METHOD AND APPARATUS FOR MAINTAINING A CONSTANT BOILING LIQUID LEVEL IN VAPOR SOLVENT DEGREASING OPERATION

FIG. 1

FIG. 2

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METHOD AND APPARATUS FOR MAINTAINING A CONSTANT BOILING LIQUID LEVEL IN VAPOR SOLVENT DEGREASING OPERATION

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Filed May 14, 1962, Ser. No. 194,270
4 Claims. (Cl. 134—11)

This invention relates to apparatus for use in the control of a process for the treatment of metals and other materials with a boiling chlorinated solvent composition. It relates particularly to an improved apparatus for maintaining a boiling treating bath comprising a chlorinated solvent composition at a substantially constant level and composition.

Chlorinated solvents are the major constituents of a variety of treating baths in which metals and other materials are cleaned, coated or modified by immersion in the boiling bath. Such processes include degreasing and cleaning in which metals, cloth fabrics and other materials are dipped into a substantially pure solvent. Processes in which the material treated is modified include metal conversion coating in which the boiling chlorinated solvent treating bath contains chemical agents, such as phosphoric, chromic and/or oxalic acid in combination with various bath adjuvants. In all of these processes, small amounts of the treating bath are continually removed with the treated materials by "drag-out" and by diffusion across the interface between the air and the vapor above the boiling bath since the treating vessel must be open for the addition and removal of the various materials to be treated. As a result, the boiling bath level in the treating vessel drops off and the path composition must be replenished from time to time. When this procedure is followed, the level of the treating bath is constantly changing and it is sometimes too low and sometimes too high. In the case of solvent compositions containing treating agents which are present differentially due to their action on the materials treated, non-continuous addition of a maintenance solution impairs the uniformity of the treating process since, under these conditions, the concentration of the active treating agents varies between upper and lower extremes and is seldom at the optimum value.

Methods for level maintenance in liquid baths wherein the gas above the liquid is a permanent gas at the ambient temperature are well known. A device of this type is commonly employed for watering poultry. However, these methods have not been adapted to maintaining the level of a boiling bath in which the gas phase over the liquid consists of vapors which undergo substantially complete condensation at ambient temperatures and have a specific gravity of about three or greater with respect to air.

It is an object of this invention to provide an apparatus for maintaining a boiling treating bath comprising a chlorinated solvent at a substantially constant level below an atmosphere consisting substantially of said boiling bath vapor.

It is a further object to provide an apparatus for maintaining a boiling bath comprising a chlorinated solvent in combination with a minor proportion of treating agent at a substantially constant level and composition.

Still other objects of the invention will become apparent from the following description.

The above-mentioned objects are accomplished by means of an apparatus for the treatment of articles with a boiling bath comprising a chlorinated solvent by the improvement comprising the combination of:

1. A treating vessel in which a chlorinated solvent composition is maintained at a constant boil and the vapors are kept at a controlled level well above the bath level;

2. An enclosed reservoir, in which liquid solvent bath maintenance composition is kept at a temperature below its boiling point, said reservoir having an opening positioned below the liquid level in said reservoir but above the boiling bath level in the treating vessel;

3. A conduit leading downwards from the opening in the reservoir into the treating vessel to the level at which it is desired to maintain the boiling liquid treating bath.

The boiling bath of this invention comprises as its major ingredient a chlorinated solvent whose vapors at the boiling point, which is preferably at about 70° C., have a density at least about three times that of air at the same temperature. Other bath constituents are present to the extent of not more than about 10% by weight. These include solvent stabilizers, chemical treating agents and various adjuvants, such as solvents, catalytic activators, etc.

The vapor level of the boiling bath vapors in the treating vessel is maintained at least one foot above the boiling liquid and is preferably about two to ten feet above this level. The conduit leading from the reservoir into the treating vessel should preferably have a diameter ranging from about three-quarters of an inch to about one inch although somewhat smaller and larger diameters are acceptable. The conduit must lead downward from the reservoir and, therefore, it may proceed on a level for short intervals, must never sink below the level at which the bath is to be kept in the treating vessel or have a reverse upwards curve which would impair the constant feed of solution from reservoir to bath.

