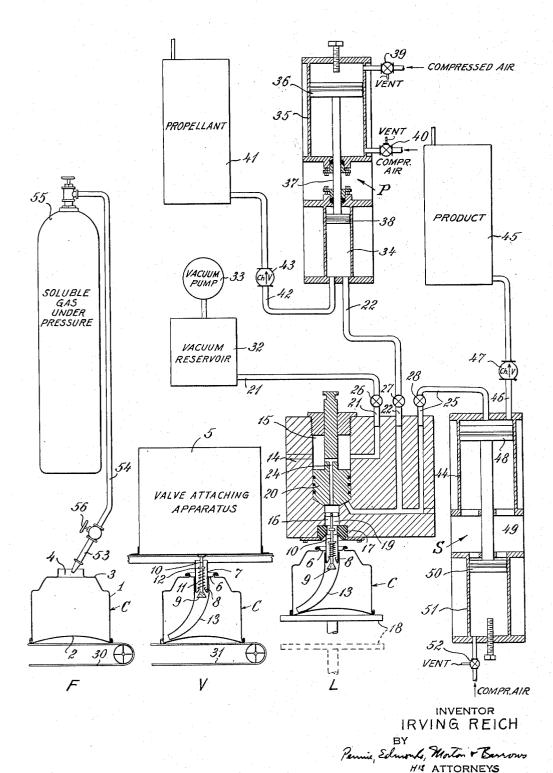
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ETHOD OF FILLING PRESSURETIGHT CONTAINERS WITH A LIQUID PRODUCT AND A VOLATILE PROPELLANT METHOD

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This invention relates to the filling of pressure-tight containers with a composition comprising a mixture of a liquid product and a volatile propellant in liquid phase, which propellant has a vapor pressure considerably above atmospheric pressure at room temperature and accordingly acts to propel the product out of the container. The propellant may also act to modify the physical state of the product as it emerges from the container, as by expanding it 10into a foam or lather. The present invention more particularly concerns a method of so packaging such a composition that no substantial partial pressure of air remains in the head space of the filled and closed container, whereby 15the pressure in such head space is substantially solely that due to the volatile propellant.

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Certain liquid compositions comprising liquid products and volatile propellants have been packaged in pressure-tight containers, such as cans, 20 equipped with outlet valves whereby the product is propelled from the can by the pressure built up therein by the propellant vapor when the outlet valve is opened. Propellants useful for this purpose necessarily have a relatively high 25 vapor pressure, usually ranging from about 30 to 80 or 85 pounds per square inch gauge at 70° F. A vapor pressure within this range is necessary to insure that all of the liquid product will be expelled from the can at the desired velocity 30 under all temperatures to which the can and its contents may be subjected in use. Although high vapor pressures near or even above the upper limit indicated are desirable for effective expulsion of the product, high pressures are clearly 35 undesirable when safety and shipping regulations are considered. Thus, if the can is made economically of light gauge metal, such as is used for ordinary canned foods and beverages, the maximum pressure developed in the can at the 40 vapor pressure permissible in the construction maximum temperature to which it is likely to be subjected must be held below a limiting value. The limiting value may be fixed by the pressure at which the can will bulge or leak or it can be fixed by shipping regulations. In any case, if the vapor pressure of the propellant is such that the maximum safe pressure is exceeded, the only alternative is to employ expensive heavy gauge metal containers.

When cans are filled with liquid product and 50 volatile propellant, a considerable proportion of the ambient air that occupies the empty cans before filling is trapped in the head space of the can, and the vapor pressure of this air increases the head space pressure to a value above that of 55 will be made to the accompanying drawing, in

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the vapor pressure of the volatile propellant. Evacuation of the cans prior to filling reduces the density of the air so trapped, but complete removal of the air prior to filling cannot be economically accomplished in commercially practical filling operations. The air pressure is not useful in propelling the product from the can and it is undesirable in that it increases the head space pressure due to the propellant and so requires the use of undesirably low vapor pressure propellants if dangerous or forbidden pressures are to be avoided.

