Dec. 14, 1954 R. R. BARNES 2,697,050

RECIRCULATION OF WASTE PICKLE LIQUOR

Filed June 23, 1949 2 Sheets-Sheet 1

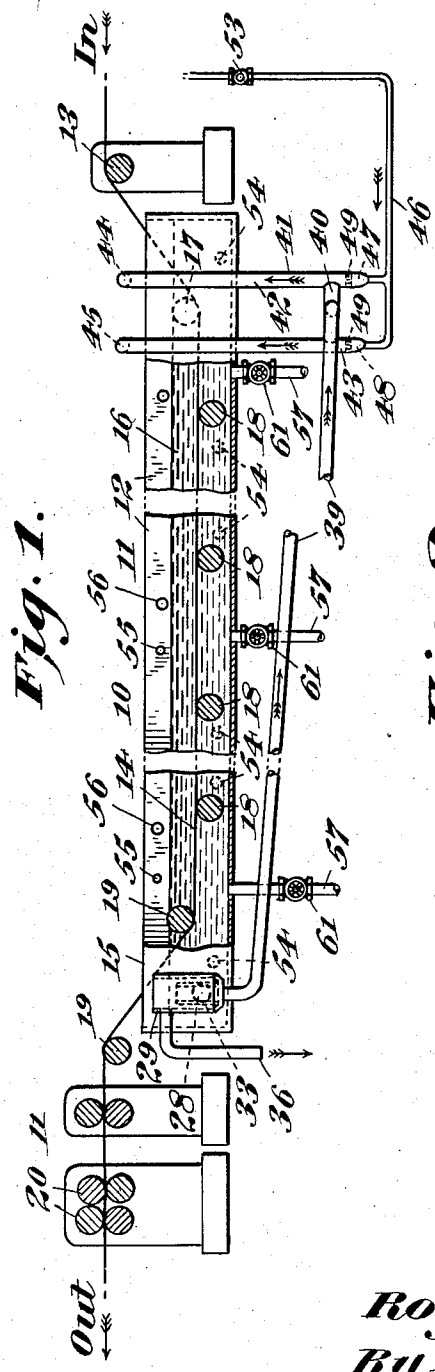


Fig. 1.

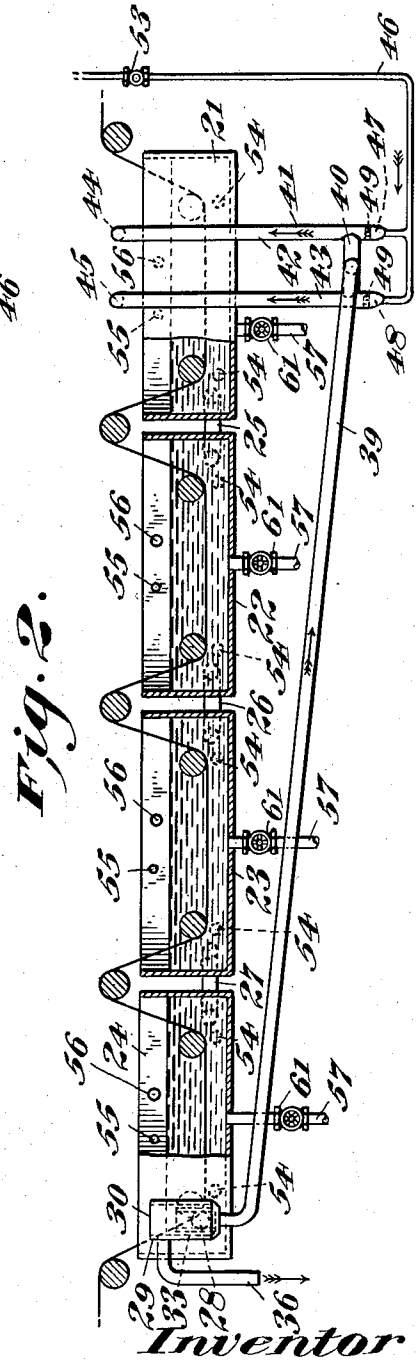


Fig. 2.

