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(72) Inventor: **DuBay, David K.**  
**Casper, Wyoming 82604 (US)**

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(74) Representative: **Crisp, David Norman et al**  
**D. YOUNG & CO.**  
**21 New Fetter Lane**  
**London EC4A 1DA (GB)**

(71) Applicant: **Defense Technology Corporation of**  
**America**  
**Casper, Wyoming 82604 (US)**

(54) **Capsaicinoid lachrymator**

(57) The present invention provides a capsaicinoid containing lachrymator. The lachrymator comprises a nonflammable carrier and capsaicinoids. The nonflammable carrier comprises propylene glycol, ethyl alcohol and water.

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## Description

### Field of the Invention

The present invention relates to a liquid composition useful as a lachrymator. More particularly, the invention concerns a lachrymator comprising capsaicinoids and a nonflammable water-based carrier.

### Background of the Invention

The prior art provides various compositions which constitute strong irritants to the eyes, mouth and nose of a human and other animals. Such compositions are generally classified as lachrymators. Lachrymators are better known, and commonly referred to as "tear gas" or "riot gas." Lachrymators serve a significant role in society because they allow the police, military and other authorities to control unruly or disruptive persons, including persons under the influence of alcohol or other mind-altering drugs, without having to resort to physical means that may inflict long-term or permanent bodily harm or damage.

Upon application of a lachrymator, the recipient of the lachrymator is overcome by eye, nose and mouth irritation and rendered harmless. More particularly, upon application, the recipient is temporarily disabled with intense burning eye pain, blepharospasm, acute bronchitis and respiratory irritation. The prior art provides various means for dispensing or applying lachrymators, such as, for example, pressurized canisters, hand grenades, and munition cartridges.

The need for an aerosol irritant-type spray or lachrymator, for enforcement and defensive purposes, is historic as well as present day evident. Social unrest, demonstrations and rioting during the 1960's caused the extensive use of military type tear gas agents, known typically as the chemicals CN (chloroacetophenone) and CS (ortho-chlorobenzalmalononitrile). These tear gases are actually irritants causing pain and discomfort to the lacrimal glands (tear ducts) and the upper respiratory system. Delivery systems for these agents were comprised mainly of grenades and projectiles, commonly designed as pyrotechnic (burning) type devices. In Wortley, Jr. et al U.S. Patent 3,192,105, there is disclosed a method of combining CN with a colloidal silica to produce a solid phase lachrymator. As disclosed in the '105 patent, this solid phase lachrymator is well suited for use in munitions which disperse the lachrymator by an explosion or similar means.

In 1966, Smith and Wesson, through acquisition of General Ordnance Equipment Company, introduced CHEMICAL MACE® lachrymator, a liquid based CN chemical formula which was filled into pressurized aerosol spray containers. CHEMICAL MACE® lachrymator has been the forerunner of all such products since its introduction in 1966. Its use greatly lessens the amount of physical force which might be necessarily applied by a law enforcement officer, corrections officer or security

officer when attempting to detain a suspect or disperse a crowd of unruly persons. Lachrymators are generally classified as "nonlethal weapons."

The original CHEMICAL MACE® lachrymator formula consisted of trichlorotrifluoroethanes (CFC's 111, 113) and cosmetic kerosene as the carrier agent/solvent, blended with the active ingredient (CN), and pressurized with carbon dioxide as the propellant. This formula was used extensively until about 1990, but beginning about 1990 there was increasing pressure on the chemical industry to phase out ozone depleting substances (i.e., chlorofluorocarbons--CFC's) under the provisions of the Clean Air Act.

The CFC's, which serve as a nonflammable carrier, provide a significant advantage for the lachrymator solution. Specifically, lachrymator solutions that employ a CFC carrier are generally less likely to ignite. More particularly, when a lachrymator is dispensed, there is always the possibility that an open flame or other source of ignition may be present that could ignite the carrier of the mixture resulting in serious bodily harm to both the user and the intended recipient of the lachrymator. A nonflammable carrier is less likely to ignite when exposed to such sources of ignition and is thus a preferred carrier for use in lachrymators.

