United States Patent Office 3,437,521
Patented Apr. 8, 1969



3,437,521

# RADIOACTIVE DECONTAMINATION

Daniel T. Buist, Rolling Hills Estates, Calif., assignor to Purex Corporation, Ltd., Lakewood, Calif., a corporation of California

No Drawing. Filed Jan. 21, 1964, Ser. No. 339,096
Int. Cl. B08b 3/08
U.S. Cl. 134—26 16 Claims

This invention relates to removal of radioactive contaminants from surfaces, and is particularly concerned with novel procedure and compositions for treating surfaces with effective decontaminating solutions, and preferably involving minimum waste disposal of the spent treating solutions containing radioactive contaminants.

Components or parts of equipment employed in the presence of radioactive matter generally become contaminated with such radiocative matter. These parts may include valves, pumps, agitators, flanges, pipe sections, tools, and the like. Such radioactive contamination of parts can result from the use of radioactive isotopes, or such parts can become radioactively contaminated as result of use as components, for example, of an experimental or research atomic reactor. Such accumulation of radioactive contaminants increases to the extent that, in the absence of proper decontamination, exposure rates for personnel involved in contact maintenance of the equipment poses a major problem.

Various prior art decontamination methods have been proposed but often without producing the desired high degree of decontamination and freedom from corrosion desired in practice. Also, prior art processes often are inefficient and may require time-consuming rinsing procedures for properly removing treating materials or solutions from the parts being processed.

Further, in removing radioactive contaminants from surfaces of parts exposed to radioactive emission, employing chemical treating solutions for this purpose, the spent treating solutions resulting from such decontamination procedures and containing radioactive contaminants must be sewered or in some manner disposed of with minimum danger to plant and animal life, and to human beings, from the effects of such radioactive contaminants. However, when treating parts for removal of radioactive contamination, large volumes of such treating solutions are often necessary, thus posing a serious problem of safe disposal of the resulting radioactive contaminant-bearing waste solutions.

It is an object of the invention to provide procedures and compositions for removing radioactive contaminants from surfaces, parts or equipment exposed to radioactive materials.

Another object of the invention is to provide a procedure for carrying out thorough and efficient washing and rinsing operations employing effective processing solutions, and designed to effect complete and rapid removal of radioactive contaminants from part surfaces.

Another object is the provision of procedures and compositions for removal of radioactive contaminants from surfaces, involving a substantial reduction in the quantities or volumes of spent treating solution containing radioactive contaminants which constitute waste solutions, thus substantially alleviating the disposal problems.

Other objects and advantages will appear hereinafter.

I have found that radioactive decontamination of a part bearing radioactive material can be effectively accomplished by a process comprising a first step of treatment in a hot liquid or solution containing a decontaminating agent, and which may also contain a chlorinated hydrocarbon, followed by treatment of the part, after partial cooling thereof, with hot vapors of a chlorinated hydrocarbon. The first stage operation functions to remove substantially all or at least the major portion of the radioactive contaminants from the article undergoing treatment, and the second stage operation functions to remove the remainder of the radioactive contaminants from such article, and particularly acts as a rinse in this respect.



In preferred practice, the article to be decontaminated is immersed in the hot decontaminating solution of the first stage operation, which is preferably at or near its boiling point, the article is then further washed to remove residual radioactive contaminants and cooled at least partially in a second solution containing a chlorinated hydrocarbon liquid maintained at a temperature substantially below the temperature of the first solution, and the cooled article is then placed in a zone in contact with hot chlorinated hydrocarbon vapors at about the boiling point thereof. Such vapors condense on the article, drain off, and so rinse the article free of any adhering decontaminating solution or residual radioactive contaminants. Such hot chlorinated hydrocarbon vapors can be supplied from a boiling solution of such chlorinated hydrocarbon positioned adjacent to the second solution, or such hot chlorinated hydrocarbon vapors can be supplied by the hot first-stage decontaminating solution where the latter contains a chlorinated hydrocarbon.

Although not preferred, if desired, the article can be cooled in air or by other means following treatment in the first stage hot decontaminating solution, to reduce the temperature of the article, and the article then placed directly in a zone in contact with hot chlorinated hydrocarbon vapors, to carry out the above-noted rinsing of the article by the condensation of such vapors on the article.