It is preferable for the operation of the invention that the opening in the reservoir be at the bottom thereof and be located at a point higher than the level at which the boiling bath is to be maintained in the treating vessel. It should also be noted that since it is impossible to support liquid in a reservoir at level equivalent to a hydraulic head greater than atmospheric pressure, there is no advantage in a reservoir positioned higher than compatible with such a liquid level. Preferably, the hydraulic pressure head (H in FIGURE 1) should not be more than about 50% of the maximal theoretical value. This maximal value in feet can be calculated from the vapor pressure of the bath in millimeters of mercury at the reservoir temperature of the bath and the density of the bath at reservoir temperature, by means of the following formula in which the maximal head in feet equals

\[ H = \frac{(760 - P_b)}{30.5} \times D_b \]

The figure 760 in this equation is the value for atmospheric pressure in millimeters of mercury and should be replaced by the actual barometer reading for accuracy. The figure 30.5 is the density of mercury and the figure 305 is the conversion factor which converts millimeters to feet. A value of approximately 21 feet is obtained when the bath consists of trichloroethylene at a reservoir temperature of 30° C. when the value for the vapor pressure of trichloroethylene is 80 mm. and its density is about 1.46. With trichloroethylene at 40° C. when its vapor pressure is 132 mm., the maximal head value would be 19 feet. As can be seen from these figures, the preferred maximum head value for trichloroethylene at a reservoir temperature of 30° C. to 40° C. would be about 10 feet.

The success of this invention is far from obvious in
that the level of the boiling bath is maintained in spite of the fact that the gas over the boiling liquid is substantially completely condensable at ordinary temperatures and there is no apparent way in which any quantity of peroxide gas could replace the chlorinated solvent bath maintenance composition in the reservoir. The apparatus combination of this invention including a treating vessel in which the bath is maintained at a constant boil was at first believed by many skilled technical personnel to be obviously inoperative and yet was found to work in a satisfactory manner. This is particularly surprising in view of the fact that the vapor layer over the boiling bath has a density much greater than air. The leveling device operates even when the bath is held in the boiling state and no articles are added and withdrawn for treatment so that the only bath loss is by gradual diffusion of bath vapor into the air. Accordingly, the disturbing effect on the vapor phase of the movement of articles being treated has apparently no part in determining the operability of the invention.

The apparatus improvement of the invention is illustrated schematically by the figures.

FIGURE 1 represents a vertical cross section of the apparatus combination of the invention.

FIGURE 2 is a vertical side view of the apparatus showing a modified form of the combination.

Both figures show the essential elements of the combination of this invention. These are treating vessel 1 in which the boiling bath is maintained, bath reservoir 16 and the conduit, comprising items 8, 13 and 15, through which the bath composition in the reservoir passes to the treating vessel as required to maintain the boiling bath at a constant level.

FIGURE 1 shows treating vessel 1 in vertical cross section. Boiling treating bath 10 is kept boiling by heating means 6 which may consist of electric heating elements, heating coils for the passage of steam or other hot fluid media, etc. with outlet pipes or power contacts 7. Alternatively these heating means may be replaced by external gas flames or other heating means. Above the surface of the boiling treating bath are cooling means 3 which may consist of cooling coils as shown with external inlet 4 and outlet 5. These may alternatively be replaced or supplemented by an external cooling jacket. Vapor level 12 controlled by the cooling means is normally located 2 to 6 above the boiling bath level 20 but may be up 10 feet or more above the bath level in some equipment. Trough 2 located below the cooling means is adapted to collect condensed fluid so that it may be removed and separated from trace contamination with water or otherwise purified for recovery. Conduits for removal of condensate from trough 2 and possible return to a point below the surface of the boiling bath are not shown. Presence or absence of a condensate trough system are not an essential part of the subject invention and may or may not be included in the treating vessel.

