

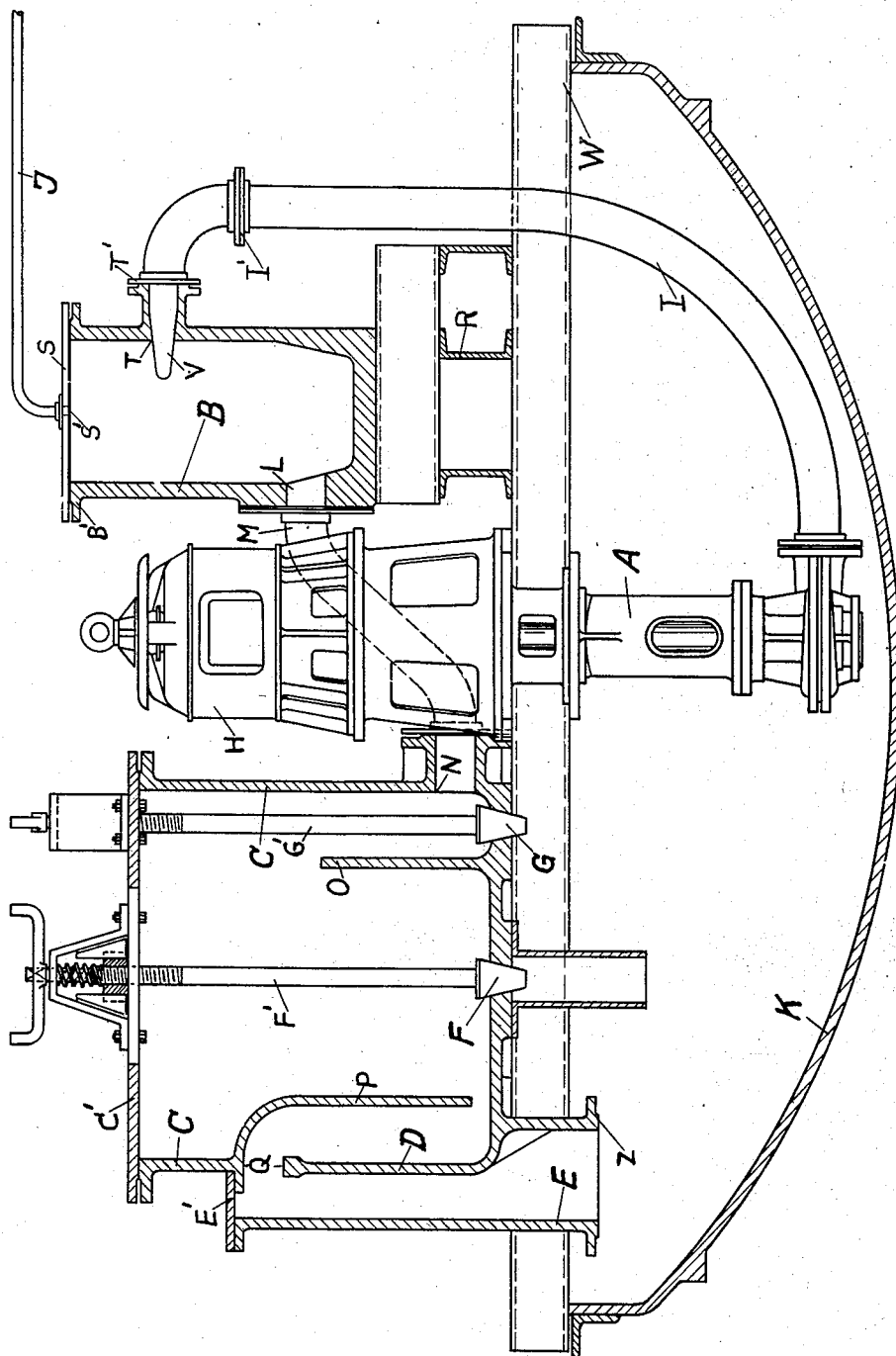
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METHOD OF REFINING MOLTEN ZINCIFEROUS LEAD

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METHOD OF REFINING MOLTEN
ZINCIFEROUS LEAD

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The present invention relates to a process of refining lead and more particularly to an improved method of dezincing molten lead with chlorine gas and to an improved apparatus therefor.