It has been found that by the use of a chlorinated hydrocarbon of low boiling point having a substantial vapor pressure, preferably trichloroethylene, such rinsing of the article can be achieved readily by placing the article in the hot trichloroethylene vapors heated to a temperature above the temperature of the article. This feature aids in subsantially reducing the total volume of spent treating solutions employed in the decontamination process.

The total volume of waste matter can be further concentrated by distilling off the chlorinated hydrocarbon solvent employed in the treating solutions of the process.

Thus, it has been found that the use of chlorinated hydrocarbon solvents in treatment solutions for radioactive decontamination are effective in (1) facilitating the decontamination operation when employing such solvents in conjunction with decontaminating agents, usually in the presence of a mutual solvent; (2) functioning as a rinse medium for the article being treated; and (3) alleviating the waste disposal problem by reducing the total volume of decontaminating solution required in the process and by being itself easily removable by distillation or other chemical means.

In preferred practice, the first stage treating liquid can consist essentially of a chlorinated hydrocarbon liquid solvent, a decontaminating agent, and a mutual solvent or coupling agent effective to render the decontaminating agent soluble in the chlorinated hydrocarbon. Chlorinated hydrocarbon liquid solvents which are suitable for use in the first stage treating solutions are preferably those which are nonflammable, do not attack common metals such as steel, aluminum, and the like, and have a boiling point not so high as to tend to decompose the decontaminating agents employed therein, as described below, when the first stage treating solution is used in its normal way as a hot solution at or near the boiling point thereof.

The chlorinated hydrocarbon solvent employed should also be stable in the presence of acids and alkalies. Trichloroethylene is considered the best solvent for purposes of the invention. While perchloroethylene is suitable, since it has a substantially higher boiling point, 121° C., as

compared to trichloroethylene having a boiling point of only 86° C., perchloroethylene when used in a first stage treating solution at or near its boiling point tends to decompose certain decontaminating agents such as, for example, oxalic acid and sulfamic acid. Ethylene dichloride is another example of a chlorinated hydrocarbon solvent which can be employed.

The decontaminating agent or decontaminant employed in the first stage treating solution in conjunction with the chlorinated hydrocarbon solvent can include a variety of materials, both acidic and alkaline. Acid decontaminants which can be employed include oxalic, citric, sulfamic, tartaric, and gluconic acids, and acid salts such as, for example, sodium acid sulfate, monosodium phosphate and monoammonium citrate. Phosphoric acid can also be employed as a decontaminant. Oxalic and citric acids are preferred acid decontaminants. Mixtures of the above-noted acid materials can also be employed as decontaminant.

Various alkaline decontaminating agents which can be employed include mixtures such as those described in Patent No. 3,108,080, to Brevik, and including glassy phosphate and an ammonium carbonate, for example, a composition of about 50% glassy phosphate, e.g., sodium polyphosphate, and about 50% of ammonium carbonate or ammonium bicarbonate. Other phosphates such as trisodium phosphate, per se, can be employed as a decontaminant. Additional examples of alkaline decontaminating agents include the compositions described in Patent Nos. 2,992,995 and 2,992,997, to Arden. These are basically alkaline compositions containing alkali and an alkanolamine such as polyalkanolmonamine and/or alkanolpolyamine. A typical alkaline decontaminating composition of this kind is noted below.

| Composition A: | Percent by weight |
|---|---|
| Caustic soda | 70 |
| Triethanolamine | 18 |
| Sodium gluconate | 12 |
| | 100 |

In order to render the decontaminating agent soluble in the chlorinated hydrocarbon liquid solvent, it is usually necessary to employ a mutual solvent or coupling agent. Exemplary materials of this type include tributyl phosphate, 2,4-pentanedione, n-butanol and isopropyl alcohol. Certain mutual solvent such as tributyl phosphate and 2,4-pentanedione, for example, also functions as decontaminating agents.

