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## 3,145,113 PRESSURIZED FOOD PACKAGE WITH HEXA FLUOROTRIMETHYLENE OXIDE AS THE PROPELLANT

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This invention relates to a new fluorocarbon propellant for use in aerosol containers, particularly those designed for food mixtures. This invention also relates to processes for producing aerated food products.

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For some time, the desirability of packaging food and 15 other formulations in aerosol containers has been appreciated by those in the packaging industry. Although many different articles have been packaged in aerosol containers through the use of a variety of propellant gases, certain problems and disadvantages have remained un-20 solved. Commonly used propellants such as nitrous oxide, carbon dioxide, and the like, prefrably those which are at least partially soluble in the dispensed products, have been used. Unfortunately, since such gases require extremely high pressure for liquefaction, they have generally 25 been contained in the aerosol bomb in compressed gaseous form. With ordinary usage the aerosol pressure rapidly drops and frequently is insufficient to empty the contents thereof. Frequently a defective valve can result in discharge of sufficient propellant to render the aerosol wholly 30 or partially unoperative.

Other propellants frequently suggested for use in aerosol containers, including various hydrocarobns, difluorodichloromethane and similar materials, impair the flavor of a food product and frequently impart undesirable taste, 35 color or odor to a propelled food mixture.

It is therefore desirable to provide a liquefied gaseous propellant, i.e., a liquid having vapor pressure which exceeds atmospheric pressure at ordinary room tempera-The liquefied gases permit larger volumes of the 40 product to be placed in the container and additionally provide substantially constant pressure in the container until the product can be completely discharged. Moreover, the liquefied gases also permit a larger and more constant supply of gas for propellant purposes and hence 45 a measure of safety in the event of leakage, partial valve failure, etc. Customarily, satisfactory liquefiable gases for propellant use have a vapor pressure of from about 5 to about 95 pounds per square inch at ordinary room temperature. 50

With food products it is apparent that the propellants, as well as the other ingredients contained in the aerosol, must not be harmful to either the manufacturer or the consumer and should permit safe packaging, storage and The propellant should also be chemically inert 55 use. under the conditions of use and of prior storage. Chemical reaction between the propellant gases and/or materials produced from the degradation of such gases and the containers used should be prevented, preferably by selection of an appropriate stable propellant. Satisfactory propellants should be non-corrosive and should not generate corrosive products, particularly fluoride ion at a rate greater than about 1 million parts of the medium per year at 130° F., when in contact with neutral, basic and acidic aqueous media.

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Hydrocarbon materials are unsatisfactory because of their objectionable taste and odor and potential fire hazard. Other organic propellants, such as methyl chloride, are unsuitable because of their water sensitivity, ability to produce corrosive hydrochloric acid, and objectionable odor or taste. Many otherwise satisfactory propellants must also be disqualified because of the color which they impart to the dispersed products. Many of the high halogenated compounds such as dichlorodifluoromethane, though generally regarded as non-corrosive, do not possess the desired hydrolytic stability to prolong contact with aqueous media at the elevated temperatures to which many packaged foods and other products, such as shaving creams, are subjected during storage and shipment.

Materials which meet the above-mentioned stringent requirements are extremely difficult to find, and only a few are in commercial use at the present time. Materials which do meet the above-mentioned qualifications are available only from a limited number of sources and are produced by relatively restricted number of processes. It is, therefore, an object of this invention to provide a propellant for general aerosol use, particularly for food product aerosols, which consists essentially of a unique liquefied gas. Another object is to provide such products and a process for producing aerated fod products through the use of novel propellants in which the liquefied gas is non-corrosive to the container, is chemically inert, is nontoxic, is hydrolytically stable, does not release objectionable fluoride ion concentrations and does not adversely affect the odor, taste, appearance, texture and color of the dispensed product. Yet another object is to provide such products in which the propellant comprises a mixture of said liquefied gas and a minor proportion of a pressurized gas to increase the over-all container pressure, which pressurized gas may or may not be soluble in the liquefied gas and also has the aforementioned advantages associated with the liquefied gas. Other objects and advantages will appear hereinafter and will be apparent to those skilled in the art.