It is well known that heretofore various methods have been proposed for treating zinciferous lead in a molten state with gaseous chlorine to remove its zinc content as zinc chloride. In general, dezincing was carried out by bubbling chlorine through a bath of molten lead or by passing the lead through an atmosphere of chlorine and by collecting the zinc chloride formed by reaction from the surface of the refined lead. Thus, for instance, a stream of lead was circulated from an open kettle containing the impure lead to a separate reaction chamber into which chlorine gas was also being fed. The lead and the molten zinc chloride formed by reaction were then returned directly to the open kettle through a pipe at the bottom of the reaction chamber which extended to a point below the surface of the lead bath. Due to its lower specific gravity the zinc chloride slowly rose to the surface of the lead bath and formed a solid crust which was removed by skimming. The aforesaid method, however, presented the disadvantage that a considerable amount of manual labor and loss of time were required to remove the zinc chloride dross, and that it was almost impossible to skim the surface of the refined molten lead clean of zinc chloride. The final slag formed was especially hard to remove because it was thinner and remained liquid much longer than the previously formed slag, and as it was too thin for ladeling, considerable time was lost in waiting for the lead bath to cool down to a sufficiently low temperature at which the slag froze so that it could be collected by skimming. It was also found in connection with the aforesaid method that the hot zinc chloride floating on top of the molten lead reacted with the air to which it was exposed to form zinc oxychloride which, even when present in small amount, was very detrimental in the production of satisfactory zinc chloride product for the market. Another objectionable feature was due to the loss of noxious chlorine fumes which occurred through volatilization of the hot zinc chloride and that the loss of chlorine could only be reduced by reducing the speed of the dezincing reaction. Thus, in order to prevent the escape of chlorine gas through the discharge pipe at the bottom of the reaction chamber, it was necessary to maintain the hydrostatic level of the lead above the pipe

by operating the reaction chamber under a partial vacuum, which was caused by supplying the chlorine gas at a rate slower than the rate of the reaction between the chlorine and the zinc. Besides reducing the dezincing speed of the lead, the vacuum also necessitated a perfect air-tight reaction chamber to prevent the air from seeping into the chamber with the subsequent formation of zinc oxychloride. It was also found necessary to keep the molten pool of lead at a temperature sufficiently low to allow the zinc chloride to freeze in order to form a solid crust or seal over the open surface of the molten lead, meant to prevent losses of chlorine in the form of volatile zinc chloride, which in an open kettle and with stirring was noticeable at about 750° F. However, the reaction between the chlorine and the zinc was exothermic and since the lead and the products of reaction were returned directly from the reaction chamber to the lead bath, the temperature of the lead bath could be controlled only by retarding the rate of the dezincing reaction.

Although many attempts have been made to remedy the foregoing shortcomings, the proposals, as far as I am aware, only provided procedures wherein the rate of dezincing was reduced and none provided a solution which was wholly satisfactory and which was very efficient.

I have discovered a procedure which eliminates the aforesaid shortcomings and which is very efficient and which provides improved and unexpected results.

It is an object of the present invention to provide an improved method of dezincing lead with chlorine gas which involves the removal of zinc from zinciferous molten lead, the continuous separation of the resulting zinc chloride from the treated lead and the returning of the treated lead to the main lead bath in a practically automatic and continuous manner which is substantially free from manual labor.

It is a further object of the invention to provide a process by means of which the refined lead is obtained in a separate body substantially free from zinc chloride.

Another object is to provide a process which is more efficient and by means of which a greater dezincing speed can be obtained without losses of chlorine gas or volatile zinc chloride.

It is also within the contemplation of the invention to provide a simple and improved apparatus for carrying the present invention into practice which has a separating device associated

with the reaction chamber to effect the separation of the zinc chloride from the treated lead.

Other objects and advantages of the invention will be apparent from the following description taken in conjunction with the accompanying drawing which illustrates in a diagrammatic manner a preferred embodiment of an apparatus for carrying the present process into practice.

In the drawing, the reference character K designates a vessel for holding a pool of molten zinciferous lead to be refined, which may be heated by any conventional method, not shown in the drawing. Extending across the vessel K and resting upon the rim thereof, are two or more steel beams W which support a unit comprising a motor H and a pump A for circulating a stream of lead to be treated. Pump A is suspended from the motor H and is preferably located near the bottom of the vessel K.

A reaction chamber B which may be supported over vessel K by channels R rests upon beams W. A flanged end B' is provided on the reaction chamber and has secured thereto a head S for sealing the end thereof. Associated with reaction chamber B is an opening S' in head S to provide an inlet for the chlorine gas, an inlet T near the top for introducing the impure lead, and an outlet L near the bottom for the withdrawal of the treated lead and the products of reaction. If desired, opening T may conveniently be placed at the top of the chamber, or inlet S' may be at the side near the top, and outlet L may be located at the bottom of chamber B. A connecting pipe I connects the discharge end of pump A to a nozzle V projecting into reaction chamber B through opening T. A pipe line J connects inlet opening S' to a source of supply of chlorine gas, not shown in the drawing.