A small but sufficient amount of water is also preferably included in the first stage treating solution containing chlorinated hydrocarbon and decontaminating agent, in order to render the solution effective for the dissolution of water-soluble contamination. Such water also functions to render the acid or alkaline decontaminating agent active in the treating solution. The water so employed may be incorporated into the solution by employing an aqueous solution of the acid or alkaline decontaminating agent, for example, when employing phosphoric acid as a decontaminant, such material can be employed as the commercial grade 85% phosphoric acid containing about 15% water. However, the amount of water employed should not be so great as to substantially reduce the amount of decontaminating agent which can be mutually dissolved in or coupled into the chlorinated hydrocarbon solvent, since the chlorinated hydrocarbon solvent and the decontaminant should be in a mutual solution for effective functioning of the treating solution for removal of radioactive contaminants.

The amount of chlorinated hydrocarbon solvent, e.g., trichloroethylene, which can be employed in the first-stage treating solution can range from about 70% to about 98%, the amount of decontaminating agent either acid or basic from about 1% to about 10%, and the amount of mutual solvent from about 1% to about 20%, and the amount of water can range from 0% to about 5%,



the above percentages being by weight. A typical formulation can be composed of the following:

| | Percent by weight |
|---|---|
| Chlorinated hydrocarbon | 84 |
| Decontaminant | 5 |
| Mutual solvent | 10 |
| Water | 1 |
| | 100 |

The chlorinated hydrocarbon liquid solvent can be omitted from the first-stage treating solution, and such solution can consist esentially of a solution of the decontaminating agent, usually an aqueous solution thereof. Thus, for example, the first-stage treating solution can be an aqueous solution of any of the above-noted acid decontaminating agents such as an aqueous solution of oxalic acid. Likewise, the first-stage treating solution can be an aqueous solution of an alkaline decontaminating agent including the above-noted alkaline decontaminants such as for example, an aqueous solution of trisodium phosphate or hexametaphosphate. If desired, the decontaminating compositions containing alkali and an alkanolamine of the above-noted Patents 2,992,995 and 2,992,997 can be employed as first-stage treating compositions. Where an aqueous solution of an acid or alkaline decontaminating agent is employed, the amount of acid or alkaline decontaminant used can range by weight from about 3% to about 50% and the amount of water from about 50% to about 97%.

The treating solution employed in the second stage of the radioactive decontamination process of the invention, and which is preferably maintained cool relative to the hot first-stage solution, can constitute solely a chlorinated hydrocarbon liquid such as those exemplified above, or such second-stage treating solution can be composed of a chlorinated hydrocarbon liquid of the type noted above and including a decontaminating agent of the types described above, and such composition may also include a mutual solvent. However, it is preferred to avoid the presence of water in the second-stage treating or washing solution containing chlorinated hydrocarbon solvent. Thus, for example, the chlorinated hydrocarbon solvent liquid can be kept substantially free of water by the addition of dehydrating agent such as silica gel. Where the second-stage treating solution contains a chlorinated hydrocarbon liquid, a decontaminating agent, and a mutual solvent, the amounts of such components employed can be within the respective ranges noted above for the first-stage treating solution.

As examples of operation of the process, where the first-stage treating solution contains a chlorinated hydrocarbon and decontaminating additive, the wash solution of the second stage may be simply a chlorinated hydrocarbon liquid per se. On the other hand, the first-stage treating solution can be in the form of a water solution of a decontaminating agent, and the second-stage solution can be in the form of a chlorinated hydrocarbon liquid containing decontaminating agent.

For best results in carrying out the invention process, the article or part to be decontaminated is treated or immersed in the first-stage decontaminating solution, preferably maintained at an elevated temperature at or close to the boiling point of the solution. In view of these relatively high temperatures of treatment, it is important that the amount of water present in conjunction with the decontaminating agent, e.g., an acid such as phosphoric, be such that any material corrosion of the surface of the article is avoided.

Following treatment of the article to be decontaminated in the first-stage treating solution for a sufficient period of time, which can range from about ¼ hour to about 3 hours, the part is then treated with the second-stage treating solution containing chlorinated hydrocarbon solvent. The preferred mode of treatment in the second-stage solution is to immerse the article in the second-

stage solution, maintained preferably at temperatures about 20° F. or more below the temperature of the boiling chlorinated hydrocarbon vapors with which the article is finally rinsed. Substantially all of the radioactive contaminants on the article surface are removed in the first-stage treatment, and in the second-stage treatment the immersion of the article in the liquid phase further washes the article and aids in removing residual decontaminating first-stage solution and residual radioactive contaminants from the article. The time period of treatment in the second-stage solution can vary, for example, from as little as one minute to about 15 minutes, depending in large measure on the size of the part being treated.