According to this invention, these objects are achieved by charging a pressurized container or aerosol bomb with an aerosol-dispensible, water containing edible food product and with a propellant from the group consisting of hexafluorotrimethylene oxide, and mixtures thereof with a proportion of an inert gas which does not liquefy at room temperature and pressures below 120 p.s.i.g. and which is capable of adjusting the pressure in the charged aerosol container to between about 60 to 120 p.s.i.g. at room temperature, e.g. 70° F. This cyclic perfluorinated ether and its preparation appears in U.S. Patent 2,594,272.

Hexafluorotrimethylene oxide has the formula:



and boils at  $-38^{\circ}$  C. It is chemically inert, non-flammable, colorless, water insoluble and imparts essentially no taste or odor to food products. It is readily liquefied 60 at the pressures normally desired in pressurized aerosol dispensing containers or bombs. It is non-corrosive to ordinary metals, such as steel, aluminum, copper, brass, tin, lead and zinc at temperatures below 130°F., even in the presence of alcohols, oils and water. Moreover, it 65 liberates fluoride ion in the presence of water at a negligible rate well below the tolerance for food products at 130° F. and below even under more extreme temperature conditions.

Because of the vapor pressure vs. temperature relationship hexafluorotrimethylene oxide is in gaseous form 5at ordinary room temperatures but is readily retained at least partially in liquid form in the aerosol containers at the ordinary pressures utilized. The vapor pressure ranges from about 4 to about 25 p.s.i.g. over the temperature range from about 20° F. to about 70° F., which 10 provides adequate discharge of the ordinary aerosol-dispensible product from such pressurized containers. Naturally, the more viscous the product, the greater the pressure differential between the contents of the container and the outside atmosphere; for example, the relatively fluid 15 formulations of low viscosity, such as paints, deodorants, perfumes, salad dressings, whipping cream, etc. Only a few pounds pressure differential is required. For the more highly viscous products, such as cake frosting, cheese spreads, condiments, higher pressures are required. In 20 most instances the pressure requirements of the aerosol are such that an auxiliary propellant is desirable to bring the internal pressure of the container to between about 60 and about 120 p.s.i.g. at 70° F. Such auxiliary propellants may be normally in gaseous form even at the 25 internal pressures of conventional aerosols. Illustrative of the known volatile, inert, auxiliary propellant which may be utilized for raising the internal pressure are chloropentafluoroethane, chlorotrifluoromethane, tetrafluoromethane, carbon dioxide, nitrous oxide, and mixtures 30 thereof, etc. These auxiliary propellants are usually present in a proportion sufficient to increase the internal pressure to the aforementioned limits, although smaller amounts may be utilized when lower internal pressures are desired. In certain instances, no auxiliary propellants 35 are required. The total internal pressure of such contained mixtures of hexafluorotrimethylene oxide and auxiliary propellants depends upon the mol fractions of the various propellants and their respective partial pressures. 40When per-halogenated propellants are utilized as auxiliary propellants, usually the mixtures contain from about one to about 10 percent by weight of the more volatile auxiliary propellants compounds. When the non-halogenated auxiliary propellants are employed, usually from about 4550 to about 85 percent of these gases are used.

In the self-propelling food mixtures of the invention, the propellants may either be emulsified or may be present as a separate layer. However, the total quantity of propellants used should be such as to provide a liquid phase of liquid propellants both initially and also throughout 50 the usable discharge life of the aerosol. This quantity may be from about 3 to about 10 percent by weight of the total contents of the container. Although larger quantities may be employed, the filled aerosol dispenser contains a small free space above the liquid contents thereof, 55 which is occupied by propellant gases. These gases represent only minor proportions of the total propellant, and the remaining propellant is either liquefied or is dissolved in or dispersed throughout the product.

In order to illustrate the invention and preferred em- 60 bodiments thereof, the following examples are given in which the proportions are by weight unless otherwise indicated:

### Example 1

A cake frosting is prepared by melting 1/4 cup of butter or oleo. Two cups of sifted confectioner's sugar and enough evaporated milk to obtain the desired consistency are then added. After the addition of one tablespoon of vanilla, the mixture is beaten until creamy.

