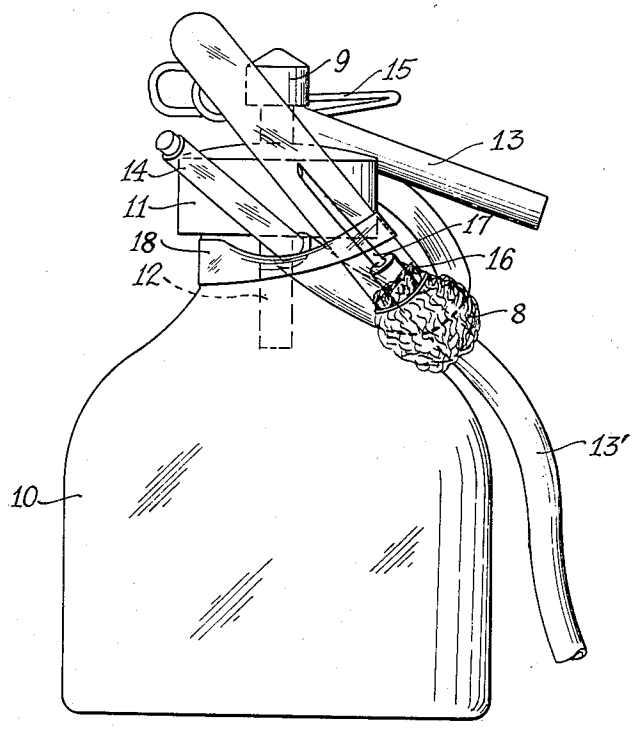
Aug. 12, 1952 J. C. DAVIS ET AL 2,606,654

PACKAGE COMBINED WITH STERILIZATION INDICATOR

Filed April 19, 1945



INVENTORS
Joseph C. Davis
Ralph T. K. Cornwell and
Edwin L. Hansen
BY Worth Wade
Attorney

Patented Aug. 12, 1952

2,606,654

UNITED STATES PATENT OFFICE

2,606,654

PACKAGE COMBINED WITH STERILIZATION INDICATOR

Joseph C. Davis, Scarsdale, N. Y., and Ralph T. K. Cornwell and Edwin L. Hansen, Fredericksburg, Va., assignors, by mesne assignments, to American Viscose Corporation, Wilmington, Del., a corporation of Delaware

Application April 19, 1945, Serial No. 589,162

2 Claims. (Cl. 206—47)

1

This invention relates in general to sterilization and, in particular, to a sterilization indicator.

In the preparation of articles for medical and surgical use, it is generaly necessary to insure their complete sterilization. The most convenient and, therefore, the most commonly used method of sterilizing is the application of heat, usually above the boiling point of water. In the manufacturing processes, such as the original manufacture of surgical instruments, it is generally a simple and routine matter to sterilize the products after their manufacture. However, when these instruments are to be used, for example, in a doctor's office or in a patient's bedroom, and must be sterilized before use, an entirely different problem is presented. In this case there is no convenient routine opportunity for sterilization, and there are many chances for confusing sterilized with unsterilized instruments. Accordingly, it is highly desirable to supply some automatic means to indicate clearly whether an article is actually sterile.

Blood donor bottles, which are used repeatedly and which must be sterilized between donations, are a typical illustration of the problem. Sterilization is generally accomplished by heating the blood donor bottle with needle and connective tubing attached to 125° C. for about one-half hour. The needle itself is protected, for example, by means of a glass test tube, so that the once sterilized needle is not subsequently contaminated. Until the time of this invention, there has generally been no uniform method for determining whether or not a particular unit has been sterilized. Keeping the sterile and the unsterile units separate has usually been left entirely to the person operating the sterilizing apparatus. In such an arrangement, of course, in spite of the utmost care, there will occasionally be errors, and sometimes a nonsterile unit will become mixed with other sterile units with possibly serious consequences.

It is an object of the present invention to provide a sterilization-indicating pellicle adapted for use on articles, such as medical and surgical supplies and instruments of all types, for holding a plurality of such articles together and indicating their condition with respect to sterility.

According to the present invention, a plurality of articles to be used together and requiring sterilization by heat treatment before such use are held together as a unitary package assembly by a sterilization indicator in the form of a band of a non-fibrous hydrophilic pellicle made of a film-

2

forming material which is substantially colorless and suffused with a heat-convertible dyestuff material, that is, an organic substance which undergoes a color change upon application of heat. It will be understood that the pellicle may be treated and thereby combined or suffused with the heat-convertible dyestuff in various ways, especially by impregnating or by coating the pellicle with a composition comprising the dyestuff material. More particularly, the band may be made by treating a hydrophilic pellicle with a solution of the heat-convertible dyestuff material evaporating the solvent therefrom at a temperature below that temperature at which the dyestuff changes color, and applying the pellicle to an article to be sterilized, whereupon during sterilization by heat, the dyestuff in the pellicle is converted into a different colored material, thus indicating that sterilization has been effected.