The article which is cooled in the second-stage treatment is then withdrawn therefrom and placed in a zone in contact with hot chlorinated hydrocarbon vapors produced from boiling chlorinated hydrocarbon solvent. Since such vapors are at a temperature substantially higher, e.g., at least about 20° higher, than the temperature of the article, the chlorinated hydrocarbon vapors condense on the surfaces of the article and drain therefrom, e.g. back into the second-stage solution, to rinse the article. The time period for treatment of the article in the hot chlorinated hydrocarbon vapors can range from about one to about 15 minutes.

An alternative method of operation, according to the invention, is to suspend the articles or parts to be decontaminated in a closed vessel and introducing steam carrying a chlorinated hydrocarbon or a mixture thereof, and decontaminating agent, and which may also include mutual solvens, all as described above, into the enclosed vessel for treatment of the parts suspended therein. For this purpose, wet or superheated steam can be used, and the chlorinated hydrocarbon-decontaminating additive solution incorporated into the steam by injection means. The vapors of chlorinated hydrocarbon, mutual solvent, and water in the steam carrier will condense on the cold surfaces of the articles to be decontaminated and drain down the sides thereof, and the particles of decontaminating agent will settle and dissolve in the aqueous film formed on the surface of the articles, forming an effective decontaminating solution for removal of the contaminants from the parts. If desired, a steam dispersion of only the decontaminating agent can be employed in the first-stage treatment.

When the articles being decontaminted become too warm to further condense the steam decontaminating agent dispersion, the parts may be allowed to cool, or a cooling fog of cold water may be directed upon them. After sufficient cooling, the process may be repeated as many times as necessary. Either before or after the above-noted steam treatment of the parts with a steam dispersion containing decontaminant, a steam dispersion of chlorinated hydrocarbon alone can be employed, either as a pretreatment or as an aftertreatment. If desired, additional treatment of the parts can be carried out employing steam dispersions of different chlorinated hydrocarbon-decontaminating agent compositions. The particular type of treatment or sequence of treating steps with the various steam dispersions of decontaminant, of chlorinated hydrocarbon-decontaminant, or of chlorinated hydrocarbon alone can depend upon the article to be decontaminated, the degree and type of contaminant, and other similar factors.

The above alternative process employing steam dispersions of decontaminating additive and/or of chlorinated hydrocarbon can be employed for radioactive decontamination of the internal walls of closed vessels, tanks or piping, such as used in nuclear reactor systems.

The above processes according to the invention result in the use of relatively small quantities of decontaminating liquids, and permit their further concentration by evaporation or distillation, or the extraction from them of the highly radioactive materials contained therein.



The following are specific examples of practice of the invention.

Specific examples of acid treating solutions containing chlorinated hydrocarbons and decontaminating agents which can be employed according to the invention are as follows.

| Composition B: | Percent by wt. |
|---|---|
| Trichloroethylene | 84 |
| Oxalic acid | 5 |
| n-Butyl alcohol | 10 |
| Water | 1 |
| | 100 |

| Composition C: | |
|---|---|
| Trichloroethylene | 84 |
| Citric acid | 5 |
| Isopropyl alcohol | 10 |
| Water | 1 |
| | 100 |

| Composition D: | |
|---|---|
| Perchloroethylene | 80 |
| Citric acid | 7 |
| Isopropyl alcohol | 11 |
| Water | 2 |
| | 100 |

| Composition E: | |
|---|---|
| Trichloroethylene | 75 |
| Sulfamic acid | 3 |
| n-Butyl alcohol | 20 |
| Water | 2 |
| | 100 |

| Composition F: | |
|---|---|
| Trichloroethylene | 88 |
| Tartaric acid | 4 |
| n-Butyl alcohol | 7 |
| Water | 1 |
| | 100 |

| Composition G: | |
|---|---|
| Trichloroethylene | 85 |
| $NaHSO_4$ | 1 |
| n-Butyl alcohol | 10 |
| Water | 4 |
| | 100 |

| Composition H: | |
|---|---|
| Trichloroethylene | 84 |
| Phosphoric acid, 85% | 6 |
| Tributyl phosphate | 10 |
| | 100 |