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RECIRCULATION OF WASTE PICKLE LIQUOR

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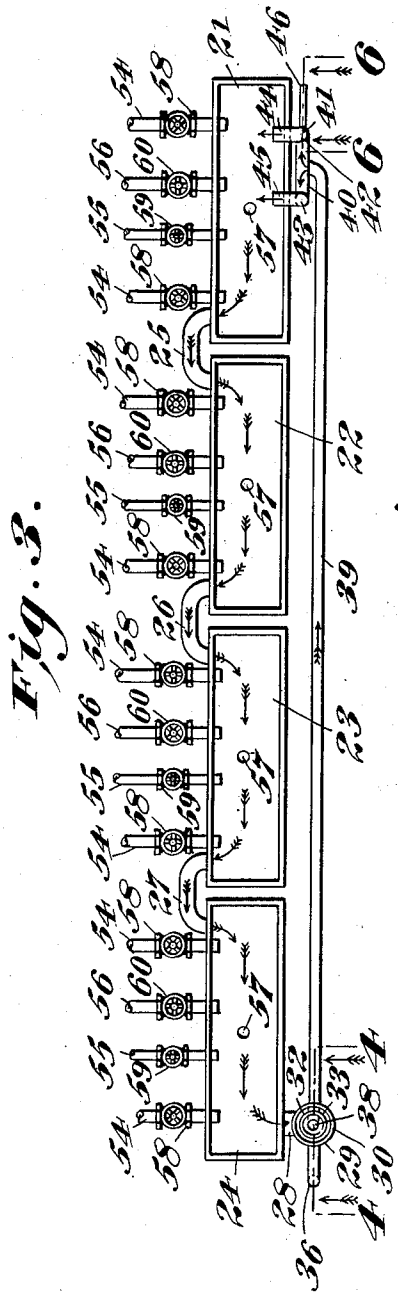


Fig. 3.

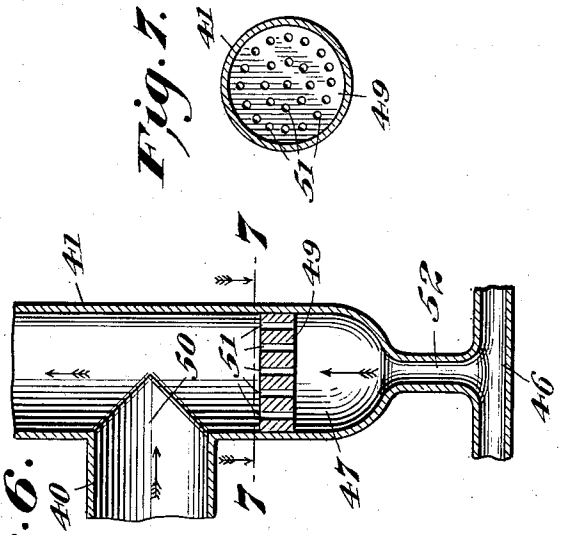


Fig. 6.

Fig. 7.

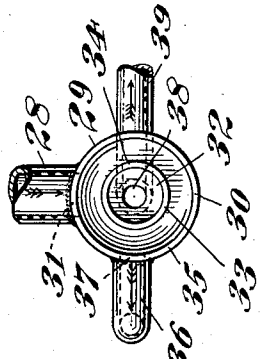


Fig. 5.

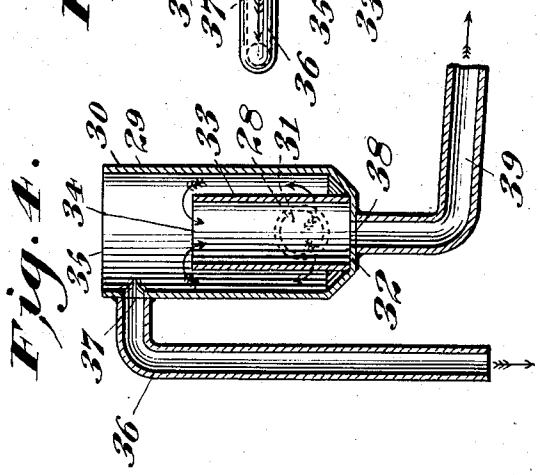


Fig. 4.

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Patented Dec. 14, 1954

2,697,050

RECIRCULATION OF WASTE PICKLE LIQUOR

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Application June 23, 1949, Serial No. 100,932

13 Claims. (Cl. 134—15)

This invention relates to improvements in process of and apparatus for pickling metal, and is particularly directed to an improvement in a process of and apparatus for pickling metal such as continuous strip metal or metal articles of general uniformity wherein the metal moves continuously through the pickling apparatus while simultaneously the pickling solution thereof is being continuously recirculated therethrough.