However, governmental actions have been taken worldwide to reduce and eliminate the use of CFC's because of the damage caused to the one layer. It is likely that CFC's will be totally phased out before the year 2000.

Manufacturers of CFC substances have been relentless in their search for acceptable substitutes. However, to this date, manufacturers have only been able to provide solvent replacements that do not totally eliminate the problem. The most recent substitute offered is a halogenated fluorohydrocarbon, HCFC-141b (often referred to as a soft CFC). However, soil CFC's still have an adverse effect on the ozone layer and their pressure has continued for developing carrier systems for lachrymators which do not employ CFC's or HCFC's.

Capsicum (also known as cayenne pepper) and its chemical equivalents such as capsaicin ( $C_{18}H_{27}NO_2$ ) have also been utilized to produce lachrymators for many years. Such lachrymators are commonly utilized in the liquid phase and are dispensed from pressurized canisters or bottles. Prior art capsicum containing lachrymators have comprised a mixture of capsicum, soybean oil and an alcohol type solvent. Capsicum containing lachrymators are preferred by some users. More particularly, some users believe that capsicum containing lachrymators are more effective than other prior art lachrymators. Additionally, some users prefer capsicum because it is a "natural" material as compared to the man-made chemicals CS and CN.

Unfortunately, because the prior art capsicum containing lachrymators also contain carriers which are primarily alcohol, they are considered unacceptable by some users because they may present too much of a

fire hazard. Additionally, some persons consider the prior art capsicum containing lachrymators to be unacceptable because they believe the carrier which is primarily alcohol may have an adverse impact upon tests which are utilized to determine the blood-alcohol level of a person exposed to the lachrymator. Thus, there is a need for a carrier system which does not adversely impact upon the blood-alcohol level of a recipient, is suitable for use with capsicum, does not contain CFC's and is nonflammable.

U.S. Patent 5,217,708 describes a capsicum lachrymator solution and canister for storing and applying such solutions. The solutions comprise capsicum (generally about 0.1% to about 0.8% by weight), about 7% to about 23% by weight of a glycol, about 31% to about 39% by weight of ethyl alcohol, and from about 46% to about 54% by weight of water.

### Summary of the Invention

The present invention provides a new and useful capsicum containing liquid lachrymator solution which comprises capsaicinoids and a nonflammable carrier, said solution comprising from about 0.1% to about 0.5% by weight of capsaicinoids, from about 7% to about 23% by weight of a glycol, from about 20% to about 30% by weight of ethanol and from about 55% to about 65% by weight of water.

### Description of the Invention

The lachrymator of the present invention includes a nonflammable carrier that is generally acceptable for use around or in the vicinity of potential sources of ignition such as, for example, lit cigarettes or cigars, candles and stoves. Furthermore, the nonflammable carrier does not contain any CFC's. Also, the lachrymator tends not to adversely impact upon the blood-alcohol level of a recipient. Additionally, the lachrymator is suitable for use in conventional pressurized aerosol spray canisters or containers and thus it does not have to be thermally vaporized by a heat source in order to perform properly. Further, the lachrymator is substantially water-based yet it is very stable for it does not freeze through a normal range of temperatures and it does not separate over periods of time. The lachrymator is also stable in that it does not spoil or lose its effectiveness over periods of time and there are no adverse interactions or reactions observed as between the various components of the lachrymator. Further, the lachrymator can be disposed of in a conventional manner.

A lachrymator solution made in accordance with the principles of the present invention comprises capsaicinoids and a nonflammable carrier. The nonflammable carrier comprises water, ethyl alcohol and glycol. The glycol may comprise ethylene glycol or propylene glycol or mixtures thereof. In a preferred embodiment, the glycol is propylene glycol. As used in this specification and claims the following terms used in the specification are

defined as follows: "capsicum" includes capsicum and chemical equivalents such as capsaicin; "lachrymator" is intended to be afforded a broad interpretation including any irritant-type product that may be utilized for defense purposes; and "nonflammable" means having a flash point in excess of about 100°F (38°C) or an ignition point above 500°F (260°C).