Specific examples of alkaline treating solutions containing a chlorinated hydrocarbon and decontaminant which can be employed according to the invention include the following.

| Composition J: | Percent by wt. |
|---|---|
| Trichloroethylene | 84 |
| Trisodium phosphate | 1 |
| Tributyl phosphate | 10 |
| Water | 5 |
| | 100 |

| Composition K: | |
|---|---|
| Tichloroethylene | 80 |
| Sodium hexametaphosphate | 1 |
| Water | 6 |
| Tributyl phosphate | 13 |
| | 100 |

Composition L:

| | |
|---|---|
| Trichloroethylene | 86 |
| Sodium polyphosphate | 1 |
| Ammonium bicarbonate | 1 |
| Tributyl phosphate | 9 |
| Water | 3 |
| | 100 |

Composition M:

| | |
|---|---|
| Trichloroethylene | 82 |
| Composition A above | 7 |
| n-Butyl alcohol | 10 |
| Water | 1 |
| | 100 |

Examples of practice of the invention are as follows:

## EXAMPLE 1

Loop piping components and associated equipment, including valves and pump components, of an experimental nuclear reactor, constructed of 304 stainless steel and contaminated with alpha-beta and gamma radiation, are immersed in a first tank containing Composition B above, including trichloroethylene and oxalic acid as decontaminating agent. Composition B is maintained at or near the boiling point of the composition. The parts are maintained in Composition B for a period of about one hour.

The parts are then withdrawn from the first tank and are transferred into a second adjacent tank containing trichloroethylene solvent alone, and at a temperature of about 20° to 30° F. below the boiling temperature of the trichloroethylene. The articles are maintained in the second tank for a time sufficient to lower the temperature of the articles approximately to that of the liquid trichloroethylene in such tank, i.e., about 10 minutes.

The parts are then removed from the second tank and raised to a vapor zone above the second tank. In the zone above the second tank, the parts are enveloped by hot vapors of trichloroethylene emanating from boiling trichloroethylene in a third adjacent tank. Such vapors condense upon the relatively cool parts and drain off such parts into the second tank, thus rinsing the parts. The parts are maintained in the above-noted vapor zone for a time period of about five minutes, and readily dry in such zone.

The radioactivity of the so treated parts is reduced from a high level prior to treatment of the parts, to a low level of radioactivity, substantially without corrosion of the base metal of the parts thus treated.

## EXAMPLE 2

Components of a nuclear reactor similar to the parts treated in Example 1 and contaminated with radioactive material, are treated according to the same procedure as in Example 1, except that following immersion of the parts in the second tank containing liquid trichloroethylene, and raising of the cooled parts to the zone above the second tank, the parts are contacted by the hot trichloroethylene vapors emanating from the first tank, omitting the third tank of Example 1. Such hot vapors condense on the parts and drain into the second tank.

Following such draining of the parts, the parts are removed from the vapor zone. The radioactive decontamination results are similar to the results obtained in Example 1.

## EXAMPLE 3

The procedure of Example 1 is followed, treating contaminated parts similar to the part treated in Example 1, but employing in place of Composition B as the first-stage treating solution, acid Compositions C to H, respectively, such compositions being maintained at a temperature close to their boiling point during treatment.

Following washing of the parts in the trichloroethylene bath in the second tank and rinsing in the hot trichloroethylene vapors, the parts are practically free of radioactive contamination.

## EXAMPLE 4

The procedure of Example 1 is followed, treating contaminated components similar to the parts treated in Example 1, but employing in place of Composition B as the first-stage treating solution, alkaline Compositions J to M above, respectively, such compositions being maintained at a temperature close to their boiling points during treatment.

Following washing in the second-stage trichloroethylene bath and in the hot trichloroethylene vapors, the parts so treated are substantially reduced in radioactivity as compared to the radioactivity of the parts prior to treatment.

## EXAMPLE 5

The procedure of Example 1 is followed, treating contaminated components similar to the parts treated in Example 1, but employing in place of Composition B as the first-stage treating solution, aqueous acid solutions of 10% oxalic acid and 10% phosphoric acid, respectively, said compositions being maintained at a temperature close to their respective boiling points.

