# United States Patent

[11] **3,606,996**


[72] Inventor **James M. Wolf**
**Ann Arbor, Mich.**



[21] Appl. No. **821,276**
[22] Filed **May 2, 1969**
[45] Patented **Sept. 21, 1971**
[73] Assignee **Chemotronics, Inc.**
**Ann Arbor, Mich.**


[54] **METHOD FOR STERILIZATION**
**11 Claims, 4 Drawing Figs.**


[52] **U.S. Cl.** ..... **21/2,** 21/82, 21/91
[51] **Int. Cl.** ..... **A61l 1/00**
[50] **Field of Search** ..... 21/1, 2, 60, 82–83, 85, 91, 92, 93



[56] **References Cited**

UNITED STATES PATENTS

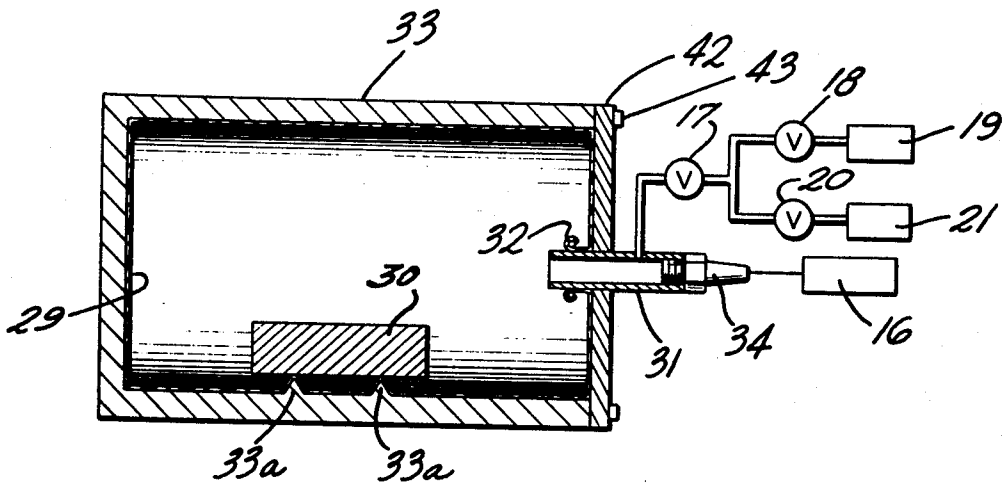
| | | | |
|---|---|---|---|
| 905,315 | 12/1908 | Hodgson | 21/2 |
| 1,343,108 | 6/1920 | Buchanan | 21/82 |
| 1,902,625 | 3/1933 | Dunham | 21/56 |
| 3,084,793 | 4/1963 | Pitman | 206/632 |
| 3,099,522 | 7/1963 | Lauterbach | 21/56 X |
| 3,175,025 | 3/1965 | Geen et al | 264/80 |
| 3,278,256 | 10/1966 | Skaller | 21/85 X |
| 3,329,759 | 7/1967 | Rice | 264/321 |
| 3,383,163 | 5/1968 | Menashi | 21/54 |
| 3,481,692 | 12/1969 | Linder | 21/56 X |



*Primary Examiner*—Morris O. Wolk
*Assistant Examiner*—Joseph T. Zatarga
*Attorneys*—Miller, Morriss, Pappas & McLeod and Ian C. McLeod



**ABSTRACT:** A method for the sterilization utilizing essentially instantaneous and transient thermal pulses generated in a gaseous medium in contact with the surfaces. The thermal pulses kill the micro-organisms and have a duration of less than about 1 second so that the surface is not damaged. The surface to be treated is substantially free of liquid material so that the thermal pulse acts directly upon the micro-organisms. The sterilization is particularly useful for medical and industrial purposes.