The lachrymator of the present invention is produced utilizing a mixture of capsaicinoids and a nonflammable carrier. More particularly, the lachrymator solutions of the present invention comprise from about 0.1% to about 0.5% by weight of capsaicinoids, from about 7% to about 23% by weight of a glycol, from about 20% to about 30% by weight of methanol, and from about 55% to about 65% by weight of water. In other embodiments, the lachrymator solutions of the present invention comprise from about 0.1% or 0.15% up to about 0.25% or 0.3% or 0.4% by weight of capsaicinoids, from about 10% or 12% to about 16% or 20% by weight of a glycol, from about 26% or 27% up to about 29% or 30% by weight of ethanol and from about 55% or 56% up to about 60% by weight of water. In a more preferred embodiment, the solutions useful as lachrymators comprise about 0.18% to about 0.22% by weight of capsaicinoids, about 12% to about 16% by weight of propylene glycol, about 27% to about 29% by weight of ethanol and about 57% to about 59% by weight of water.

Common sources of capsaicinoids useful in this invention include peppers such as cayenne peppers or chili peppers. The process usually involves drying and grinding peppers into a fine powder. Oleoresin capsicum present in the powder generally is extracted through the use of an organic solvent such as hexane. This mixture of powder and solvent is referred to as Miscella. Once the oleoresin extraction is completed, the solvent is removed by evaporation or distillation. After the solvent is removed from the Miscella, the remaining oily substance is raw oleoresin capsicum, the strength of which is dependent upon the grade of the peppers that were utilized. The raw oleoresin capsicum generally is diluted to various strengths according to customer specifications utilizing a glycol and water if the diluted product is to be water-soluble. Emulsifiers and surfactants may be included in the diluted mixture to improve the stability of the mixture (generally a suspension). These diluted products are generally referred to in the industry as oleoresin capsicum regardless of the concentration or strength of the raw oleoresin capsicum contained in the mixtures.

Oleoresin capsicum consists of three major components that cause the pungency or hotness. These components are known as capsaicinoids, and the three major capsaicinoids in oleoresin capsicum are capsaicin, dihydrocapsaicin and nordihydrocapsaicin. Minor capsaicinoids present in oleoresin capsicum include homocapsaicin and homodihydrocapsaicin. The capsaicinoid content of a given solution or mixture is the determining factor of how hot a product will be. For

example, the higher the capsaicinoid concentration, the hotter the product, and vice versa. Capsaicinoid concentration is the determination of the active components in oleoresin capsicum. Thus, a measurement of the amount of capsaicinoids in oleoresin capsicum provides an accurate level of pungency. The amounts and identification of the capsaicinoids in oleoresin capsicum can be detected utilizing high performance liquid chromatography (HPLC).

One example of a capsicum and propylene glycol mixture suitable for use in the present invention and which is commercially available is an oleoresin capsicum sold by Kalsec, Inc. of Kalamazoo, Michigan under the designation "Oleoresin Capsicum, W/S, decolorized 2% MC. This particular propylene glycol/water-based oleoresin capsicum solution contains about 5% oleoresin capsicum containing about 2.0% by weight of capsaicinoids.

An example of a suitable source of ethyl alcohol for use with the present invention is a denatured ethyl alcohol sold under the trade designation SDA-40-B by Midwest Grain Products, Inc. of Weston, Missouri 64098. SDA-40-B denatured ethyl alcohol contains about 99.9% by weight ethyl alcohol and about 0.1% by weight tertiary butyl alcohol and BITREX (denatonium benzoate).

An example of a suitable source of propylene glycol is a food grade propylene glycol USP available under the product code 70531 from the Dow Chemical Company of Midland, Michigan 48674.

When preparing the lachrymators of the invention, the indicated components are mixed together in a vessel using a stirrer for a minimum of about 10 to 15 minutes. In one embodiment, the oleoresin capsicum is first mixed with the ethanol followed by addition of the glycol and water. When preparing large quantities of the formulations as set forth in the following examples, the components can be mixed in a proportion shown for a period of about one hour in 55-gallon drums utilizing mixers. Thorough mixing is required in order to eliminate any separation of the components of the solution which would require additional mixing of the solution at a later time. Upon completion of the mixing step, the solution should appear clear and homogenous when viewed with a flashlight.