Following washing in the second-stage trichloroethylene bath and rinsing in the hot trichloroethylene vapors, the parts are practically free of radioactive contamination.

## EXAMPLE 6

The procedure of Example 1 is followed, treating contaminated components similar to the parts treated in Example 1, but employing in place of Composition B as the first-stage treating solution, an aqueous acid solution of 15% oxalic acid, said solution being maintained at a temperature close to its boiling point, and employing in place of the trichloroethylene bath in the second-stage treatment the following Composition N.

| Composition N: | Percent by wt. |
|---|---|
| Trichloroethylene | 84 |
| Phospholium (anhydrous phosphoric acid) | 6 |
| Tributyl phosphate | 10 |
| | 100 |

Following washing by treatment of the parts in the liquid phase of Composition N, and rinsing in the hot trichloroethylene vapors, substantially all of the radioactive contaminants initially present on the parts are removed.

## EXAMPLE 7

Composition M is injected by means of a metering pump into superheated steam. The resulting steam dispersion or aerosol of Composition M is then introduced into a vessel of a nuclear reactor system, the interior wall of said vessel containing radioactive materials. Such vessel wall is maintained relatively cool. The vapors of trichloroethylene and n-butyl alcohol cosolvent and water condense on the interior wall of the vessel and drain down the sides to the bottom thereof, and the components of the decontaminating additive, Composition A, on contact with the hot condensate, form a hot aqueous active decontaminating solution effectively removing radioactive contaminants from the vessel wall.

The spent condensate in the vessel can be drained, and after permitting the vessel wall to cool, a steam suspension of trichloroethylene is introduced into the vessel, the water and solvent vapors condensing on contact with the vessel wall, and draining down the wall to the bottom. The spent hot condensate is withdrawn from the vessel. The interior wall of the vessel is substantially free of radioactive contaminants and can be placed back into service.

## EXAMPLE 8

Tools and parts contaminated with radioactive mate-

rials are suspended from the top of a closed chamber. Composition B is injected into saturated steam by means of a metering pump, in a concentration of about 1 ounce of Composition B per gallon of feed water used in producing the steam.

The resulting steam suspension of Composition B is then introduced into the vessel containing the suspended parts to be decontaminated. The steam suspension condenses on the relatively cool surface of the suspended parts forming a hot decontaminating solution which drains down the part surfaces and deposits in the bottom of the tank. Such treatment can be carried out for a period of about ¼ hour to about 3 hours, with periodic cooling of the parts, if desired.

Then trichloroethylene is injected into wet or saturated steam, and the resulting steam-solvent dispersion is introduced into the vessel, a water-solvent mixture condensing on the cooler surfaces of the parts to rinse same free of the previously applied decontaminating Composition B, the rinsing liquid draining down into the bottom of the tank.

If desired, prior to introduction of the steam-trichloroethylene dispersion, a cooling spray or fog of cold water can be introduced into the vessel and into contact with the suspended parts therein, to cool the surfaces thereof, and facilitate condensation of the subsequent steam-trichloroethylene rinsing medium on the part surfaces.

The parts so treated are removed from the vessel, and are substantially free of radioactive contamination.

The vessel in which the parts are treated, as noted above, has an opening in the bottom thereof, and the condensed water and Composition B collecting on the bottom of vessel during the initial treatment of the parts with the steam dispersion of Composition B, is drawn off by suitable valving to a receiver, prior to introduction of the steam-trichloroethylene dispersion. After rinsing of the parts with the latter dispersion, the condensed steam-trichloroethylene mixture at the bottom of the vessel can be drawn off into another receiver.

A series of three receiving tanks is used, one for highly radioactive effluent, another for moderately radioactive effluent, and a third for weakly radioactive effluent. These effluents may be further concentrated by distillation, resulting in the waste disposal of relatively small quantities of radioactive effluent.

The receiving tanks for the above effluent are equipped with means for selectively removing solvent from aqueous layers in which the contaminants are generally concentrated, permitting reuse of the solvent.