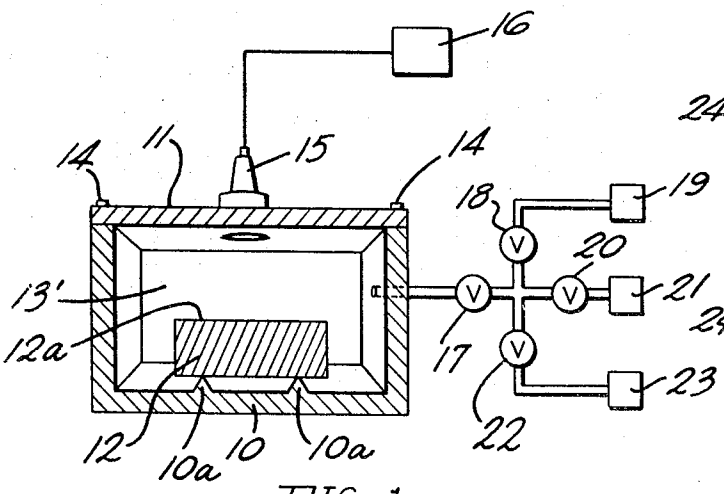



FIG. 1

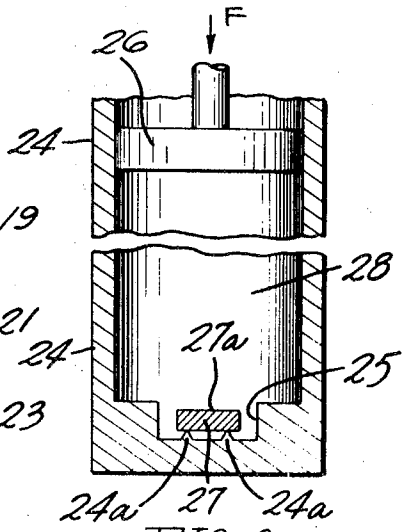



FIG. 2

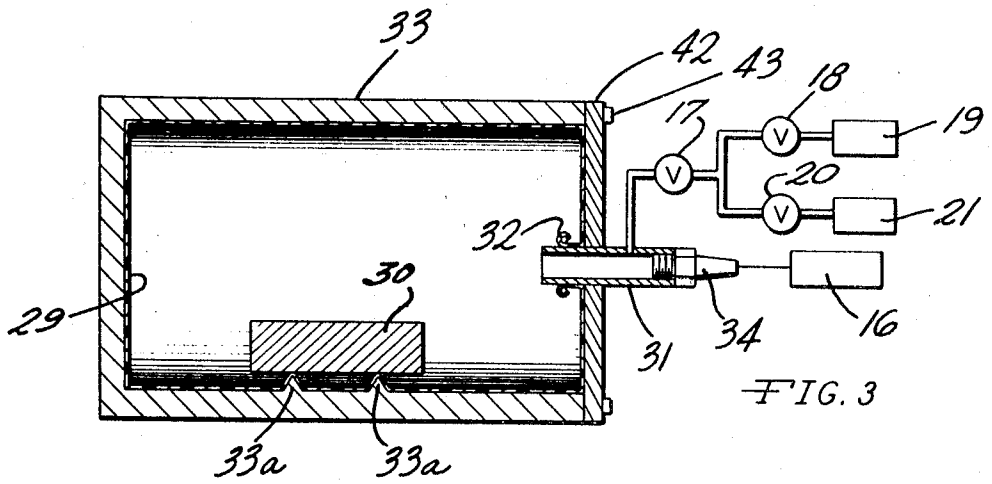



FIG. 3

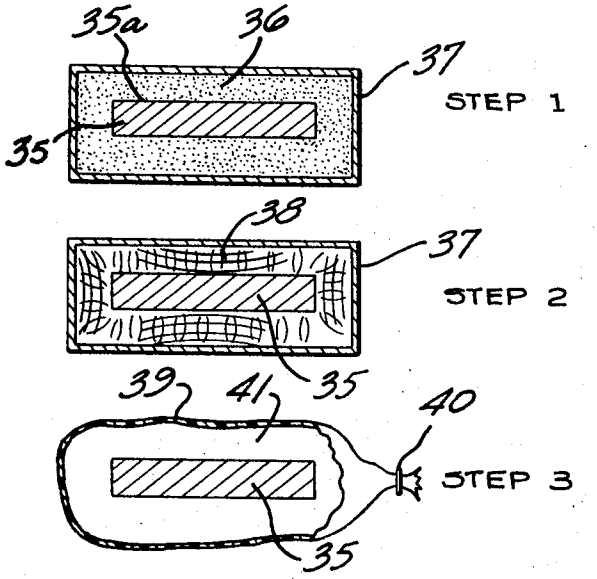



FIG. 4

INVENTOR
JAMES M. WOLF
BY
Miller Morrison Pappas & Mcleod
ATTORNEYS

# METHOD FOR STERILIZATION

## BACKGROUND OF THE INVENTION

The present invention is concerned with methods for sterilization wherein heat in the form of transient thermal pulses generated in a gaseous medium in contact with the surface to be sterilized is utilized. More particularly the present invention utilizes thermal pulses generated in the gaseous medium for less than about 1 second which are produced by the ignition of a combustible composition as the gaseous medium or by the adiabatic compression and decompression of the gaseous medium.