The prior art discloses numerous specific examples of so-called continuous pickling and it was early adapted for the pickling of steel sheets. Patents 1,392,780 and 1,392,781 were granted to Marsh et al. in 1921 covering a method of and apparatus for continuously pickling individual steel sheets one after another. There followed in 1925 a Patent 1,544,506 to Tytus disclosing the welding together of individual metal sheets to form a strip which simplified the pickling of the sheets in a continuous process. Next Fellows in his Patent 1,837,159 of 1931 disclosed a method of and apparatus for processing strip metal. These early inventors paved the way for the methods of and apparatus for the present day pickling of commercial broad strip metal used in large quantities by the industrial arts.

The present day practice in the pickling of continuous strip metal is believed to be largely carried on under methods of and apparatus for pickling disclosed in one or more of the following patents: 2,155,854 to Barnes et al. of April 25, 1939; 2,166,583 to Critten of July 18, 1939; and 2,216,544 to True of October 1, 1940. Each of these patents disclose improvements along different lines of approach to the old and well known problem of the economical and efficient use of and disposal of the used or so-called spent pickle solution per se. The patent to Barnes et al. is characterized by continuous regeneration of the pickle solution by continuously withdrawing a portion of the pickling solution from the pickling bath and in cooling this portion to reduce its ferrous sulphate concentration before returning it to the bath for reuse. The patent to Critten is characterized by the provision of extra tank capacity above that needed to maintain a continuous pickling operation, and as the pickling solution of a tank becomes inefficient for its position means are provided for temporarily taking this tank out of use without interfering with the apparatus as a whole while its contents are being discharged to a first tank, and the emptied tank is then recharged with fresh pickling solution and returned to service. The pickling solution discharged to the first tank is further used therein until finally exhausted and is then finally discharged to waste. The patent to True is characterized by means for continuously pickling strip metal comprising a plurality of pickling tanks and means for successively transferring pickling solution from a last tank to a preceding one contra the passage of the metal strip until the said pickling solution is discharged to waste from the first tank thereof. Summarized—Barnes et al. regenerates the pickling solution continuously by sulphate removal therefrom; Critten and True represent different methods of batch utilization to a point of sulphate concentration no longer considered efficient in their apparatuses.

My invention lies in an improvement in process of and apparatus for pickling continuous strip or the like characterized in having the pickling solution in its entirety continuously recirculated through the apparatus simultaneously with movement therethrough of the metal to be pickled without the specific regeneration of the pickling solution as a whole as is essential in the typical prior art disclosures of Marsh et al.; Fellows; and Barnes et al.; nor of the batch disposal methods of the so-called spent solution essential in the typical prior art disclosures of Tytus; Critten; and True.

My invention is further characterized by its adaptability to maintain a substantially constant uniformity in the physical and in the chemical characteristics of the pickling solution in its entirety during the pickling operation. To those skilled in the art it will be obvious that operating under known constant volume of pickling solution in continuous recirculation that desired changes in the chemical make-up thereof may be made by a withdrawn portion thereof being replaced by an equal volume of predetermined value with respect to acid and sulphate concentration. Furthermore by reason of continuous enforced recirculation of the entire pickling solution through the apparatus there is a continuous mixing effect brought about by the frictional resistances to relative flow between the pickling solution and the containing surfaces of the tanks and their interconnected piping, and between the pickling solution and the relatively rapidly moving metal, and that these mixing effects are further accentuated by the turbulence set up by pump operation in cyclically returning the pickling solution to the incoming end of the tank system. This continuous and rapid mixing effect throughout the extent of the pickling apparatus likewise tends to rapidly stabilize the desired regulated temperature of the pickling solution after withdrawals from and replacements to the pickling solution have been made. Furthermore this continuous and rapid intermixing is effective regardless of whether the passage of the metal through the pickling apparatus is in the direction of the flow of the pickling solution or contra thereof; and also regardless as to the use of a single long pickling tank or of a plurality of tanks interconnected for unidirectional flow therein.