## EXAMPLE 9

The procedure of Example 8 is repeated, employing as the initial treating medium a superheated steam dispersion of phosphoric acid, produced by injecting an 85% aqueous phosphoric acid into superheated steam. Following such treatment, a cooling water spray is introduced into the vessel containing the parts to be decontaminated, and then a superheated steam dispersion of Composition C, formed by injecting such composition into the superheated steam, is introduced.

Results similar to those of Example 8 are obtained

From the foregoing, it is seen that the invention provides novel processes and compositions for removal of radioactive contaminants from surfaces, and substantially reducing the waste disposal problem presented by the spent treating liquids containing radioactive materials.

While I have described particular embodiments of my invention for the purpose of illustration, it should be understood that various modifications and adaptations thereof may be made within the spirit of the invention, as set forth in the appended claims.

I claim:

**1.** A process for removal of radioactive contaminants from the surface of an article, which comprises contacting said article with a first hot fluid medium containing a decontaminating agent effective to remove said radioactive contaminants, cooling said article, contacting said article with hot vapors of a chlorinated hydrocarbon solvent at a temperature above the temperature of said cooled article, and causing said vapors to condense on said article and drain from the surface thereof, rinsing said surface.

**2.** A process as defined in claim **1**, wherein said chlorinated hydrocarbon is trichloroethylene.

**3.** A process as defined in claim **1**, wherein said chlorinated hydrocarbon is trichloroethylene, and said decontaminating agent is an acid material selected from the group consisting of oxalic, citric, sulfamic, tartaric, gluconic and phosphoric acids, sodium acid sulfate, monosodium phosphate and monoammonium citrate.

**4.** A process for removal of radioactive contaminants from the surface of an article, which comprises contacting said article with a first hot fluid medium, cooling said article, and then contacting said article with the vapor phase of a second hot fluid medium, said vapor phase having a temperature above the temperature of said cooled article, and causing said vapor phase to condense on said article and to rinse the surface of said article, at least said first fluid medium containing a decontaminating agent effective to remove said radioactive contaminants, and said second fluid medium containing a chlorinated hydrocarbon liquid solvent having a low boiling point in the range between the boiling point of trichloroethylene and perchloroethylene.

**5.** A process for removal of radioactive contaminants from the surface of an article, which comprises immersing said article in the liquid phase of a first fluid medium maintained at a temperature close to its boiling point, said first fluid medium containing an acid to alkaline decontaminating agent and an effective amount of water for the dissolution of radioactive contaminants, immersing said article in the liquid phase of a second fluid medium maintained at a temperature below the temperature of said first fluid medium, said second fluid medium containing a chlorinated hydrocarbon liquid solvent having a low boiling point in the range between the boiling points of trichloroethylene and perchloroethylene, and maintained at a temperature at least about 20° F. below the boiling point thereof, removing said article from said second fluid medium, and contacting said article with hot vapors of a chlorinated hydrocarbon as defined above, at a temperature above the temperature of said article, and causing said hot vapors to condense on said article to rinse the surface of said article.

**6.** A process as defined in claim **5**, wherein said chlorinated hydrocarbon is trichloroethylene.

**7.** A process as defined in claim **5**, wherein said first fluid medium consists essentially of an aqueous solution of a decontaminating agent.

**8.** A process as defined in claim **5**, wherein said first fluid medium consists essentially of an aqueous solution of a decontaminating agent, said second fluid medium contains a decontaminating agent compatible with said chlorinated hydrocarbon liquid in said second medium.

**9.** A process as defined in claim **5**, wherein said first fluid medium also includes a chlorinated hydrocarbon liquid solvent having a low boiling point in the range between the boiling points of trichloroethylene and perchloroethylene, and a mutual solvent for said decontaminating agent and said chlorinated hydrocarbon.

**10.** A process as defined in claim **9**, wherein said chlorinated hydrocarbon is trichloroethylene, said decontaminating agent is an acid material selected from the group consisting of oxalic, citric, sulfamic, tartaric, gluconic and phosphoric acids, sodium acid sulfate, monosodium phosphate and monoammonium citrate, and wherein said mutual solvent is a member chosen from the group consisting of isopropyl alcohol, n-butyl alcohol, tributyl phosphate and 2,4-pentanedione.