The prior art methods of sterilization having a direct relation to the present invention particularly rely upon heating in some form for relatively long periods of time to kill micro-organisms. See for instance Kirk–Othmer, VOL. 13, (1968) beginning at pg, 457 wherein various commercial sterilization methods are discussed. In general temperatures in the range of 100° to 200° C. are used for periods of time ranging from several minutes to hours. Also known is direct flame heating of the surface to be sterilized wherein the bulk of the article is heated as well to temperatures approaching the flame temperature (about 2,600° C. maximum); however, this technique can produce damage to the bulk of the article and cannot be used with flammable materials. Also the use of a direct flame produces nonuniform heating of the article.

The generation of thermal pulses in a gaseous medium is known to the prior art as particularly shown by U.S. Pat. No. 3,175,025 and 3,329,759; however, it was not recognized that articles could be sterilized as a result of treatment and thus they were not handled so as to prevent micro-organism contamination, particularly contamination by pathogenic micro-organisms.

## SUMMARY

It is therefore an object of the present invention to provide a method whereby sterilization can be achieved utilizing transient thermal pulses which uniformly heat the surfaces of the article. It is further an object of the present invention to provide a method wherein the article is undamaged by the thermal pulse because of the transient nature of the heating. These and other objects will become increasingly apparent from the following description.

The objects of the present invention are provided by the method for the treatment of surfaces requiring sterilization in order to kill micro-organisms on the surfaces which comprises: (a) contacting the surfaces to be sterilized with a gaseous medium, the surfaces in contact with the gaseous medium being substantially free of liquid material; (b) transiently exposing the surfaces to a thermal pulse generated in the gaseous medium having peak temperatures substantially above the minimum temperatures required to kill the micro-organisms without damage to the surface; and (c) handling the surfaces so as to at least prevent pathogenic micro-organism contamination before subsequent use. The use of combustible gaseous material as the gaseous medium which is ignited to produce the thermal pulse is particularly preferred.

## DESCRIPTION OF DRAWING

FIGS. 1 to 3 schematically illustrate various forms of equipment which can be used in the process of the present invention. FIGS. 1 and 3 illustrate preferred forms wherein a combustible material is provided in a confined space and ignited to generate the thermal pulse. FIG. 2 illustrates a piston and cylinder arrangement for adiabatically compressing and decompressing a gaseous medium to generate the thermal pulse.

FIG. 4 schematically illustrates the steps of the method of the present invention including provision of the gaseous medium, generation of the thermal pulse and one form of handling to prevent micro-organism contamination using a sealed bag.



## DESCRIPTION OF THE PREFERRED EMBODIMENTS

The equipment which can be utilized in the process of the present invention is generally known to the prior art as shown by the above cited U.S. Pat. Nos. 3,175,025 and 3,329,759. The gaseous medium is provided in contact with the surface to be treated in a confined space or chamber along with a means for generating the thermal pulse in the gaseous medium, such as by the use of a combustible gaseous material and ignition or by the use of substantially adiabatic compression and decompression of the gaseous medium. The following is a brief description of preferred specific forms of such equipment.

Referring to the drawing and FIG. 1, a confined space **13** is defined by chamber **10** and cover **11** secured for instance by bolts **14**. Within the confined space **13** mounted on projections **10***a* is provided the article **12** to be sterilized. An ignition device **15** is provided within the confined space **13** which is actuated by a spark generator means **16**. A conduit and valve **18** leads into the confined space **13** which is connected to vacuum means **19** with valve **18** and provided to evacuate the confined space **13** of air. Gaseous fuel supply means **21** is provided with valve **20** to supply fuel to the confined space **13** after evacuation for ignition by the ignition means **15** and preferably and optionally a blanket gas supply means **23** with valve **22** to provide a gaseous material in the confined space **13** after ignition of the gaseous fuel to aid in the prevention of contamination after sterilization when the cover **11** is removed.