Bath reservoir 16 as shown may consist of a drum or tank mounted on a support so its fluid contents 18 are above the level 20 which is to be maintained in the treating vessel 1. It may be held by an independent support 17 as shown in FIGURE 1 or by a supporting arm or frame 20 on the side of the treating vessel as shown in the modified combination in FIGURE 2. The conduit for passage of the bath composition from the reservoir to the treating vessel normally consists of pipe 15 with valve 14 leading from the bottom part of the reservoir, pipe 8 with valve 9 leading into the treating vessel at the level to be maintained by the boiling bath and flexible tube 13 connected to valves 15 and 8. The valves are kept open when the invention is in use and are useful for closing off the pipes when the reservoir is not attached to the treating vessel or when the bath has been allowed to cool down at times when the treating process is not in operation. Since the bath level drops when it is cooled or even when it is not boiling, at least one of the valves is closed so that when it is desired to start operating, the bath will not be too high as would be the case if it were allowed to come to the desired level when the bath was cold. The liquid head from the top of the reservoir at bath level 19 to the chlorinated solvent bath maintenance level 20 is indicated by the dotted line H in FIGURE 1.

It should be noted that the invention is not limited to the specific apparatus shown in the figures but is limited only to the combination of a treating vessel containing a boiling chlorinated solvent bath, reservoir and conduit. Numerical limitations in this combination will be readily apparent to anyone skilled in the art. As an example, it should be noted that instead of cutting through the wall of the treating vessel as shown in the figures, the conduit may enter the bath at the top or at any point above the boiling bath level and then proceed downward to the bath level. A pipe entering the bath through the open top can be adjusted to various bath levels when desired and has a flexibility not shown by the apparatus in FIGURE 1.

It should be noted that when the bath is operated with a chlorinated solvent containing treating agents which are lost differentially by their action on the materials treated which tends to exhaust them from the bath, the bath maintenance solution will not contain the same concentration of additives as the boiling bath itself. The concentration of these agents in a maintenance bath in such cases will be determined by the experimental study of the bath in question and is a function of the rate at which these constituents are lost under operating conditions.

As previously stated, the preferred chlorinated solvents for use in the bath of this invention are those which have a boiling point at least of about 70°C. and whose boiling vapor has a density of at least three times that of air at the same temperatures. Such solvents include trichloroethylene, perchloroethylene, carbon tetrachloride, 1,1,1-trichloroethylene and ethylene dichloride.

I claim:

1. In an apparatus of the type used for the treatment of articles with a boiling treating bath composition comprising a chlorinated solvent, whose vapor at the boiling point has a density of at least three times that of air at the same temperature, the improvement whereby such a boiling bath may be maintained automatically at a predetermined and substantially constant level below a dense atmosphere of bath vapor, said improvement comprising, in combination, (a) an open-topped treating vessel adapted to contain said bath composition and equipped with heating means, whereby said bath composition may be maintained at a constant boil, and cooling means, whereby boiling bath vapor may be contained as a dense vapor zone extending from directly above said boiling bath to a controlled level well above the predetermined boiling level,

(b) an enclosed reservoir adapted to contain a maintenance composition for said bath composition and having an opening positioned at a low level in said reservoir but above the predetermined boiling bath level in said heating vessel, and

(c) a conduit leading downwards from said reservoir opening into said treating vessel at the predetermined level at which it is desired to maintain said boiling bath composition.

2. The apparatus of claim 1 wherein the vertical distance between the point at which the said conduit enters the treating vessel and the controlled level at which said cooling means are adapted to maintain the top of the dense vapor zone is in the range of 2 to 10 feet.

3. In a process for treating articles with a boiling liquid comprising a chlorinated solution whose vapor at the boiling point has a density of at least three times that of air at the same temperature, the improvement for maintaining said boiling path at a substantially constant level
below an atmosphere of bath vapor by continuous addition of a solvent maintenance composition, comprising
the steps of:

(a) maintaining said chlorinated solvent bath composition at the boiling point in a treating vessel while
maintaining the bath vapor at a controlled level well above the boiling bath,
(b) providing liquid chlorinated solvent maintenance composition for said chlorinated solvent bath in an
enclosed reservoir having an opening positioned below the liquid level in said reservoir but above the
boiling bath level in said treating vessel, and
(c) allowing said liquid maintenance composition to pass from said reservoir to said treating vessel by
means of a conduit leading downwards from said opening in said reservoir to the level at which it is
desired to maintain said boiling liquid in said treating vessel.

4. The process of claim 3 in which the boiling bath vapor level is kept 2 to 10 feet above the boiling bath level.

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