11. A process for removal of radioactive contaminants from the surface of an article, which comprises immersing said article in a first liquid containing by weight about 1% to about 10% of a decontaminating agent, about 70% to about 98% trichloroethylene, about 1% to about 20% of a mutual solvent for said trichloroethylene and said decontaminating agent, and a small effective amount of water up to about 5% for the dissolution of radioactive contaminants, said first liquid maintained at a temperature close to the boiling point thereof, immersing said article in a second liquid consisting essentially of trichloroethylene maintained at a temperature at least about 20° F. below the boiling temperature of said trichloroethylene, said article being maintained in said second liquid for a period sufficient to lower its temperature approximately to that of said second liquid, and contacting said article with hot vapors of trichloroethylene at about the boiling point thereof, and causing said vapors to condense on said article and to rinse the surface of said article.

12. A process for removal of radioactive contaminants from the surface of an article, which comprises immersing said article in a first liquid containing an aqueous solution of a decontaminating agent including by weight about 3 to about 50% decontaminating agent and about 50% to about 97% water, said first liquid maintained at a temperature close to the boiling point thereof, immersing said article in a second liquid consisting essentially of trichloroethylene maintained at a temperature at least about 20° F. below the boiling temperature of said trichloroethylene, said article being maintained in said second liquid for a period sufficient to lower its temperature approximately to that of said second liquid, and contacting said article with hot vapors of trichloroethylene at about the boiling point thereof, and causing said vapors to condense on said article and to rinse the surface of said article.

13. A process for removal of radioactive contaminants from the surface of an article, which comprises placing said article in a confined zone, introducing a first steam dispersion of a first substance into said zone, and then introducing a second steam dispersion of a second substance into said zone, while maintaining the surface of said article at a temperature below the temperature of said first and second steam dispersions, at least said first substance including a decontaminating agent effective to remove said radioactive contaminants, and said second substance including a chlorinated hydrocarbon solvent having a low boiling point in the range between the boiling points of trichloroethylene and perchloroethylene.



14. A process as defined in claim 13, wherein said chlorinated hydrocarbon is trichloroethylene.

15. A process as defined in claim 13, including withdrawing a first liquid effluent from said zone, said first effluent containing water and said decontaminating agent, withdrawing a second liquid effluent from said zone, said second effluent containing water and said chlorinated hydrocarbon, concentrating said first effluent and recovering said chlorinated hydrocarbon from said second effluent.

16. A process for removal of radioactive contaminants from the surface of an article, which comprises placing said article in a confined zone, introducing a first steam dispersion of a first substance into said zone, and then introducing a second steam dispersion of a second substance into said zone, while maintaining the surface of said article at a temperature below the temperature of said first and second steam dispersions, said first substance including a decontaminating agent effective to remove said radioactive contaminants, a chlorinated hydrocarbon solvent having a low boiling point in the range between the boiling points of trichloroethylene and perchloroethylene, and a mutual solvent for said decontaminating agent and said chlorinated hydrocarbon, and said second substance including a chlorinated hydrocarbon as defined above.


**References Cited**

UNITED STATES PATENTS

| | | | |
|---|---|---|---|
| 2,023,496 | 12/1935 | Todd | 134—22 |
| 2,101,840 | 12/1937 | Dinley | 134—11 XR |
| 2,348,465 | 5/1944 | Geiringer | 134—31 XR |
| 2,778,792 | 1/1957 | Szilard | 134—22 XR |
| 2,852,419 | 9/1958 | Overholt et al. | 134—27 XR |
| 2,915,387 | 12/1959 | Kolodney | 134—41 XR |
| 2,924,576 | 2/1960 | Bersworth et al. | |
| 2,981,643 | 4/1961 | Baybarz. | |
| 2,982,702 | 5/1961 | Wehrmann | 134—41 XR |
| 3,013,909 | 12/1961 | Pancer et al. | 134—27 XR |
| 3,046,163 | 7/1962 | Kearney et al. | 134—11 |
| 3,047,434 | 7/1962 | Bulat | 134—1 |
| 3,080,262 | 3/1963 | Newman | 134—3 |
| 3,084,076 | 4/1963 | Loucks et al. | 134—22 |



MORRIS O. WOLK, *Primary Examiner.*

J. ZATARGA, *Assistant Examiner.*

U.S. Cl. X.R.

134—2, 3, 11, 22, 30, 31