Referring to FIG. 2, a cylinder **24** is provided with a recess **25** for holding the article **27** to be sterilized and is mounted on projections **24***a*. A piston **26** is fitted to the cylinder **24** and a gaseous medium is provided within the defined confined space **28**. The gas is compressed substantially adiabatically by applying a force F to the piston **26**, thus heating the gaseous medium and the surface **27***a* of the article **27** for sterilization and then immediately decompressed to prevent overheating of the article.

Referring to FIG. 3, an article **30** to be sterilized is provided within a flexible bag **29**, such as polyethylene bag, which is fitted with or sealed to an ignition tube **31** at the open end of the bag **29** such as by a band **32**. The end of the tube **31** outside of the bag **29** is fitted with an ignition device **34**. At least the bag **29** with the article **30** can be provided in a clean chamber **33** or preferably the chamber **33** fits the confines of the bag **29** such that it is not ruptured by the ignition of the combustible gaseous material. A cover **42** with bolts **43** provides closure of the chamber **33**. The ignition device **34** is provided with the spark actuator means **16** and conduit and valve **17** is attached to the exposed end of the tube **31** with the vacuum means **19** and valve **18** and gaseous fuel supply means **21** and valve **20** and operated as is the device of FIG. **1**. The blanket of gaseous material after sterilization is unnecessary, since the clean chamber **33** or bag **29** is utilized and this is also possible with the devices of FIGS. **1** and **2**.

Regardless of the equipment used, the method of the present invention involves three basic steps as illustrated in FIG. **4** in Steps **1**, **2** and **3**. In Step **1**, the article **35** is provided in the confined space **37** with the gaseous medium **36** in contact with the surfaces **35***a* of the article **35** to be sterilized. The surfaces **35***a* must be substantially free from liquid material whether from the gaseous medium **36** or originally present on the article **35**. If necessary the article **35** is dried to remove liquid material. The gaseous medium **36** is preferably dry. In Step **2**, the gaseous medium is either compressed and decompressed or ignited to create a thermal pulse **38** having peak temperatures substantially above the minimum temperatures required to kill the micro-organisms and without damage to the article **35**. In Step **3**, the surfaces **35***a* of the article **35** are handled so as to at least prevent contamination by pathogenic micro-organisms before subsequent use. Preferably the article **35** is aseptically handled to prevent any micro-organism contamination. Thus the article **35** can be transferred from the

confined space **37** and placed in a bag **39**, such as a polyethylene bag, sealed such as by a clip **40** at the open end and containing a sterile atmosphere **39**. Where the device shown in FIG. **3** is utilized such that the chamber **3** prevents rupture of the bag **29** during ignition of the combustible gaseous material by confining the bag **29**, this has been found to be a simple and inexpensive means of preventing contamination before use and is preferred. The ignition products of the combustible gaseous material are sterile and thus do not need to be replaced with a blanket gaseous material although it is preferred to use the blanket gaseous material in instances where the ignition products could be damaging to the article in order to prevent chemical contamination of or reaction with the article treated.

The article to be sterilized must be bathed by the thermal pulse on all surfaces to be sterilized. This is accomplished by mounting the article on support means in the confined space which allow the gaseous medium to contact the surfaces to be treated, such as the optional projections **10***a*, **24***a* and **33***a* shown in FIGS. **1** to **3** which provide minimum contact with the surfaces to be sterilized. Alternatively the article can be repetitively treated with repositioning of the surfaces of the article to insure treatment with the thermal pulse.

The gaseous medium utilized will depend upon the equipment used and the composition of the article being sterilized. Where adiabatic compression equipment is used it is preferred to use a gaseous medium which is compatible with the article being treated. Thus rare gases or hydrogen or nitrogen have been used with good result. Where the composition of the article is resistant to destructive oxidation, such as stainless steel, oxygen can be used which further augments the treatment with the thermal pulse because of oxidative destruction of the micro-organisms.