The following examples illustrate the lachrymator solutions of the present invention. Unless otherwise indicated in the following examples and elsewhere in the specification and claims, all parts and percentages are by weight, temperatures are in degrees Centigrade and pressures are at or near atmospheric.

#### **Example 1**

Denatured ethyl alcohol (114 parts) is charged to a mixing vessel followed by the addition of 42 parts of the Kalsec oleoresin capsicum solution described above (containing about 2.0% of capsaicinoids) which is first thoroughly mixed to restore homogeneity before it is

added to the ethyl alcohol. After the oleoresin capsicum is thoroughly mixed with the ethanol, 19.5 parts of propylene glycol USP grade are added to the mixing vessel followed by 209.6 parts of distilled water. The contents of the vessel are mixed for an additional 15 minutes. The mixture is the desired product which is analyzed and found to contain about 0.20% by weight of capsaicinoids, 14% by weight of propylene glycol, 28% by weight of ethanol and 58% by weight of water.

The in following Examples 2-5, the amount of the Kalsec oleoresin capsicum solution used in the examples is an amount sufficient to provide the indicated amount of capsaicinoids.

#### **Example 2**

	%/wt
Capsaicinoids	0.3
Propylene glycol	14
Ethanol	26
Water	59.7

#### **Example 3**

Capsaicinoids	0.22
Propylene glycol	16
Ethanol	26
Water	57.78

#### **Example 4**

Capsaicinoids	0.4
Propylene glycol	14
Ethanol	28
Water	57.6

**Example 5**

Capsaicinoids	0.15
Propylene glycol	12
Ethanol	27.85
Water	60

The lachrymator solutions made in accordance with the present invention may be utilized in any number of conventional pressurized aerosol spray containers or canisters. In order to be properly utilized in a pressurized canister, the nonflammable carrier should be a liquid or a gas at ambient temperature (i.e., about 70°F (21°C) and at ambient pressure. Preferably, the carrier has a boiling point of less than about 225°F (about 107°C) in order to ensure sufficient dispersion or distribution of the capsaicin once the solution is dispensed from the pressurized canister. Additionally, the nonflammable carrier does not solidify or freeze at low temperatures as low as -20°F. Further, the amounts of propylene glycol, ethanol and water present in the solution must be such that the components of the lachrymator solution do not separate out or come out of solution over extended periods of time such as three days, preferably about three to four weeks.

The lachrymator solution of the present invention offers several advantages over the lachrymators claimed in U.S. Patent 5,217,708. For example, these solutions of the present invention are characterized as having a higher flash point and a lower vapor pressure. For example, a lachrymator solution prepared in accordance with the procedure of Example XV in U.S. Patent 5,217,708 is found to have a flash point of about 80°F (25°C). A lachrymator solution prepared in accordance with the procedure of Example 1 of the present application is found to have a flash point of 89°F (31.6°C). Because of increased concern over flammability, this increase in flash point is a significant improvement. The reduction in vapor pressure exhibited by the solutions of the present invention result in more stable products and products which are able to withstand higher temperature extremes.

Another advantage of the solutions of the present invention when compared to the solutions described and claimed in U.S. Patent 5,217,708 is the presence of larger sized droplets when the product is aerosolized. The ballistic droplets that are produced from an aerosol lachrymator are comprised proportionately of the product ingredients. As the liquids and the droplets begin to evaporate, the droplets become smaller, thus leaving smaller particles. The smaller the aerodynamic particle size, the further into the lungs the particle may travel when inhaled, and since lachrymators are strong irri-

tants, smaller particle sizes are not beneficial since they could yield undesirable effects. The relative amounts of ethanol and water in an aerosolized droplet, therefore, have an effect on the relative size of the droplet and particle size. Because ethanol evaporates more readily than water, those droplets containing more ethanol become smaller and leave smaller particles. Therefore, since the solutions of the present invention contain relatively lesser amounts of ethanol than the solutions described in the '708 patent, the droplets containing more water and less ethanol will not reduce in size as much as the droplets formed from the solution of the '708 patent, and the particles are larger in size.

While the invention has been explained in relation to its preferred embodiments, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification.