A separating chamber C for separating the treated lead from the zinc chloride may be supported over vessel K by beams W at a lower level than reaction chamber B. The top of the separating chamber C may be flanged and a head C' may be secured to it for the purpose of sealing the top thereof. An inlet opening N is provided near the bottom of chamber C which may be connected by connecting line M to discharge opening L of reaction chamber B. Directly in front and at a convenient distance from inlet opening N is a baffle O upwardly extending from the inside bottom of chamber C for the purpose of deflecting the incoming stream of molten fluids upwardly. Inlet opening N may, however, be placed at a higher level in which case the baffle O may be dispensed with.

In connection with separating chamber C is also a siphon-like discharge system which may comprise an outlet opening Q at the side D of the chamber, an internal baffle P for the purpose of preventing the outflowing of the zinc chloride and an external baffle E to prevent splashing and to direct the flow of the refined lead from outlet Q directly to vessel K. Baffle P, which may be cast as an integral part of chamber C extending inwardly from the top of opening Q to a point near the bottom of the chamber, forms with side D an internal closed duct which is open at the bottom and leads to opening Q. Outside baffle E forms with wall D an external closed duct, which may be sealed at the upper end by plate E' and which extends downwardly from opening Q by which it is connected to the aforementioned internal duct to a wider outlet Z. The lower end of outlet Z may be made to extend below the surface of molten lead in vessel K and, if desired,

the herein described outlet system of chamber C may be made to work as a siphon.

For the purpose of draining the zinc chloride from the separating chamber C, valves F and G may be provided at the bottom thereof which may be operated, for instance, by valve stems F' and G' extending through the cover C'.

Separating chamber C may remain full of molten fluids up to the level of outlet opening Q, however it has sufficient capacity to hold the zinc chloride formed in treating all the lead in vessel K. Besides controlling the level in chamber C, outlet Q also may control the level of the molten fluids in reaction chamber B which is so placed in relation to chamber C as to maintain the level above discharge opening L for the purpose of preventing escape of chlorine gas thereof. The level of the molten fluids in chamber B may also be controlled by the height of baffle O, if it is found desirable to place outlet opening Q at a lower level.

In carrying the present invention into practice, the pump A is first started and a stream of the lead to be refined is thereby made to circulate for a few minutes through the apparatus in order to warm up reaction chamber B and to establish a level of molten lead up to outlet Q of chamber C, thereby sealing discharge opening L of chamber B, for the purpose of preventing escape of chlorine gas thereof. The rate of circulation of the lead is then adjusted to any desired value and the chlorine is turned on at a predetermined rate of flow which depends on the amount of lead circulated and on the amount of zinc in the lead.

The treated lead and the zinc chloride resulting from the reaction between the chlorine gas and the zinc in the lead flow out of reaction chamber B through outlet opening L and are led by connecting pipe M into separating chamber C through inlet opening N thereof. The separation of the lighter zinc chloride from the heavier treated lead in separating chamber C is effected by gravity. In order to facilitate their separation the incoming stream of mixed fluids is upwardly deflected by baffle O and led to, or near the surface of the molten bath in chamber C, thereby the heavier treated lead sinks to a lower level and the lighter zinc chloride is left floating at the top. Two layers are thus formed in separatory chamber C, the upper layer consisting substantially of zinc chloride, and the lower layer of treated lead which is increasingly free from zinc chloride at the lower levels thereof. Separating chamber C can remain full up to the level of outlet opening Q, but it is sufficiently large to hold all the zinc chloride formed in dezincing all the lead in kettle K. Flange P, moreover, is made to extend into the lower layer of the treated lead, thereby substantially entrapping the upper layer of zinc chloride at all times during the refining operation and allowing the discharging of treated lead only substantially free from zinc chloride.

At the end of the refining process, the flow of chlorine and of the lead are stopped and valves F and G at the bottom of separating chamber C are opened to drain out the refined lead remaining in the chamber. The zinc chloride is then drained through the same valve openings into separate containers.