I have found that this adaptability to maintain substantial stability of the entire pickling solution through continuous recirculation enables me to control the pickling operation thereof with assured efficiency, and to operate with a pronounced economy over the "once-through" type of operation of the prior art with respect to the amount of usable acid that is finally discharged to waste. It is also self-evident that the elimination of all auxiliary processes of and apparatuses for the regeneration of the pickling solution common in the prior art results in further economy.

It is therefore a primary object of my invention to provide improvements in process of and apparatus for the pickling of metal passing continuously through a pickling solution maintained at pickling strength wherein the said pickling solution in its entirety is being continuously recirculated successively through its container means and its interconnecting piping means in such a manner that the usual process steps for and apparatuses for the reduction of ferrous sulphate concentration and for batch improvement are eliminated as essential elements in the said pickling apparatus.

It is a further object of my invention to construct an apparatus for pickling strip metal passing continuously through a pickling solution maintained at pickling strength, wherein the said pickling solution is being continuously recirculated successively through a pickling tank system thereof and from the outgoing end of said tank system cyclically returned to the incoming end of said tank system solely through interconnected piping means under energized flow.

It is another object of my invention to maintain a desired uniformity of pickling strength in said continuously recirculated pickling solution by making withdrawals from and replacements to said pickling solution of predetermined volumetric values and of predetermined acid concentration thereof based on titrations made at stated intervals during the course of the pickling operation.

Further objects and advantages will be apparent from the following description wherein details of design and operation will be given in full with reference to the accompanying drawings illustrating the preferred embodiment of my invention, and wherein similar reference numerals designate similar parts throughout the several views.

Fig. 1 is a drawing showing in diagrammatic form a side elevation of apparatus embodying my invention for the pickling of continuous strip metal, and disclosing in schematic form a suitable strip actuating means as hereinafter referred to and described,

Fig. 2 is a drawing showing in diagrammatic form a side elevation of apparatus in part section illustrating a preferred embodiment of my invention for the pickling of continuous strip metal,

Fig. 3 is a plan view thereof with the metal strip and its actuating mechanism removed and showing in particular the recirculation flow of the pickling solution through the apparatus,

Fig. 4 is an enlarged fragmentary section of the overflow **29** taken along the line **4**—**4** of Fig. 3,

Fig. 5 is a plan view in full of Fig. 4,

Fig. 6 is an enlarged fragmentary section of the air-jet pump taken along the line **6**—**6** of Fig. 3, and

Fig. 7 is an enlarged fragmentary view taken along the line **7**—**7** of Fig. 6.

Referring now to the drawings and more particularly for the moment to Fig. 1, the numeral **10** designates the complete ensemble of an apparatus for pickling continuous strip metal embodying essential units of my invention. As shown in Fig. 1 the apparatus **10** comprises two substantially independent units—a strip actuating means **11**, and a pickling unit **12**.

The strip actuating means **11** forms no part of my invention except to the extent in which it is an essential cooperating element along with the equally essential pickling unit **12** in a complete pickling system. The strip actuating means **11** in a pickling apparatus **10** embodying my invention may be of any suitable construction adapted to continuously pass metal strips through and submerged in a pickling solution of the pickling unit **12** per se, and therethrough in either direction with respect to the flow therethrough of the recirculating pickling solution. The specific showing of Fig. 1 with regard to this latter relationship of the strip actuating means **11** and of the pickling unit **12** is a modification of my invention and not a limitation thereto.

Typical strip actuating means are disclosed in Letters Patent 1,765,498 granted to Naugle et al. June 24, 1930, and reference may be made thereto for the details thereof. In Fig. 1 I have schematically indicated such a suitable strip actuating unit as including a guide roll **13** (7) from which the work strip **14** under treatment is here shown as being directed into the forward end of an elongated pickling tank **15** (8), and held in submergence therein in the pickling solution **16** (8*a*) by guide roll **17** (8*b*), and supported therein on rolls **18** (8*c*) while it is moving endwise therethrough. The work strip **14** is continuously drawn through the tank **15** (8) and leaves the opposite end thereof under the direction of guide rolls **19** (12*a*) and under the driving action of the power driven pinch rolls **20** (14). These elements are old and well known in the art, and the numerals appearing in parentheses are those used to designate the same parts in the above noted Patent 1,765,498, granted to Naugle et al., June 24, 1930, to which reference may be had for disclosure in full of a suitable strip actuating means **11**.