For a more complete understanding of the nature and objects of the present invention, reference should be had to the accompanying drawing in which the figure is a perspective view of one embodiment of the article in the form of a band applied to a blood donor bottle.

The hydrophilic pellicle used in the invention may be formed from any water-swelling film-forming material such, for example, as regenerated cellulose which may be regenerated from viscose, cuprammonium solutions of cellulose, other inorganic solvent solutions of cellulose, solutions of cellulose in organic solvents, such as quarternary ammonium compounds and the like, or formed by deesterifying cellulose esters, or deetherifying cellulose ethers; also hydrophilic cellulose derivatives, such as cellulose esters, cellulose ethers, cellulose ether-esters, cellulose ether-xanthates, and the like.

The pellicle has the form of a seamless shrinkable band which serves as the sterilization indicator and also binds the various articles together during sterilization. When labeled containers are subjected to sterilization, the label frequently becomes detached. To prevent this and also indicate that the article is sterilized, the present invention contemplates that the label will be covered with a transparent shrinkable band which will tightly bind the label to the container during sterilization. In this embodiment, either the label or the transparent band may carry the heat-convertible dyestuff.

The expression "heat-convertible dyestuff" is intended to include all organic substances which undergo a chemical change resulting in a change in color upon being subjected to a temperature

above room temperature, particularly at a temperature above 100° C. The color change may be (*a*) from one color to another color, or (*b*) from colored to colorless, or (*c*) from colorless to colored. Dyes or dye intermediates or derivatives are preferred. "Fugitive" dyes, which are color unstable, and either change to another color or become colorless, are satisfactory. Of these, dyes which are color unstable in the presence of steam are particularly desirable. Also, many dye intermediates, particularly the leuco bases, which are colorless or colored and are transformed into the corresponding dyestuff by heat, have been found operable. Many dyestuffs may be treated with zinc to give colored or colorless compounds. These derivatives may be transformed to the original dye by heat. These also may be used. It will be appreciated that where color changes are more effectively accomplished in the presence of an oxidation catalyst, such as sodium or potassium dichromate, or an oxidation inhibitor, such ingredients may be included with the heat-convertible dyestuff in preparation of the indicator. Typical operative heat-convertible dyestuffs are listed in the following table. The color index number listed refers to the description of the dye in "Society of Dyers and Colourists: Colour Index," edited by F. M. Rowe, published by the Society in 1924, at Bradford, England.

Dyestuff	Color Change	Color Index No.
Pyrazole Fast Green CLL	Green to colorless	(1)
Calcocid Bordeaux BXL, 5 parts	Brown to green	88
Calcocid Yellow MCG, 1 part		640
Calcocid Green CGEx, 1 part		1078
Sodium Bicarbonate, 93 parts		
Auramine Conc	Yellow to colorless	655
Thioflavine TCN Conc	do	815
Victoria Pure Blue BO	Blue to colorless	729
Methyl Violet Conc	Blue-purple to colorless	680
Amphinaphthoquinone	Red to grey	
Methyl Green	Green to violet	684
Iodine Green	do	686
Leuco base of Thionine Blue and sodium potassium chromate.	Colorless to blue	926
Leuco base of Toluidine Blue and sodium potassium chromate.	do	925
Chrysoidine	Brown to colorless	20
Naphthyl Blue-black N	do	310
Pontacyl Sulphon Blue 5R Conc	Blue to brown	289

[1] A green dye derived from Pyrazole manufactured by Sandoz Chemical Company

The dyestuff may be applied to or incorporated in the pellicle in any suitable manner, having regard for the nature of the dyestuff. When the dyestuff is soluble in water or aqueous solutions, the pellicle may be dipped in the aqueous solution containing the dyestuff, or the solution may be sprayed upon the pellicle or printed and shown in predetermined areas. The treated pellicle is then subjected to a drying operation, preferably at room temperature, or in any case, below the conversion temperature of the particular dyestuff employed.

When the dyestuff is insoluble in aqueous media, it may be dissolved in an organic solvent and the pellicle treated with such solution. In general, hydrophilic materials will absorb dyestuffs from such organic solutions, but if the absorption is not sufficient, it may be increased by introducing in the organic solution of the dye a swelling agent for the particular hydrophilic material employed, such as water, alcohol, the lower aliphatic esters, and the like.

