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PROCESS FOR PREPARING AEROSOL PACKAGES

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8 Claims. (Cl. 53—22)

ABSTRACT OF THE DISCLOSURE

Process for preparing aerosol by introducing into an aerosol container a dispensable liquid concentrate, closing the container with a valved closure and then introducing through the valved closure as propellant a saturated solution of a compressed gas selected from the group consisting of carbon dioxide, nitrous oxide and nitrogen in a normally gaseous, liquefied chlorofluorinated saturated aliphatic hydrocarbon.

This invention relates to aerosol packages containing a concentrate and a volatile propellant in liquid phase.

It is well known commercially to package liquid concentrates and volatile liquefied propellants in aerosol containers whereby the product is propelled from the container by the pressure built up therein by the propellant vapor when the container outlet valve is opened. Propellants employed for this purpose necessarily have a relatively high vapor pressure usually ranging from about 25 to 90 p.s.i.g. at 70° F. A vapor pressure within this range is required to insure that all of the liquid product will be expelled from the container at the desired velocity under all temperatures to which the container and contents may be subjected.

Chlorofluorinated saturated aliphatic hydrocarbons have been conventionally used in liquefied form as propellants in the aerosol art. These propellants, however, are relatively expensive. Accordingly, the aerosol industry has been seeking methods which will reduce the cost of propellants.

Compressed gases have been employed as propellants because of their extremely low cost and inertness. However, the failure of spray patterns due to pressure drop resulting from low stability of the gases in the liquid concentrates leads to limited use of compressed gases. The chlorofluorinated saturated aliphatic hydrocarbons, on the other hand, provide constancy of pressure throughout use and minimal required head space.

In an effort to retain the advantages of the liquefied chlorofluorinated saturated aliphatic hydrocarbons, while reducing the cost of the aerosol formulations, the chlorofluorinated saturated aliphatic hydrocarbon propellants have been used in conjunction with compressed gases. Such use has involved sequential introduction into the aerosol container of the chlorofluorinated saturated aliphatic hydrocarbon and the compressed gas. The vapor pressures developed in the containers have, however, been unduly high. High vapor pressures are obviously undesirable due to stringent safety and shipping regulations concerning aerosol containers.

It is therefore an object of the present invention to provide aerosol packages prepared by an improved economical procedure.

A particular object of the invention is to provide aerosol packages prepared by a procedure whereby the amount of chlorofluorinated aliphatic hydrocarbon propellant is reduced while retaining affective low vapor pressures.

Other objects and advantages of the invention will be apparent from the following description.

In accordance with the present invention, aerosol packages are prepared by introducing into an aerosol container a liquid concentrate, closing the container with a valved closure and then introducing through the valved closure a saturated solution of a compressed gas selected from the group consisting of carbon dioxide, nitrous oxide and nitrogen in a liquefied chlorfluorinated saturated aliphatic hydrocarbon as propellant.

The liquid concentrates of this invention are composed essentially of an organic or aqueous liquid medium comprising generally about 65 to 90% by weight of the formulation. Among suitable organic liquid media are monohydric alcohols containing 1 to 3 carbon atoms, such as ethyl alcohol, n-propyl alcohol, isopropyl alcohol, etc. Part of the alcohol medium may be replaced by water, if desired. Other organic liquid media include chlorinated hydrocarbons such as methylene chloride, dialkyl ketones such as acetone, glycerol, monoalkylene glycols or polyalkylene glycols in which the alkylene groups contain 2 to 4 carbon atoms such as ethylene glycol and diethylene glycol, monoalkyl ethers of ethylene glycol and diethylene glycol such as ethylene glycol monomethyl ether, diethylene glycol monoethyl ether, etc. The aqueous liquid medium is typically water itself.

