.0.....	63
10.0.....	71

The moisture content of the hydrogen chloride of course depends upon the moisture and oxygen content of the hydrogen and chlorine, which in turn depends upon the care with which these gases have been dried, and the precautions that have been taken to exclude air, such as avoidance of pressures below atmospheric that could cause infiltration at the joints. In practice I find that the moisture content may be 0.5 to 10 percent, or more typically 1 to 3 percent. As stated above, if the apparatus is of copper the cooling water should be kept at least 3° C. above these temperatures, while if it is of iron the cooling water should be not less than 15° C., and preferably 20° C., above these temperatures. In practice, in an apparatus of iron of the character described, with cooling water from a source at 0° to 20° C., when producing hydrogen chloride having a dew point of 40° C., a water temperature of 60° C. at the exit from the water jacket is found to give very satisfactory results.

However, I do not wish to be limited to the exact range of temperatures given herein. Neither do I wish to be limited to the exact details of construction illustrated and described, as many modifications within the scope of the in-

vention will suggest themselves to persons skilled in the art.

I claim as my invention:

1. The method of synthesizing hydrogen chloride from chlorine and hydrogen contaminated with at least one member of the group consisting of moisture and oxygen, resulting in a product having a dew point above 28° C., which comprises bringing the gases together in a metal burner upwardly directed in an upright jacketed metal combustion chamber, causing them to combine therein, and protecting the surfaces of the combustion chamber against the resulting hot moist gaseous mixture by continuously introducing cooling liquid at the top of the jacket and withdrawing it from the bottom thereof, the rate of introduction of the cooling liquid and the total volume of liquid in the jacket being so co-related with the volume of the combustion chamber and heat generated therein as to cause prompt and effective mixing of incoming cooling liquid with the warm liquid in the jacket by unobstructed convection currents within the jacket and to maintain the body of cooling liquid in the jacket at a relatively uniform temperature below its boiling point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

2. The method of synthesizing hydrogen chloride from chlorine and hydrogen contaminated with at least one member of the group consisting of moisture and oxygen, resulting in a product having a dew point above 28° C., which comprises bringing the gases together in an upwardly directed metal burner in an upright jacketed metal combustion chamber, causing them to combine therein, and protecting the surfaces of the combustion chamber against the resulting hot moist gaseous mixture by continuously introducing cooling liquid at the top of the jacket, withdrawing it from the bottom thereof, conducting it to a level above that at which it enters the jacket and there discharging it through an unobstructed exit; the rate of introduction of the cooling liquid and the total volume of liquid in the jacket being so co-related with the volume of the combustion chamber and heat generated therein as to cause prompt and effective mixing of incoming cooling liquid with warm liquid in the jacket by unobstructed convection currents within the jacket and to maintain the body of cooling liquid in the jacket at a relatively uniform temperature below its boiling point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

3. The method of synthesizing hydrogen chloride from chlorine and hydrogen contaminated with at least one member of the group consisting of moisture and oxygen resulting in a product having a dew point above 28° C., which comprises bringing the gases together in an upwardly directed metal burner in an upright jacketed metal combustion chamber, causing them to combine therein, and protecting the surfaces of the combustion chamber against the resulting hot moist gaseous mixture by continuously introducing cooling liquid at the top of the jacket and withdrawing it from the bottom thereof, the outer dimensions of the jacket being not less than 7/8ths those of the combustion chamber and the rate of introduction of cooling liquid being so regulated as to cause prompt and effective mixing of

incoming cooling liquid with warm liquid in the jacket by unobstructed convection currents within the jacket and to maintain the body of cooling liquid in the jacket at a relatively uniform temperature below its boiling point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

4. The method of synthesizing hydrogen chloride from chlorine and hydrogen contaminated with at least one member of the group consisting of moisture and oxygen, resulting in a product having a dew point above 28° C., which comprises bringing the gases together in an upwardly directed metal burner in an upright jacketed metal combustion chamber and causing them to combine therein, protecting the surface of the combustion chamber against the resulting hot moist gaseous mixture by continuously introducing cooling liquid at the top of the jacket and withdrawing it from the bottom thereof and venting off from the top of said jacket any fixed gases entrained therewith, the rate of introduction of cooling liquid and the total volume of liquid in the jacket being so co-related with the volume of the combustion chamber and heat generated therein as to cause prompt and effective mixing of incoming cooling liquid with warm liquid in the jacket by unobstructed convection currents within the jacket and to maintain the body of cooling liquid in the jacket at a relatively uniform temperature below its boiling point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

ing point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

5. The method of synthesizing hydrogen chloride from chlorine and hydrogen contaminated with at least one member of the group consisting of moisture and oxygen resulting in a product having a dew point of 28° to 71° C., which comprises bringing the gases together in an upwardly directed metal burner in an upright jacketed metal combustion chamber, causing them to combine therein, and protecting the surfaces of the combustion chamber against the resulting hot moist gaseous mixture by continuously introducing cooling liquid at the top of the jacket and withdrawing it from the bottom thereof, the rate of introduction of cooling liquid and the total volume of liquid in the jacket being so co-related with the volume of the combustion chamber and heat generated therein as to cause prompt and effective mixing of incoming cooling liquid with warm liquid in the jacket by unobstructed convection currents within the jacket and to maintain the body of cooling liquid in the jacket at a relatively uniform temperature below its boiling point but above the dew point of the gases, thereby preventing the cooling of portions of the wall of the combustion chamber to a temperature at which the gases corrode the metal.

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