Alternatively, the dyestuff may be applied to the pellicle in combination with a film-forming binder. With water-soluble dyes, the binder may be one of the hydrophilic materials above named with reference to the pellicle, and with organic dyes, the binder may be a lacquer base such, for example, as a synthetic resin or an organic solvent-soluble cellulose derivative. In this embodiment the dyestuff and the lacquer base are dissolved in suitable volatile organic solvents to form a lacquer. The hydrophilic pellicle is then coated with the lacquer and the solvent evaporated at a temperature below the conversion temperature of the particular dyestuff. In the absence of pigments, such coatings will normally be transparent. This method is advantageously used when the dyestuff will not dye the pellicle.

By way of illustrating but not by way of limiting the invention, there will be given the following examples:

Example I

Referring to the figure, there is shown a blood donor unit comprising a glass container **10** having a screw cap **11** through which passes a glass tube **12** closed by a temporary cap **9** and a rubber tube **13** terminating in a glass connecting tube **14** and provided with a clamp **15**. The unit also comprises a glass tube **16** containing the injection needle **17** connected to another section of rubber tubing **13'** and closed temporarily with a wad **8** of cotton. During sterilization, it is desirable to hold these miscellaneous items together as a unit. This is accomplished by encircling the neck of the bottle and the various loose articles with a common shrinkable band **18** formed of regenerated cellulose and containing as the heat-convertible dyestuff, for example, Pyrazole Fast Green CLL.

The band is prepared by running regenerated cellulose sheets through a water solution of the green dye of sufficient concentration to give a dark green color to the sheet when dry. The sheet is then dried at room temperature and cut into bands, which may be shipped and stored dry until used. The dry band is moistened and applied to the bottle and articles while wet. It is allowed to dry at room temperature, whereupon it shrinks and binds the various articles tightly to the bottle.

Example II

A continuous tubing formed of regenerated cellulose in the original wet state is passed into a bath containing 30 per cent aqueous solution of sorbitol. The run of tubing through the bath is sufficiently long to permit the sorbitol solution to saturate the wall of the tubing. From the plasticizing bath, the tubing is passed to a drier in

which it is dried until at least the outer surface is free from liquid moisture. The tubing is then passed through a coating bath of the following composition: Urea-aldehyde resin in the solvent-soluble stage—75 parts; alkyd resin—20 parts; ammonium thiocyanate—.05 part; Pontacyl Sulphon Blue 5R conc. (Color Index No. 289)—sufficient amount to give a definite blue tint to the regenerated cellulose; solvent comprising 90 per cent toluene, 5 per cent butyl alcohol, and 5 per cent methyl cellosolve—sufficient quantities to produce the desired consistency of solution.

From the coating bath, the tubing is passed through a suitable heating chamber where it is quickly heated to from 90° C. to 95° C. for one to two minutes. During this heating, the solvents are evaporated and the urea-aldehyde resin polymerized to a hard form. The coated tubing is then passed to a printing press where it is provided with any desired legend or design. From the printing press, the tubing is passed into a chopper where it is severed into bands. The dry bands are suitable for storage or shipment as such.

The band is conditioned by soaking in water for a period of approximately one hour and then slipped over the neck of a bottle, such as a blood donor unit as described in Example I and allowed to dry. Upon sterilization of the article, the coated band changes in color from blue to brown. Thus, it is easily ascertained which units have been sterilized and which have not.

It will be apparent from the above discussion that this invention has numerous advantages. Chief among these is the prevention of errors during sterilization which would result in the use of unsterilized instruments or apparatus. Also of importance, however, are its low cost and its ease of preparation. It is thus adapted to manufacture by machine operation in quantity. Because of its simplicity, it is completely reliable and is not subject, for example, to breakage, spoiling, mechanical failure, etc.

Since many changes may be made in the process and many different embodiments of the indicator are possible following the principles of the invention, the invention is not to be limited except as indicated by the appended claims.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A package assembly comprising a plurality of articles to be made sterile before use by heat treatment and a shrunk band extending around the articles and holding them together tightly, said band being made of a film-forming material which is substantially colorless and being suffused with a heat-convertible dyestuff which undergoes a change in color at a temperature between 100° C. and 160° C.

2. An assembly as defined in claim 1 in which the film-forming material is regenerated cellulose.

JOSEPH C. DAVIS.
RALPH T. K. CORNWELL.
EDWIN L. HANSEN.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,844,199	Bicknell et al.	Feb. 9, 1932
1,894,015	Bernstein	Jan. 10, 1933
2,049,867	Richards	Aug. 4, 1936
2,118,144	Berman et al.	May 24, 1938
2,222,067	Chaney et al.	Nov. 19, 1940
2,308,087	Lappala	Jan. 12, 1943