With the above considerations in mind, it is proposed, in accordance with the present invention, to provide a method of packaging compositions that comprise essentially liquid products and volatile propellants, which comprises first displacing the ambient air in the empty can or container with a gas that is soluble in the composition to be packaged, then preferably evacuating the gas filled container to subatmospheric pressure, then introducing the composition to be packaged in sufficient quantity and under sufficient pressure to bring the pressure in the can to a value considerably above atmospheric pressure, and finally closing the can. In some cases, the step of evacuating the can after displacement of the air can be omitted. The result of the employment of these combined steps is that, when the composition is introduced into the can, the pressure therein is suddenly increased and the gas in the can dissolves in or comprises a part of the composition and so does not add its partial pressure to the pressure of the gaseous volatile propellant in the can head space. With this procedure, the head space pressure comprises substantially only the pressure of the volatile propellant, and accordingly a propellant can be used which has the maximum of can or container being filled.

The gas employed to flush out and displace the air in the can prior to filling may be soluble in either constituent of the composition filled. 45 In one form of the invention, where the product is an aqueous solution or suspension, the flushing gas is preferably a water-soluble gas and hence is soluble in the product constituent of the filled composition. It is also contemplated that the flushing gas may be soluble in the propellant ingredient of the composition, in which case the flushing gas may comprise the propellant in gaseous phase.

In describing the invention in detail, reference

which the single figure illustrates in diagrammatic and simplified form one arrangement of apparatus by which the method of the invention may be carried out.

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In the drawing, there is shown for the purpose 5 of illustration a can or container C comprising cylindrical side wall I, a bottom wall 2 sealed thereto and an integral top wall 3 having a central valve receiving opening 4 therein. The valve assembly of the illustrated can may be sealed in- 10 to the can opening 4 by suitable attaching mechanism, such as a seamer, of known construction, diagrammatically illustrated at 5. The can may be carried to the flushing station F and the valve attaching station V by hand or by suitable con- 15 ble cylinder construction and includes a product veyors diagrammatically illustrated by the belts 30 and 31.

The can valve assembly shown by way of illustration comprises a disc 6 having a valve tube 7 extending through and sealed in a central open- 20 ing therein. The tube 7 has an inturned flange 8 at its inner end which forms a valve seat for cooperation with a valve plug 9 carried by a stem 10 extending through the tube 7. The stem 10 is biased to move to the valve closing position 25 by a spring 11 compressed between the inner face of the flange 8 and a stop 12 struck from the stem 10. A syphon tube 13 may be fitted over the inner end of the valve tube 7 and extend to a point near the bottom of the can to insure substantially com-30 plete expulsion of the can contents when the can is in a substantially vertical or slightly tilted position.

The can is preferably filled with the composition at the filling station L after the can valve 35 assembly is attached thereto, and the flushing operation preferably takes place at the flushing station F before the valve assembly is secured to the can. The filling operation may involve partial evacuation of the can and essentially involves 40 the introduction of product and propellant from separate sources. The can is preferably sealed in gas-tight connection with the filling conduits during the evacuating and filling operations. Various forms of evacuating and filling apparatus 45 may be used.

As shown, the filling mechanism includes a block 14 having a valve chamber 15 therein communicating with a filling opening 16, the downwardly disposed mouth of which is surrounded by 50 a sealing gasket 17 shaped to engage and form a tight seal with the valve tube 7 of a can C when the can is lifted into contact therewith by a platform lift 18 of suitable construction. A valve operating rod 19 is centrally secured in the filling 55 opening 16 of the block 14 so as to engage the valve stem 10 and so open the can valve when the can is lifted to the filling position, as shown.