One-hundred grams of this cake frosting mix is added to an aerosol container, leaving sufficient volume of free space for propellant vapors. Such methods for inserting the frosting in the aerosol and pressurizing the container is conventional. The container is then capped with a 75 pressure to from about 50 to about 110 p.s.i.g. at 70°

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foam valve and about 4 grams of hexafluorotrimethylene oxide is admitted to the can through a conventional pressure loader. The can is further pressurized through the valve with an 85/15 nitrous oxide/carbon dioxide mixture to a total pressure of about 100 p.s.i.g. at 70° F. The container thus loaded is tumbled for 1/2 hour and the contents are discharged at 70° F. into an aluminum dish. The discharged product is a full-bodied foam having a smooth creamy appearance. The texture and appearance of the product is essentially unchanged after standing overnight at room temperature. The pressure in the container is maintained throughout the discharge life of the aerosol.

#### Example 2

Pressurized corn syrup is packaged in a four-ounce plastic bottle using about 130 grams of commercial corn syrup and about 15 grams of hexafluorotrimethylene oxide as a liquefied gas propellant. The propellant forms a bottom layer and the dip tube of the standard foam valve is cut off above the propellant surface. When the valve is operated, a steady discharge of syrup is obtained.

#### Example 3

Using the cake frosting of Example 1 about 300 grams of this frosting is added to a 16-ounce aerosol can and purged of air by hexafluorotrimethylene oxide gas. After capping the can with a foam valve about 30 grams of liquefied hexafluorotrimethylene oxide is added, the total charge representing about an 80 percent fill. After shaking, about 85 percent of the contents may be discharged as a fluffy white frosting.

Using an 85/15 percent mixture of nitrous oxide and carbon dioxide as a pressurized gas propellant instead of the hexafluorotrimethylene oxide and pressuring the same cake frosting mix to 100 p.s.i.g. the discharged product is pasty and inferior in both texture and appearance.

#### Example 4

The procedure of Example 3 is then repeated, inserting about 10 grams of liquefied hexafluorotrimethylene oxide and further charging said container with sufficient chlorotrifluoromethane to raise the pressure to 100 p.s.i.g. The food product dispenses smoothly and with good appearance until the container is empty.

It will be understood that the preceding examples are illustrative only and that the invention is not limited to the specific embodiments disclosed therein. It will also be apparent that this invention provides a unique combination of an aerosol dispensable product and a novel propellant, therefore, which is primarily in the form of a liquefied gas and which has advantages of propellants heretofore used, particularly for use with food formulations.

I claim:

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1. A pressurized aerosol dispensing container in which is contained an aerosol dispensible, water-containing food product and a propellant consisting eccentrically of hexafluorotrimethylene oxide at least partially in liquefied form and sufficient volatile inert gas to adjust the container pressure to from about 50 to about 110 p.s.i.g. at 70° F.

2. The pressurized aerosol dispensing container of claim 1 in which said volatile inert gas is perhalogenated and constitutes from about 1 to about 10 percent by weight of the total propellant.

3. The pressurized aerosol dispensing container of claim 1 in which said volatile inert gas is non-halogenated and constitutes from about 50 to about 85 percent by weight of the total propellant.

4. A pressurized aerosol dispensing container in which 70is contained an aerosol dispensable, water-containing food product and a propellant consisting essentially of hexafluorotrimethylene oxide at least partially in liquified form and sufficient volatile inert gas to adjust the container

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F., the hexatrifluoromethylene oxide in liquified form constituting from about 3 to about 10 percent by weight of the total contents of the container.

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# UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 3,145,113

August 18, 1964

Roy J. Mordaunt

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 25, for "liquefaction" read -- liquefication --; column 2, line 25, for "fod" read -- food --; column 4, line 57, for "eccentrically" read -- essentially --.

Signed and sealed this 12th day of January 1965.

(SEAL) Attest:

ERNEST W. SWIDER Attesting Officer EDWARD J. BRENNER Commissioner of Patents

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