**Claims**

1. A solution for use as a lachrymator comprising capsaicinoids and a nonflammable carrier, said solution comprising from about 0.1% to about 0.5% by weight of capsaicinoids, from about 7% to about 23% by weight of a glycol, from about 20% to about 30% by weight of ethanol and from about 55% to about 65% by weight of water.
2. A solution according to claim 1 wherein said glycol comprises propylene glycol.
3. A solution according to claim 1 or claim 2 comprising from about 0.1% to about 0.4% by weight of capsaicinoids.
4. A solution according to claim 3 comprising from about 0.15% to about 0.25% by weight of capsaicinoids.
5. A solution according to any one of claims 2 to 4 comprising from about 10% to about 20% by weight of propylene glycol.
6. A solution according to any one of the preceding claims comprising from about 26% to about 30% by weight of ethanol.
7. A solution according to any one of the preceding claims comprising from about 56% to about 60% by weight of water.
8. A solution according to any one of the preceding claims wherein the capsaicinoids comprise capsaicin, dihydrocapsaicin and nordihydrocapsaicin.
9. A lachrymator solution for use in a pressurized aerosol container that does not have a source of heat for vaporizing said solution, said solution comprising capsaicinoids and a nonflammable carrier suitable for use as a lachrymator.

ble for use in a lachrymator, said carrier being liquid at ambient temperature and pressure and having a boiling point of less than about 110°C, and said solution comprises from about 0.1% to about 0.4% by weight of capsaicinoids, from about 7% to about 23% by weight of propylene glycol, from about 20% to about 30% by weight of ethanol and from about 55% to about 65% by weight of water.

10. A lachrymator solution according to claim 9 comprising from about 0.15% to about 0.25% by weight of capsaicinoids. 10
11. A lachrymator solution according to claim 9 or claim 10 comprising from about 10% to about 20% by weight of propylene glycol. 15
12. A lachrymator solution according to any one of claims 9 to 11 comprising from about 25% to about 30% by weight of ethanol. 20
13. A lachrymator solution according to any one of claims 9 to 12 comprising from about 55% to about 60% by weight of water. 25
14. A lachrymator solution according to any one of claims 9 to 14 wherein the capsaicinoids comprise capsaicin, dihydrocapsaicin and nordihydrocapsaicin. 30
15. A lachrymator solution for use in a pressurized aerosol container wherein said solution comprises capsaicinoids and a nonflammable carrier suitable for use in the lachrymator, and said solution comprises from about 0.15% to about 0.25% by weight of capsaicinoids, from about 10% to about 20% by weight of propylene glycol, from about 25% to about 30% by weight of ethanol and from about 55% to about 60% by weight of water. 35
16. A lachrymator solution according to claim 15 wherein the capsaicinoids comprise capsaicin, dihydrocapsaicin and nordihydrocapsaicin. 40

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## EUROPEAN SEARCH REPORT

Application Number  
EP 95 30 0595

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
D,Y	US-A-5 217 708 (B.D. PINKNEY) * claims *	1-16	C06D7/00
Y	--- P.A. SANDERS 'Principles of Aerosol Technology' 1970 , VAN NOSTRAND REINHOLD COMPANY , NEW YORK, USA * page 4 - page 7 * * page 70 *	1-16	
A	--- US-A-5 307 960 (J.P. FAY, SR.) * column 3, line 60 - column 4, line 12 *	1,9,15	
A	--- R.C. WEAST 'Handbook of Chemistry and Physics, 54th Edition' 1973 , CHEMICAL RUBBER COMPANY PRESS , CLEVELAND, OHIO, USA * page C-231 *	1,9,15	
A	--- Section Ch, Week 9011, Derwent Publications Ltd., London, GB; Class B07, AN 90-079285 & JP-A-2 032 190 (OSAKA AEROSOL KOGYO KK) 1 February 1990 * abstract *	1,9,15	TECHNICAL FIELDS SEARCHED (Int.Cl.6)
A	--- US-A-3 900 560 (A. JACOBSON) * column 4, line 28 - line 44 * -----	1,9,15	C06D C09K
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 10 May 1995	Examiner Schut, R
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ***** & : member of the same patent family, corresponding document	

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