The invention relates to a plastic preform suitable to be stretch blow molded in order to form an aerosol container (20) or to an injection stretch blow molded aerosol container (20), wherein the preform or container are made of a polymeric material that comprises a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least 0.7 dL/g.
AEROSOL PLASTIC CONTAINER MADE FROM AN ISOSORBIDE CONTAINING COPOLYESTER AND AEROSOL DISPENSER COMPRISING SAID AEROSOL PLASTIC CONTAINER

TECHNICAL FIELD

[0001] The present invention relates to a novel injection stretch blow molded aerosol container and to a novel preform suitable to be stretch blow molded in order to form an aerosol container. The invention also relates to a novel aerosol plastic dispenser for dispensing an aerosol or other comparably pressurized product.

PRIOR ART

[0002] Aerosol dispensers are well known in the art. More especially, aerosol dispensers comprise an aerosol container that contains an aerosol (or other comparably pressurized product), and that is fitted with a valve dispensing device for dispensing the aerosol. Aerosol dispensers comprising an aerosol container made of plastic are for example disclosed in US patent application US 2004/0149781 and in PCT application WO 2007/140407.

[0003] The term “aerosol” used herein encompasses both aerosol, literally, and also other liquids or flowable products that can be dispensed from pressurized containers in a manner comparable to aerolized products. Such other liquids or flowable products include but are not limited to foam or gel preparations or to liquid products delivered from pressurized containers but not necessarily in a pulverized form.

[0004] Examples of typical aerosol compositions can be notably but not exclusively insecticides, insect repellents, hairsprays, cosmetic sprays, air fresheners, cleaning preparation, shave preparations including foams and gels.

[0005] When a plastic container is used for making an aerosol container, the high internal pressure inside the plastic container can detrimentally lead to mechanical deformations of the plastic aerosol container, and even worse to the burst of the aerosol plastic container. This problem of mechanical deformations and burst of the aerosol plastic container under the effect of high internal pressure is even more critical when the container has an aesthetic and ergonomic shape with a con cave sidewall gripping portion, as the one depicted for example on FIG. 1 or 2 of US patent application US 2004/0149781 or for example on FIGS. 1, 5, 6B, 6C, 7A to 7F, 8A, 8D, 8E, 8G, 8H of PCT application WO 2007/140407. Said concave sidewall gripping portion is actually more sensible to mechanical deformation under high internal pressure compared with a container having a cylindrical body with a straight sidewall or with a convex sidewall.

[0006] This is the reason why an aerosol plastic container has to be pressure-resistant in order to withstand high internal pressure. More particularly, to date in the European community, plastic aerosol dispensers have to fulfill the technical requirements of standard FEA 621 of March 2007 meeting the provisions of the Aerosol Dispensers Directive 75/324/EEC of May 1975 and related to the measurement of internal pressure resistance of empty containers without valves.

[0007] PET (Polyethylene Terephthalate) is a well-known polyester that is widely used for making biaxially stretched containers, and more particularly Injection Stretched Blow Molded (ISBM) containers. ISBM grade PET has typically an Intrinsic Viscosity (IV) between 0.7 dL/g and 0.8 dL/g and a glass transition temperature (Tg) of about 75°C to 80°C.

[0008] Typically, polyesters, like for example PET homo or copolymer, can advantageously exhibit strain-induced crystallization upon substantial orientation, in a region above the Natural Stretch Ratio (NSR) of the polymer. It is well-known that for obtaining ISBM polyester containers having good mechanical properties, the biaxial stretching of the polyester must be sufficiently important to be in the strain-hardening region of the polyester, slightly beyond the NSR of the polymer.

[0009] The NSR of a polymer can be knowingly determined in a free-blowing experiment. Free-blowing of thermoplastics, in particular PET and PET copolymers, is a well known technique used to obtain empirical data on the stretching behavior of a particular resin formulation. The method of free blowing PET preforms is described in “Blow Molding Handbook”, edited by Donald V. Rosato, Dominick V. Rosato, Munich 1989. The term “free-blowing” means that a preform is blow-molded without using a mold. Free-blowing a bottle from a preform involves heating the preform to a temperature above its glass transition temperature and then expanding the preform outside of a mold so that it is free to expand without restriction until the onset of strain hardening. Strain hardening can be detected in a stress-strain curve as an upswing in stress following the flow plateau. To a large extent the strain hardening is associated with molecular ordering processes in the resin. If the blow pressure and heating of the preform is properly set for a given preform, it will continue to expand until all of the PET is oriented to the point that stretching will stop at about the natural stretch ratio, or slightly beyond.