A piston valve 20 is slidably disposed in the valve chamber 15. In its lower position as shown, the 60 piston valve connects a vacuum conduit 21 with the filling opening 16 through a passage 24 in the piston valve 29. In its raised position, the piston valve 20 closes the vacuum conduit 21 and connects the propellant conduit 22 and the prod- 65 uct conduit 25 to the filling opening 16. Suitable valves 26, 27 and 28, here shown as manually operable valves, are provided to respectively control the vacuum, propellant and product conduits 21, 22 and 25. The vacuum conduit 21 is 70 connected to a suitable source of vacuum illustrated as a reservoir 32 evacuated by a vacuum pump 33. The propellant conduit 22 is connected to the propellant cylinder 34 of a propellant me-

prising essentially a power cylinder 35 carrying a piston 36 connected by a rod 37 to a piston 38 in the propellant cylinder 34. Compressed air or other motive fluid from a suitable source is alternately admitted to and vented from opposite ends of the power cylinder 35 under control of the valves 39 and 40 in order to reciprocate the connected pistons 36 and 38 and so force successive measured charges of propellant from the cylinder 34 and into the successive cans to be filled. Volatile propellant in liquid phase is supplied to the propellant cylinder 34 from a supply tank 41 through a conduit 42 including a check valve 43.

The product metering device S is also of doucylinder 44 connected to the product conduit 25 and to a product supply tank 45 through a conduit 46 including a check valve 47. A piston 48 in the product cylinder 44 is connected by a rod 49 to a power cylinder piston 50. Compressed air or other motive fluid is admitted to and vented from the power cylinder 51 by means of a valve 52, whereby the connected pistons are reciprocated to deliver successive measured charges of liquid product to the successive cans to be filled. In operating the apparatus described, a can C is lifted into sealed contact with the gasket 17 around the filling opening 16 and at the same time its valve stem 10 is depressed to open the can valve. The filling apparatus is then operated by a suitable manipulation of the described valves to first partially evacuate the can, where partial evacuation is used, and then supply measured quantities of product and of volatile propellant in liquid phase to the can, thus raising the pressure in the can to a pressure considerably above atmospheric pressure, a portion of the filled volatile propellant vaporizing and occupying the head space of the can. The pressures under which the product and propellant are introduced into the can are maintained at high superatmospheric values considerably above the vapor pressure of the volatile propellant at the prevailing temperature, so that the filling operation is rapid and no substantial vaporization of the propellant occurs during filling. After filling, the can C is lowered and its valve closes.

In accordance with the present invention, there is provided at the flushing station F a suitable duct for introducing the soluble flushing gas to displace the ambient air in the empty can. As shown, there is provided a tube or nozzle 53 connected by a flexible conduit 54 with a cylinder or tank 55 containing the flushing gas under pressure. A valve 56 adjacent the nozzle 53 controls the flow of gas therefrom. Preferably before the valve assembly is secured in the opening 4 of the can, the nozzle 53 is placed adjacent or partially in the can opening 4, the valve 56 is opened and a stream of gas is introduced into the can to substantially completely displace the ambient air therein.

In accordance with the invention, the flushing gas used to displace the ambient air in the empty can is soluble in the composition to be filled into the can. The method of the invention may be employed with advantage in the filling of compositions comprising products with propellants that are relatively insoluble in the products. Examples of such compositions are those comprising aqueous solutions or suspensions of soap, synthetic detergents or other water-soluble or suspendable materials, and volatile propellants that are relatively insoluble in water or aqueous solutering device, generally designated P and com- 75 tions. Such propellants include the relatively in5

soluble saturated aliphatic hydrocarbons of suitable vapor pressure, including propane, butane, iso-butane and cyclo-butane, as well as the substantially water insoluble chlorine and fluorine substituted hydrocarbons and particularly those in which all of the hydrogen atoms are replaced by chlorine and fluorine and in which the number of fluorine atoms in the molecule equals or exceeds the number of chlorine atoms, such as dichlordifluormethane (CCl₂F₂), 1,2 dichlor 1,1,2,2 10 tetrafluorethane (CClF2.CClF2) and trichlortrifluorethane (C₂Cl₃F₃). Aqueous product and volatile propellant compositions of this type are disclosed in detail in copending application Serial No. 125,032, filed November 2, 1949 and entitled 15 "Lather Producing Composition and Method."