The rate of flow of the molten lead and of chlorine gas into reaction chamber B may be adjusted for a higher speed of dezincing reaction than it has heretofore been possible, because escape of chlorine from chamber B is substantially prevented by controlling the level of the molten fluids

above outlet opening thereof, as described more fully hereinabove. Moreover, the rate of the reaction between the chlorine gas and the zinc in the lead need not be retarded so as to keep the temperature of the lead bath below the temperature at which zinc chloride begins to volatilize, or below about 750° F., because during the process the hot zinc chloride is kept in a closed chamber, which practically eliminates losses through volatilization and through oxidation. Separating chamber C tends to somewhat reduce the temperature of the treated lead which is returned to kettle K, thus preventing excessive rising in temperature of the main body of molten lead in kettle K, such as was experienced in prior processes and required special cooling means to cool the bottom and sides of the kettle in order to avoid excessive temperatures.

It is to be observed from the above description that the present invention provides an efficient and improved process for dezincing lead by means of chlorine in which the zinc chloride and the refined lead are obtained in two separate bodies, the zinc chloride substantially in a marketable form and the refined lead substantially free of zinc chloride.

It is also to be seen that a considerable amount of delay and manual labor are avoided, that losses of chlorine through volatilization of zinc chloride are practically eliminated, and that the speed of the dezincing process can be carried at a faster rate than it has heretofore been possible.

It is to be understood that the hereinabove description of an apparatus has been given for illustrative purpose only and that many variations and modifications may be resorted to without departing from the spirit of the invention. Thus, for example, separating chamber C may be substituted by an open chamber, or a centrifugal machine, or the like, by which the heavier lead may be centrifugally separated from the lighter zinc chloride.

Although in describing the present invention, the removal of zinc from molten lead has been given as a specific example, it is to be observed that other metallic impurities, whose heat of reaction with chlorine are higher than that of lead, can also be removed by the same process as any one skilled in the art will readily understand.

I claim:—

1. The improved method of refining molten zinciferous lead with chlorine gas which comprises treating a portion of zinc-bearing molten lead withdrawn from a pool of molten zinciferous lead in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, withdrawing the treated lead containing zinc chloride to a separatory chamber for effecting separation of the zinc chloride from the treated lead, and returning the treated lead substantially free from zinc chloride to the pool of molten lead.

2. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, effecting the separation of the zinc chloride from the treated lead outside of the main pool of molten lead, and returning the treated lead substantially free from zinc chloride to said pool of molten lead.

3. The improved method of refining molten zinciferous lead with chlorine gas which com-

prises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, effecting the separation of the zinc chloride from the treated lead outside of the reaction chamber and outside of the main pool of molten lead, and returning the treated lead substantially free from zinc chloride to said pool of molten lead.

4. The improved method of refining molten zinciferous lead with chlorine gas which comprises, establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, transferring the zinc chloride and the treated lead to a separatory chamber for effecting separation of said zinc chloride from said treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead.

5. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, transferring the zinc chloride and the treated lead to a separatory chamber for effecting separation by gravity of said zinc chloride from said treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead.

6. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, transferring the zinc chloride and the treated lead to a separatory chamber, directing the flow of said zinc chloride and said treated lead to a point higher than the bottom of said separating chamber to promote separation by gravity thereof and to form an upper layer of substantially only zinc chloride and a lower layer of substantially only treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead.

7. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, conveying the treated lead containing zinc chloride from the reaction chamber to the bottom of the separatory chamber, upwardly deflecting the flow of said treated lead containing zinc chloride to promote separation by gravity thereof and to form an upper layer of substantially only zinc chloride and a lower layer of substantially only treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead.

8. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, conveying the treated lead containing zinc chloride from the reaction chamber to the bottom of the separatory chamber, upwardly deflecting the flow of said treated lead containing zinc chloride to promote separation by gravity thereof and to

form an upper layer of substantially only zinc chloride and a lower layer of substantially only treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead in substantially a continuous manner.

9. The improved method of refining molten zinciferous lead with chlorine gas which comprises establishing a pool of molten lead containing zinc, treating portions of the lead with chlorine gas in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, conveying the treated lead containing zinc chloride from the reaction chamber to the bottom of the separatory chamber, upwardly deflecting the flow of said treated lead containing zinc chloride to promote separation by gravity thereof to form an upper layer of substantially only zinc chloride and a lower level of substantially only treated lead, and returning the treated lead substantially free from zinc chloride to the main pool of molten lead in substantially a continuous manner while preventing the outward flow of the zinc chloride with the treated lead.