In Fig. 1 I have shown my invention embodied in a pickling unit **12** comprising a single long tank **15**. In Figs. 2 and 3 I show a preferred embodiment of my invention as applied to a pickling unit comprising a plurality of pickling tanks **21**, **22**, **23**, and **24** interconnected for liquid flow therethrough successively from the first tank **21** to the last tank **24** by means of interconnecting pipes **25**, **26**, and **27** respectively. It is obvious that the pipes **25**, **26**, and **27** are unnecessary in Fig. 1, and since otherwise the apparatuses in Fig. 1 and in Figs. 2 and 3 are identical, that a description of my invention as applied to Figs. 2 and 3 will suffice for a clear understanding of its adaptation to the single long tank **15** of Fig. 1.

As shown in Figs. 2 and 3 the outgoing end of the tank **24** comprises the pipe **28** communicating the interior of the tank **24** with the interior of the safety overflow **29**. Referring now to Figs. 4 and 5—the safety overflow **29** comprises a pipe-like open-top container **30** having an inlet opening **31** from the pipe **28** near its bottom **32** thereof. Rising within the container **30** and attached to the bottom **32** thereof is a smaller diameter open-top pipe **33** having its top peripheral edge **34** extending above the inlet opening **31** but spaced from and below the top **35** of the container **30**. An emergency outlet overflow

pipe **36** has its discharge opening **37** through the wall of the container **30** spaced from and above the top **34** of the pipe **33** but below the top **35** of the container **30**. An outlet opening **38** in the bottom **32** of the container **30** and within the confines of the pipe **33** communicates the container **30** and the inner pipe **33** with the solution gravity flow interconnecting pipe **39**.

The interconnecting pipe **39** (Fig. 2) slopes downwardly from the safety overflow **29** at the solution outgoing end of tank **24** to communicate with the connecting pipe **40** of the solution elevating piping **41** at the solution incoming end of tank **21**. The solution elevating piping **41** comprises the connecting pipe **40** communicating the interconnecting pipe **39** with each of the two air-jet elevating pipes **42** and **43** rising vertically to outlet ends **44** and **45** respectively adapted to discharge into the solution incoming end of tank **21**. At their lower ends the elevating pipes **42** and **43** extend downwardly below the connecting pipe **40** to communicate with the air pipe **46** and from air-jet pump chambers **47** and **48**. The air-jet pump structure is shown in enlarged detail in Figs. 6 and 7 for pump chamber **47** and is equally applicable for pump chamber **48**. The air-jet pump chamber **47** comprises a diaphragm **49** disposed transverse the downward extension of the elevation pipe **42** and below the communicating opening **50** in the connecting pipe **40**. The diaphragm **49** is perforated with a plurality of passageways **51** of relatively small diameter. Below the diaphragm **49** the pump chamber **47** is contracted to form a passageway **52** communicating with the air pipe **46**. The air pipe **46** communicates with a source of air under pressure (not shown) and is provided with a control valve means **53**.

My invention is adapted to utilize certain conventional operating means for the heating of the pickling solution, and for making withdrawals from and replacements to the said pickling solution in the tanks. As examples of such means the Figs. 1, 2, and 3 indicate for use in each of the tanks **15** of Fig. 1, and **21**, **22**, **23**, and **24** of Figs. 2 and 3 of heating pipes **54** for injecting live steam into the pickling solution; acid pipes **55** and water pipes **56** for convenience in adding pickling solution; and drain pipes **57** for making pickling solution withdrawals from said vats. Each of the pipes **54**, **55**, **56**, and **57** are provided with suitable valve control means **58**, **59**, **60**, and **61** respectively.

The work strip of Fig. 2 is shown being guided into and out of the individual pickling tanks **21**, **22**, **23**, and **24** by strip actuating means similar to that shown in Fig. 1, and it is to be understood that when the pickling apparatus is in operation the work strip is being continuously passed through the pickling apparatus of Fig. 2 by similar means as shown and described for Fig. 1, and its structure need not be here repeated.