Any chlorofluorinated saturated aliphatic hydrocarbon may be used in liquefied form as the essential propellant in the process of this invention. These propellants generally have vapor pressures within the range of about 5 to 300 p.s.i.g. and preferably about 10 to 85 p.s.i.g. at 70° F. Dichlorodifluoromethane, trichloromonofluoromethane, dichlorotetrafluoroethane and mixtures thereof have been found to be particularly outstanding propellants. Among other propellants which may be employed are monochlorodifluoromethane, trichlorotrifluoroethane, dichloromonofluoromethane and mixtures thereof. In any specific application, the propellant used must possess solubility in the liquid concentrate.

The compressed gases used in the present invention are carbon dioxide, nitrous oxide and nitrogen. The selected compressed gas is pressured at the desired temperature into the liquefied chlorofluorinated saturated aliphatic propellant in order to obtain a saturated solution of the compressed gas in the propellant. This is assured by pressuring the compressed gas into the propellant to about 90 p.s.i.g.

The amount of propellant may vary from about 10 to 35% by weight of the formulation. The quantity employed is generally determined by the particular propellant used and its vapor pressure at the existing temperature.

In actual commercial operation, the saturated solution of compressed gas in liquefied chlorofluorinated saturated aliphatic hydrocarbon propellant may be readily prepared

in a tank truck or storage tank. First the liquefied propellant is added to the truck or tank, and the compressed gas is then added under pressure by means of a gas transpiration system. Pipes longitudinally arranged at the bottom of the truck or tank and uniformly perforated may serve in this capacity. The compressed gas is added while the propellant is thoroughly circulated or agitated, and the gas addition is discontinued after the propellant has become completely saturated. The degree of saturation can be determined by means of a continuous sampling chromatograph. The solution of the compressed gas in the propellant is then ready for use in the process of this invention.

Depending on the nature of the product desired, the formulations may contain various additives which are soluble therein including perfumes, medicinal substances, lubricants, resins, waxes, aldehydes, ketones, petroleum hydrocarbons, ethers, menthol, camphor, etc.

In filling an aerosol container in accordance with the present invention, a liquid concentrate is first introduced into the container. The container is then purged of air and is closed with a valved closure. Finally, a saturated solution of the compressed gas in liquefied propellant is introduced through the valved closure by means of a pressure-injector. The resulting composition is thereby confined in the container under the vapor pressure of the propellant.

Introduction of a compressed gas into a aerosol container subsequent to a liquefied chlorofluorinated saturated aliphatic hydrocarbon must be accomplished at pressure of about 90 p.s.i.g. in order to fill the container within a reasonable period of time. The container then retains a vapor pressure of about 90 p.s.i.g. It was quite unexpected, therefore, that by simply introducing a saturated solution of compressed gas in a liquefied propellant, as described above, a considerably lower vapor pressure is attained. In the case of organic liquid concentrates, the vapor pressure developed is generally not above about 40 p.s.i.g.

The procedure of the present invention possesses the distinct advantage of permitting use of considerably reduced quantities of chlorofluorinated saturated aliphatic hydrocarbon propellant, as compared to use of the propellant per se. Moreover, the procedure enables the obtainment of substantially lower vapor pressure then developed from separate introduction of the propellant and compressed gas. Further, much less compressed gas has been found to be required in the procedure of this invention than that necessitated by separate introduction of the propellant and compressed gas.

Experiments were carried out using a 50:50 (by weight) mixture of liquefied dichlorodifluoromethane and trichloromonofluoromethane in formulations containing ethyl alcohol as liquid concentrate. Up to 65% of propellant by weight of the formulation was required in order to attain adequate vapor pressures. Frequently with such high percentage of propellant, over-atomization of the concentrate occurs. On the other hand, when a saturated solution of carbon dioxide in the same propellant was employed, the mixture in the aerosol container possessed an ideal vapor pressure of about 34 p.s.i.g. using only 35% of propellant by weight of the formulation.

The following examples are further illustrative of the present invention.