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When packaging compositions of the type above identified, the flushing gas preferably comprises a compound that is readily soluble in water or aqueous solutions. Carbon dioxide and nitrous 20 oxide are preferable because they are relatively inert, inexpensive and substantially odorless. When the aqueous product is slightly alkaline, as may be the case with ordinary scap solutions, carbon dioxide is preferred because it is readily 25 soluble in alkaline solutions. If the aqueous product is neutral, nitrous oxide is preferable to carbon dioxide as the flushing gas. Other watersoluble gases, such as ammonia and sulfur dioxide, could be used but would be objectionable 30 in some applications because of their odor.

When a product soluble flushing gas is used, it displaces the air in the can and the subsequent partial evacuation of the can, if employed, reduces the density of the gas. Then when the 35 product and propellant are introduced, the remaining flushing gas dissolves in the aqueous product, such solution being aided by the turbulent flow of the product into the can and by the increase in pressure to a super-atmospheric value 40 due to filling under pressure, as above described. Due to such dissolving of the flushing gas, the pressure in the head space of the filled can is substantially solely that due to the vaporized propellant, and accordingly does not substantially 45 exceed the vapor pressure of the propellant at the prevailing temperature. The flushing gas may be soluble in the propellant rather than in the product, in which case the flushing gas may comprise the volatile propellant in vapor phase, examples 50 of which have been given above. When the flushing gas thus comprises the same compound as the volatile propellant but in vapor phase, the residue of the flushing gas can be partially evacuated if partial evacuation is employed, and 55 at the time of filling the gas is either dissolved in the filled propellant in liquid phase or remains as part of the head space vapor, but does not increase the head space pressure above the vapor pressure of the volatile propellant introduced. 60 The use as the flushing gas of gaseous volatile propellant is generally less desirable than the use of a product soluble gas, for the reason that the propellant is ordinarily relatively expensive, as compared to product soluble gases, such as those 65 indicated above.

According to one aspect of the invention, I omit the step of partially evacuating the can after displacement of the air therein by the flushing gas. According to this modification, the method 70 liquid product and a volatile propellant in liquid comprises displacing the ambient air in the can with the flushing gas and then filling the can with the composition under superatmospheric pressure as before, and then closing the can. This modification of the method is particularly effec- 75 pressure in the container above atmospheric pres-

tive when the flushing gas comprises the volatile propellant of the composition to be filled. Thus, for example, where the composition comprises a liquid product and a volatile propellant of the type disclosed above, the ambient air is displaced from the can by flushing with a charge of the propellant in vapor phase, and the composition is then filled into the can under pressure as before, the amount of volatile propellant in liquid phase that is filled being reduced by an amount substantially equal to the amount of propellant remaining in the flushed can at the start of the filling operation. With this procedure, the filled can contains only the liquid product and the propellant, and no excess pressure due to air is present in the filled can.

The above-described modification of the improved method may also be employed when using a flushing gas that is soluble in the liquid product constituent of the composition. Thus, where the composition includes an aqueous product, and the flushing gas is a water-soluble material, such as carbon dioxide or nitrous oxide, the partial evacuation step may be omitted and the flushing gas is substantially completely dissolved in the aqueous product constituent of the subsequently filled composition. With this procedure, the pressure in the filled container is substantially solely that due to the vapor pressure of the volatile propellant.

In carrying out the above-described modification of the method with the apparatus here illustrated, it is merely necessary to cut off the evacuating apparatus by closing the valve 26 and maintaining the piston valve in its raised position, so that the sole operation at the filling station L is the introduction of the liquid product and the volatile propellant in liquid phase into the can. I claim:

1. The method of packaging in a pressure-tight container a composition comprising a liquid product and a volatile propellant in liquid phase which comprises substantially completely displacing air from the container with a gas that is soluble in the composition, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pressure, and then closing the container, whereby the soluble gas in the container when the composition is introduced is substantially completely dissolved in the composition and the gas pressure in the head space of the container after filling is substantially solely that due to the vapor of the volatile propellant.

2. The method of packaging in a pressure-tight container a composition comprising a liquid product and a volatile propellant in liquid phase which comprises substantially completely displacing air from the container with the volatile propellant to be filled in vapor phase, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pressure, and then closing the container, whereby the gas pressure in the head space of the container after closing is substantially solely that due to the vapor of the volatile propellant.