In preparation for the pickling of continuous strip metal the pickling unit **12** of my invention will have been filled with suitable pickling solution to its operating capacity as determined by the height of the overflow peripheral edge **34** of the open-top pipe **33** of the safety overflow **29**. In filling the tank or tanks of the pickling unit **12** the pickling solution will seek its level throughout the extent thereof including the safety overflow **29** in communication with the outgoing end of the tank system thereof. When the solution level in the unit **12** reaches the level of the overflow edge **34** and continues to rise the excess thereabove will discharge over the edge **34** and flow downwardly through the interconnecting pipe **39** to the solution elevating piping **41** at the incoming end of the tank system, and will then rise in the air-jet elevating pipes **42** and **43** until the solution level in unit **12** is reached. At this stage a slight operating excess is allowed in the unit **12** by setting the solution level therein at an intermediate height between the overflow edge **34** and the emergency overflow pipe **36** of the safety overflow **29** sufficient to maintain the continuous recirculation of the pickling solution within the pickling unit **12** at the capacity of the air-jet pumps when in operation. With the pickling unit **12** suitably filled with pickling solution it may be put into recirculating operation by opening the air control valve **53** to air pressure, thereby putting the air-jet pumps **47** and **48** into action, whereby the entire body of pickling solution in the pickling unit **12** is placed in continuous cyclical recirculating flow due to the discharge of the air-jet pumps flowing the pickling solution into the in-

coming end of and through the tank system, and thence successively through the safety overflow 29, the interconnecting pipe 39, the solution elevating piping 41, and back again into the solution incoming end of the tank system, and whence in continuous repetition of this cyclical recirculation through the pickling unit 12.

With the pickling unit 12 in recirculating operation and the pickling solution properly heated to the desired temperature by means of the heating pipes 54 under control of the valves 58, the strip actuating unit 11 is next placed in operation to continuously pass metal strip through and submerged in the pickling solution of the pickling unit 12.

During the pickling operation titrations are made at different points in the vat system and at stated intervals of time. Based on these observations withdrawals from the solution body and replacements thereto of acid and water are made in suitable proportions to modify and maintain the pickling strength thereof in accordance with the operating requirements of the specific work strip undergoing treatment. A certain constant loss of volumetric capacity due to the dragout of the strip is compensated for in the additions made. If live steam jets are used as the heating means the condensate therefrom tends to offset the dragout loss, and a continuous addition of a small flow of water may be used to maintain the vat capacity in balance.

I claim:

1. Apparatus for pickling continuous metal strip, comprising elongated tank means adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into a first end of said tank means and with outlet piping means for continuously discharging an equivalent quantity of pickling solution from its opposite end thereof whereby a continuously flowing pickling bath is maintained therebetween, means for continuously passing metal strip submerged in said bath therethrough, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution in its entirety within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

2. Apparatus for pickling continuous metal strip, comprising a plurality of pickling tanks suitably interconnected for continuous liquid flow successively from a first one to a last one thereof, said first one of said tanks being adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into said first tank, and said last one of said tanks being adapted with outlet piping means for continuously discharging therefrom an equivalent quantity of pickling solution to that received in said first tank whereby a continuously flowing pickling bath is maintained flowing successively through said tanks, means for continuously passing said metal strip submerged in said bath through each of said tanks, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution in its entirety within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

3. Apparatus for pickling metal, comprising elongated tank means adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into a first end of said tank means and with outlet piping means for continuously discharging an equivalent quantity of pickling solution from its opposite end thereof whereby a continuously flowing pickling bath is maintained therebetween, said outlet piping means comprising means for maintaining an operating solution level in said tank means, means for continuously passing metal submerged in said bath therethrough, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

4. Apparatus for pickling continuous metal strip, comprising elongated tank means adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into a first end of said tank means and with outlet piping means for continuously discharging an equivalent quantity of pickling solution from its opposite end thereof whereby a continuously flowing pickling bath is maintained therebetween, said outlet piping means comprising means for maintaining an operating solution level in said tank means, means for continuously passing metal strip submerged in said bath therethrough, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

5. Apparatus for pickling continuous metal strip, comprising a plurality of pickling tanks suitably interconnected for continuous liquid flow successively from a first one to a last one thereof, said first one of said tanks being adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into said first tank, and said last one of said tanks being adapted with outlet piping means for continuously discharging therefrom an equivalent quantity of pickling solution to that received in said first tank whereby a continuously flowing pickling bath is maintained flowing successively through said tanks, said outlet piping means comprising means for maintaining an operating solution level in said tanks, means for continuously passing said metal strip submerged in said bath through each of said tanks, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