10. The improved method of refining molten zinciferous lead with chlorine gas which comprises treating a portion of zinc-bearing molten lead withdrawn from a pool of molten zinciferous lead in a reaction chamber for converting the zinc contaminating said lead into zinc chloride, withdrawing the treated lead containing zinc chloride to a separatory chamber for effecting separation of the zinc chloride from the treated lead, equalizing the levels of the molten products of reaction in the reaction chamber and in the separatory chamber, controlling the level in said separatory chamber so that the level in the reaction chamber is maintained above the discharge opening thereof to prevent escape of chlorine gas therefrom, and returning the treated lead substantially free from zinc chloride to the pool of molten lead.

11. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a separate gas chlorinating chamber connected to said vessel for converting the zinc contaminating said molten lead into zinc chloride, and a separate chamber connected with said chlorinating chamber and with said vessel for effecting a separation of said zinc chloride from said treated lead and for returning treated lead substantially free from zinc chloride to said vessel.

12. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a separate gas chlorinating chamber connected to said vessel for treating portions of the zinciferous lead with chlorine gas for converting the zinc contaminating the lead into zinc chloride, means for circulating molten zinciferous lead from said vessel to said reaction chamber, means for introducing chlorine gas into the reaction chamber, a separate vessel connected with said chlorinating chamber and with said vessel for effecting the separation of zinc chloride from treated lead, means for transferring treated lead containing zinc chloride from the reaction chamber to the separatory vessel, and means for returning treated lead substantially free from zinc chloride to said vessel.

13. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a reaction chamber for treat-

ing portions of the zinciferous lead with chlorine gas for converting the zinc contaminating the lead into zinc chloride, means for circulating molten zinciferous lead from said vessel to said reaction chamber, means for introducing chlorine gas into the reaction chamber, a separatory vessel for effecting the separation of zinc chloride from treated lead by gravity, means for directing the flow of the treated lead containing zinc chloride from the reaction chamber to a point higher than the bottom of the separatory vessel for promoting separation by gravity thereof and for forming an upper layer of substantially zinc chloride and a lower level of substantially treated lead, and means for returning treated lead substantially free from zinc chloride to said vessel.

14. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a reaction chamber for treating portions of the zinciferous lead with chlorine gas for converting the zinc contaminating the lead into zinc chloride, means for circulating molten zinciferous lead from said vessel to said reaction chamber, means for introducing chlorine gas into the reaction chamber, a separatory vessel for effecting the separation of zinc chloride from treated lead by gravity, means for conveying the treated lead containing zinc chloride from the reaction chamber to the bottom of the separatory vessel, means for upwardly deflecting the flow of said treated lead containing zinc chloride for promoting separation by gravity thereof and for forming an upper layer of substantially zinc chloride and a lower level of substantially treated lead, and means for returning treated lead substantially free from zinc chloride to said vessel.

15. In an apparatus for refining molten zinciferous lead with chlorine gas, that improvement which comprises a separating vessel for effecting the separation of zinc chloride from treated lead, comprising a chamber having an inlet opening near the or at the bottom and an outlet opening, a vertical baffle located adjacent of said inlet opening and extending upwardly from the bottom of the chamber in an intermediate region to deflect upwardly the flow of the incoming molten lead containing zinc chloride, an internal closed duct leading to the outlet opening from a point near the bottom substantially for preventing the outward flow of the zinc chloride, a closed duct downwardly extending from the outlet opening to return the treated lead substantially free from zinc chloride.

16. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a separate gas chlorinating chamber connected to said vessel for converting the zinc contaminating said lead into zinc chloride, and a separate vessel connected with said chlorinating chamber and with said vessel for effecting a separation of said zinc chloride from said treated lead having enough capacity to hold substantially all the zinc chloride formed in refining substantially the entire pool of molten lead.

17. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a reaction chamber for converting the zinc contaminating said lead into zinc chloride, a separatory vessel for effecting a separation of said zinc chloride from said treated lead having enough capacity to hold substan-

5 tially all the zinc chloride formed in refining substantially the entire pool of molten lead, and having an outlet opening for returning treated lead substantially free from zinc chloride which is at substantially a higher level than the outlet opening of said reaction chamber.

10 18. An improved apparatus for refining molten zinciferous lead with chlorine gas which comprises a vessel for holding a pool of molten lead containing zinc, a reaction chamber for converting the zinc contaminating said lead into zinc

chloride, a separatory vessel for effecting a separation of said zinc chloride from said treated lead having enough capacity to hold substantially all the zinc chloride formed in refining substantially the entire pool of molten lead, and having a vertical baffle located adjacent to the inlet opening of said separatory vessel which extends upwardly from the bottom of the chamber to a level substantially higher than the outlet opening of said reaction chamber.

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10