3. The method of packaging in a pressure-tight container a composition comprising an aqueous phase which comprises substantially completely displacing air from the container with a water soluble gas, introducing the composition into the container under pressure to thereby raise the

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sure and then closing the container, whereby the water soluble gas in the container when the composition is introduced is substantially completely dissolved in the aqueous liquid product and the gas pressure in the head space of the container after filling is substantially solely that due to the vapor of the volatile propellant.

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4. The method of packaging in a pressure-tight container a composition comprising a liquid product and a volatile propellant in liquid phase which 10 liquid product and a volatile propellant in liquid comprises substantially completely displacing air from the container with a gas that is soluble in the composition, evacuating the container to an absolute pressure substantially below atmospheric pressure, introducing the composition into 15the container under pressure to thereby raise the pressure in the container above atmospheric pressure, and then closing the container, whereby the soluble gas remaining in the container when the composition is introduced is substantially 20 completely dissolved in the composition and the gas pressure in the head space of the container after filling is substantially solely that due to the vapor of the volatile propellant.

5. The method of packaging in a pressure- $_{25}$ pellant. tight container a composition comprising an aqueous liquid product and a volatile propellant in liquid phase which comprises substantially completely displacing air from the container with a water soluble gas, evacuating the container to an absolute pressure substantially below atmospheric pressure, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pressure and then closing the container, 35 whereby the water soluble gas remaining in the container when the composition is introduced is substantially completely dissolved in the aqueous liquid product and the gas pressure in the head space of the container after filling is substan-40 tially solely that due to the vapor of the volatile propellant.

6. The method of packaging in a pressure-tight container a composition comprising an aqueous liquid product and a volatile propellant in liquid phase which comprises substantially completely displacing air from the container with carbon dioxide, evacuating the container to an absolute pressure substantially below atmospheric pressure, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pressure, and then closing the container, whereby the carbon dioxide remaining in the container when the composition is introduced is substantially completely dissolved in the aqueous liquid product and the gas pressure in the head space of the container after filling is substantially solely that due to the vapor of the volatile propellant.

7. The method of packaging in a pressure-tight container a composition comprising an aqueous liquid product and a volatile propellant in liquid phase which comprises substantially completely displacing air from the container with nitrous oxide, evacuating the container to an absolute pressure substantially below atmospheric pressure, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pres-

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sure, and then closing the container, whereby the nitrous oxide remaining in the container when the composition is introduced is substantially completely dissolved in the aqueous liquid product and the gas pressure in the head space of the container after filling is substantially solely that due to the vapor of the volatile propellant.

8. In a method of packaging in a pressure-tight container a composition comprising an aqueous phase which comprises evacuating the container to a sub-atmospheric pressure and then introducing the composition under superatmospheric pressure and closing the container, the improvement which comprises substantially completely displacing air in the unfilled container at substantially atmospheric pressure with a gas soluble in the composition prior to evacuation of the container, whereby the gas remaining in the container when the composition is introduced is substantially completely dissolved in the composition and so does not increase the head space pressure in the filled container substantially above the pressure due to the vapor of the volatile pro-

9. In a method of packaging in a pressure-tight container a composition comprising an aqueous liquid product and a volatile propellant in liquid phase which comprises evacuating the container to a sub-atmospheric pressure and then introducing the composition under superatmospheric pressure and closing the container, the improvement which comprises substantially completely displacing air in the unfilled container at substantially atmospheric pressure with a water soluble gas prior to evacuation of the container. whereby the gas remaining in the container when the composition is introduced is substantially completely dissolved in the aqueous product and

so does not increase the head space pressure in the filled container substantially above the pressure due to the vapor of the volatile propellant.

10. The method of packaging in a pressuretight container a composition comprising a liquid

product and a volatile propellant in liquid phase 45 which comprises substantially completely displacing air from the container with the volatile propellant to be filled in vapor phase, evacuating the container to an absolute pressure substantially below atmospheric pressure, introducing the composition into the container under pressure to thereby raise the pressure in the container above atmospheric pressure, and then closing the container, whereby the gas pressure in the head space of the container after closing is substan-55 tially solely that due to the vapor of the volatile propellant.

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