6. Apparatus for pickling metal, comprising elongated tank means adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into a first end of said tank means and with outlet piping means for continuously discharging an equivalent quantity of pickling solution from its opposite end thereof whereby a continuously flowing pickling bath is maintained therebetween, said outlet piping means comprising means for maintaining an operating solution level in said tank means and means for discharging to waste abnormal excess solution level therein, means for continuously passing metal submerged in said bath therethrough, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

7. Apparatus for pickling continuous metal strip, comprising elongated tank means adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into a first end of said tank means and with outlet piping means for continuously discharging an equivalent quantity of pickling solution from its opposite end thereof whereby a continuously flowing pickling bath is maintained therebetween, said outlet piping means comprising means for maintaining an operating solution level in said tank means and means for discharging to waste abnormal excess solution level therein, means for continuously passing metal strip submerged in said bath therethrough, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whrereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

8. Apparatus for pickling continuous metal strip, comprising a plurality of pickling tanks suitably interconnected for continuous liquid flow successively from a first one to a last one thereof, said first one of said tanks being

adapted with inlet piping means for continuously passing a regulated quantity of pickling solution into said first tank, and said last one of said tanks being adapted with outlet piping means for continuously discharging therefrom an equivalent quantity of pickling solution to that received in said first tank whereby a continuously flowing pickling bath is maintained flowing successively through said tanks, said outlet piping means comprising means for maintaining an operating solution level in said tanks and means for discharging to waste abnormal excess solution level therein, means for continuously passing said metal strip submerged in said bath through each of said tanks, interconnecting piping means communicating said outlet piping means with said inlet piping means, said interconnecting piping means being adapted for energized flow of pickling solution therethrough for delivery to said inlet piping means whereby the pickling solution within said apparatus is cyclically maintained in continuous recirculation therethrough, and means for heating said pickling solution.

9. A process for the pickling of metal continuously passing immersed through a pickling bath, comprising the energizing of the pickling solution of said pickling bath in its entirety for continuous cyclical recirculation through said bath and for repetitively immersed contact with said metal, and regulating the chemical characteristics of said pickling solution by making periodic withdrawals from and replacements thereto of predetermined volumetric and acid concentration values thereof during said pickling operation.

10. A process for the pickling of strip metal continuously passing immersed through a pickling bath, comprising the energizing of the pickling solution of said pickling bath in its entirety for continuous cyclical recirculation through said bath and for repetitively immersed contact with said metal strip, maintaining a substantially uniform regulable temperature of said pickling solution, and regulating the chemical characteristics of said pickling solution by making periodic withdrawals from and replacements thereto of predetermined volumetric and acid concentration values thereof during said pickling operation.

11. A process for the pickling of metal continuously passing through a longitudinally disposed pickling bath, comprising the heating of and the energizing-for-flow of the pickling solution of said pickling bath in its entirety for continuous unidirectional cyclical recirculation through said bath and through bath terminals interconnecting return conduit therefor, thereby effecting uniform heating of said pickling solution and repetitively immersed contact thereof with said metal.

12. A process for the pickling of metal continuously passing through a longitudinally disposed pickling bath, comprising the heating of and the energizing-for-flow of the pickling solution of said pickling bath in its entirety for continuous unidirectional cyclical recirculation through said bath and through bath terminals interconnecting return conduit therefor, thereby effecting uniform heating of said pickling solution and repetitively immersed contact thereof with said metal, and regulating the chemical characteristics of said pickling solution by making periodic withdrawals from and replacements thereto of predetermined volumetric and acid concentration values thereof during said pickling operation.

13. A process for the pickling of metal continuously passing through a longitudinally disposed pickling bath, comprising the heating of and the energizing-for-flow of the pickling solution of said pickling bath in its entirety for continuous unidirectional cyclical recirculation through said bath and through bath terminals interconnecting return conduit therefor, thereby effecting uniform heating of said pickling solution and repetitively immersed contact thereof with said metal, maintaining a substantially uniform regulable temperature of said heated pickling solution, and regulating the chemical characteristics of said pickling solution by making periodic withdrawals from and replacements thereto of predetermined volumetric and acid concentration values thereof during said pickling operation.

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