Example I

Ethyl alcohol was introduced into each of two conventional aerosol containers. The containers were purged of air and closed with valved closures. Saturated solutions of compressed gas in liquefied chlorofluorinated saturated aliphatic hydrocarbon propellants were then introduced through the valved closures. In each case, the compressed gas was pressured into the liquefied propellant to 90 p.s.i.g. The results obtained are set forth in the following table. In the table, percentages are by weight.

TABLE I

	Percent of Formulation	
	I	II
Formulation:		
12/11/CO_2[1] (54%/46%/CO_2) (CO_2 to 90 p.s.i.g. at 75° F.)	35	-------
12/11/N_2O[1] (50%/50%/N_2O) (N_2O to 90 p.s.i.g. at 75° F.)	-------	35
Ethanol	65	65
	100	100
Data:		
Vapor Pressure, Full Can at 75° F., p.s.i.g.	40	40
Vapor Pressure, ½ Full Can at 75° F., p.s.i.g.	36	35
Spray Rate, Full Can, gm./min.	40	48
Spray Rate, ½ Full Can, gm./min.	42	39

[1] 12=dichlorodifluoromethane and 11=trichloromonofluoromethane

Similar results are obtained using nitrogen as the compressed gas in the above formulations.

Example II

Water was introduced into a conventional aerosol container. The container was purged of air and closed with a valved closure. A saturated solution of compressed CO_2 in liquefied propellant was then introduced through the valved closure. The results obtained are set forth in the table below, in which percentages are by weight.

TABLE II

Formulation:	
12/CO_2[1] ------- Percentage of formulation --	20
(CO_2 to 90 p.s.i.g. at 70° F.) water based concentrate ------- do ----	80
	100
Data:	
Vapor pressure full can at 70° F. ----- p.s.i.g --	56
Vapor pressure ⅕ full can at 70° F. --- p.s.i.g --	46
Spray rate full can ------- gm./min --	48
Spray rate ⅕ full can ------- gm./min --	36

[1] 12=dichlorodifluoromethane.

Since many different embodiments may be made in this invention without departing from the scope and spirit thereof, it is intended to be limited only as indicated in the appended claims.

I claim:

1. A process for preparaing an aerosol package which comprises introducing into an aerosol container as dispensable liquid concentrate, closing the container with a valved closure and then introducing through the valved closure as propellant a saturated solution of a compressed gas selected from the group consisting of carbon dioxide, nitrous oxide and nitrogen in a normally gaseous, liquefied chlorofluorinated saturated aliphatic hydrocarbon.

2. The process of claim 1 in which the compressed gas is carbon dioxide.

3. The process of claim 1 in which the chlorofluorinated saturated aliphatic hydrocarbon is a member of the group consisting of diclorodifluoromethane, trichloromonofluoromethane, dichlorotetrafluoroethane and mixtures thereof.

4. The process of claim 1 in which the liquid concentrate is composed essentially of an organic liquid as medium.

5. The process of claim 1 in which the liquid concentrate is composed essentially of a saturated aliphatic monohydric alcohol containing 1 to 3 carbon atoms as medium.

6. The process of claim 1 in which the liquid concentrate is composed essentially of an aqueous liquid as medium.

7. The process of claim 1 in which the liquid concentrate is composed essentially of water as medium.

8. A process for preparing an aerosol package which

comprises introducing into an aerosol container a dispensable liquid concentrate composed essentially of ethyl alcohol as medium, closing the container with a valved closure and then introducing through the valved closure as propellant a saturated solution of carbon dioxide in a liquefied chlorofluorinated saturated aliphatic hydrocarbon of the group consisting of dichlorodifluoromethane, trichloromonofluoromethane, dichlorotetrafluoroethane and mixtures thereof.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

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It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 12, after "aerosol" insert -- packages --. Column 3, line 45, "pressure" should read -- pressures --; same line 45, "then" should read -- than --. Column 4, line 48, "preparaing" should read -- preparing --; line 49, "as" should read -- a --.

Signed and sealed this 16th day of December 1969.

(SEAL)

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Commissioner of Patents