Where ignition of a combustible gaseous material is used, it is preferred to use a mixture of an oxidizable gaseous material and an oxidizer gaseous material which is compatible with the material being treated such that the byproducts of combustion do not contaminate or react with the article or otherwise adversely affect the article. For this reason methane (or other lower alkanes, alkenes and alkynes containing 1 to 4 carbon atoms) and hydrogen are the preferred oxidizable materials and air or oxygen are the preferred oxidizer materials since water and/or carbon dioxide is generated by the reaction. Oxidizing conditions, as discussed above, can be produced by providing a stoichiometric excess of the oxidizer, oxygen. Other oxidizable materials which can be used are for instance ammonia, hydrazine, hydrogen sulfide, carbon monoxide and various hydrocarbons such as acetylene, ethylene oxide and cyanogen. Suitable oxidizer materials are pure oxygen which is preferred, oxygen with gaseous diluents such as air, air alone, halogens such as fluorine and chlorine, ozone and various nitrogen oxides. Mixtures of single or multiple oxidizer materials with single or multiple oxidizable materials can be used, but these are not preferred because of the possible effect on the article of the byproducts of the reaction. The energy produced by the combustible gaseous material can be increased or decreased by regulating the charge pressure prior to ignition. Various compounds which rapidly decompose with accompanying transient release of thermal energy upon suitable initiation can also be used singly or multiply to produce the transient elevated gaseous temperatures such as acetylene and nitrogen dioxide.

In each instance the thermal pulse generated is transient, and usually this step is complete in less than **1** second. The treatment does not damage or destroy the article because the surfaces in contact with the gaseous medium are only briefly in contact with the thermal pulse while the bulk of the article is only heated to a limited degree. Also, the total energy of the thermal pulse is maintained at less then the energy which would damage or destroy the article. For this reason articles composed of metals, ceramics, plastics and naturally occuring materials can be sterilized. Woven and nonwoven fabrics, such as blankets and bandages and the like, stainless steel medical instruments and other articles with surfaces requiring sterilization have been successfully treated.

Having generally described the method of the present invention, the following are specific examples.

## EXAMPLE 1

Utilizing the equipment illustrated in FIG. **1**, a roll of square woven fabric (cotton and polyester fiber) 6 inches long and 4 inches in diameter was provided in the chamber **10** measuring 4½ in diameter by 6¼ inches high. The chamber **10** was closed with the cover **11** and bolted with bolts **14**. The chamber was evacuated to 10 of Hg and then a combustible mixture of 60 percent hydrogen and 40 percent oxygen by volume (molar excess of oxygen) at three atmospheres absolute was provided in the confined space and ignited. A blanket of nitrogen was then provided in the confined space and then the cover was removed and the article transferred to a sterile bag for subsequent use. It was found upon culture testing of a portion of the article that it was sterile and that the article was undamaged by the treatment.

## EXAMPLE 2

The treatment of example 1 was repeated with the same size roll of cotton muslin using a mixture of 66⅔ percent hydrogen and 33⅓ percent oxygen by volume (stoichiometric mixture) at two atmospheres absolute. The results were the same as those of example 1.

## EXAMPLE 3

The treatment of example 1 was repeated with a polypropylene perforated tube measuring 6 inches long by 1⅜ inches in diameter used as a support for microfiltration using a mixture of 75 percent hydrogen and 25 percent oxygen by volume at 4 atmospheres absolute. The results were the same as those of example 1.

## EXAMPLE 4

The procedure of example 1 was repeated using a 2 diameter by 2½ inches high chamber with a stainless steel (300 series) surgical pin measuring 2 inches long by ½ inch in diameter using a mixture of 52 percent hydrogen and 48 percent oxygen (stoichiometric excess of oxygen) by volume at 500 p.s.i.g. The results were equivalent to those of example 1

## EXAMPLE 5

The procedure of example 4 was repeated with a gold alloy dental crown using a mixture of 75 percent hydrogen and 25 percent oxygen by volume (stoichiometric excess of hydrogen) at 500 p.s.i.g. The results were equivalent to those of example 1.

## EXAMPLE 6

The procedure of example 1 was repeated with a nonwoven fabric of 3 denier polypropylene fiber measuring 6 inches long by 4 inches in diameter using a mixture of 66⅔ percent hydrogen and 33⅓ percent oxygen by volume (stoichiometric) at two atmospheres absolute. The results were equivalent to those of example 1.

## EXAMPLE 7

Using the equipment illustrated in FIG. **2**, wherein the confined space **28** measured 18 inches long and 2 inches in diameter, portions of the articles of examples 1 to 6 were provided in the recess **25** in an atmosphere of nitrogen as a neutral atmosphere, hydrogen as a reducing atmosphere or in certain instances oxygen as an oxidizing atmosphere and the gas rapidly compressed and decompressed along the 18 inch length of the chamber by the piston **26** such that there was a gas compression ratio of about 40 to 1. The results were similar to those of examples 1 to 6.

EXAMPLE 8

Using the equipment of FIG. 3, a large steel enclosure was provided measuring 3 feet in diameter by 6 feet long. A large roll of cotton gauze was provided in a polyethylene bag 29 and the enclosure 33 filled with the bag and roll. The treatment of example 2 was repeated with equivalent results.

EXAMPLE 9

To test the effectiveness of sterilization a polyethylene film surface was smeared with the pathogenic bacteria Escherichia Coli and subjected to the treatment of example 2. It was found the E. Coli was killed.

EXAMPLE 10

To test the effectiveness of sterilization, the bacteria Bacillus Stearothermophilus was smeared on a steel surface and subjected to the treatment of example 4. This bacteria is utilized by those skilled in the sterilization art to determine the efficacy of sterilization and normally requires steam at 250° F. for 15 minutes to kill it. Culturing at above about 55° C. is required for growth. It was found that the bacteria was killed.

In the manner of examples 1 to 10, micro-organisms including virus, bacteria, mold and yeast were treated by the method of the present invention and killed. It was found that the thermal pulse preferentially heated the micro-organisms on the surfaces to the destruction point because of their very small size and thin cross section so long as there was no masking effect by liquids or solids on the surfaces to be sterilized.

In the handling of the article after sterilization, any one of various known techniques can be utilized. The article can be kept in the sterilizing unit before use or be placed in a sterile container. In any event, care must be taken to prevent contamination by at least pathogenic micro-organisms before use and possibly even during use for some applications.

It is intended that the foregoing description be only illustrative of the present invention and it is intended that the present invention be limited only by the hereinafter appended claims.

I claim:

1. The method for the treatment of surfaces requiring sterilization in order to destroy micro-organisms on the surface which comprises:

a. providing a surface to be sterilized inside a bag in a sealed and confined space defined by a rigid chamber confining the bag to prevent rupture;

b. contacting the surfaces to be sterilized inside the bag with a gaseous medium, the surfaces in contact with the gaseous medium being substantially free of liquid material which masks the surfaces from sterilization;

c. transiently exposing the surfaces to a thermal pulse produced by the ignition of a combustible mixture of gaseous oxidizer and oxidizable materials said pulse being generated in the gaseous medium in the bag which is confined by the rigid chamber and having peak temperatures substantially above the minimum temperatures required to destroy the micro-organisms without damage to the surfaces; and

d. removing the bag from the rigid chamber such that it is sealed so as to at least prevent pathogenic micro-organism contamination of the surface before subsequent use.

2. The method of claim 1 wherein the surfaces are in the form of a fabric which is sterilized in the sealed and confined space.

3. The method of claim 1 wherein the surfaces are in the form of an instrument which is sterilized in the sealed and confined space.

4. The method of claim 1 wherein oxidizing conditions are maintained in the gaseous medium during generation of the thermal pulse.

5. The method of claim 1 wherein the surface to be treated is dried to remove liquid material before being contacted with the gaseous medium and wherein the gaseous medium is substantially dry.

6. The method of claim 1 wherein the surface is aseptically handled prior to use to prevent micro-organism contamination.

7. The method of claim 1 wherein the gaseous medium consists of the mixture of gaseous oxidizer and oxidizable materials

8. The method of claim 7 wherein the combustible gaseous material is a mixture of hydrogen and air.

9. The method of claim 7 wherein the combustible gaseous material is a mixture of hydrogen and oxygen.

10. The method of claim 7 wherein the combustible gaseous material is a mixture of methane and air.

11. The method of claim 7 wherein the combustible material is a mixture of methane and oxygen.