[0010] Standard ISBM grade PET, even in the strain-hardening region, is however generally not suitable for making pressure-resistant PET aerosol containers that withstand high internal pressure, and more generally for making pressure-resistant ISBM aerosol containers that would fulfill the technical requirements of standard FEA 621 of March 2007.

[0011] The diol 1,4,3,6-di-hydroxy-D-sorbitol, referred to hereinafter as isosorbide, the structure of which is illustrated below, is readily made from renewable resources, such as sugars and starches, in particular natural starch extracted from maize, wheat, potatoes and peas. For example, isosorbide can be made from D-glucose by hydrogenation followed by acid-catalyzed dehydration.

[0012] Isosorbide has been already used as a monomer for incorporation into polyesters such as PET at low levels. The incorporation of isosorbide as comonomer in a copolyester knowingly reduces the intrinsic viscosity of the copolyester.

[0013] Isosorbide containing copolymers, and in particular Polyethylene Terephthalate containing Isosorbide (PETI) polymers, as well as their process of manufacturing by melt polymerization or by solvent polymerization are thus well-known in the art.
[0014] Isosorbide containing copolymers, and in particular Polyethylene Terephthalate containing Isosorbide (PETT) polymers, are used to date in many applications, and for example for making films or containers. In particular, Polyethylene Terephthalate containing Isosorbide (PETT) can be used for making hot-fillable containers that withstand high temperatures.

[0015] As clearly identified in the following publication, the incorporation of isosorbide as comonomer in a copolyester increases the glass transition temperature ($T_g$) of the copolyester (see FIG. 2 — Variation of $T_g$ versus mol % of Isosorbide), and has also an impact on the strain hardening characteristics of the polymer: “Properties and Strain Hardening Character of Polyethylene Terephthalate Containing Isosorbide”, Ramesh M. Kohil, Polymers Engineering and Science—2009 pages 544-553.

[0016] Although Isosorbide containing copolymers, and in particular Polyethylene Terephthalate containing Isosorbide (PETT) polymers can be used to date in many applications, one cannot find on the market ISBM aerosol containers made from an isosorbide containing copolyesters and in particular made from a PETT polymer.

[0017] There is consequently an unsatisfied need to propose ISBM pressure-resistant aerosol containers made from an isosorbide containing copolyester and in particular made from a PETT polymer.

OBJECTIVE OF THE INVENTION

[0018] A main objective of the invention is thus to propose a novel pressure-resistant aerosol plastic container made from an isosorbide containing copolyester, and in particular (but not only) made from a PETT polymer.

SUMMARY OF THE INVENTION

[0019] A first object of the invention is a plastic preform adapted to be stretch blow molded in order to form an aerosol container or an injection stretch blow molded aerosol container, said preform or aerosol container being made from a polymeric material that comprises a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least 0.7 dL/g.

[0020] The ISBM aerosol container of the invention exhibit very good mechanical properties, in particular can advantageously withstand high internal pressure.

[0021] Aerosol containers are generally containers of small volumes, typically not more than 750 ml. Consequently, when aerosol containers are made by using ISBM technology, only low stretch ratios can be practiced.

[0022] The incorporation of isosorbide as comonomer in a copolyester has a strong impact on the onset of the strain-hardening region of the copolyester, and more particularly increases the NSR of the copolyester. Otherwise stated, isosorbide containing copolyesters, and in particular Polyethylene Terephthalate containing Isosorbide (PETT) polymers, require a much higher elongation for reaching the strain-hardening region than a standard PET homopolymer.

[0023] As a result, prior to the invention, it was generally considered that the use of isosorbide containing copolymers, especially with high level of isosorbide, would eventually improve the thermal properties of the container because a higher $T_g$ would achieved, but would not be suitable for making pressure-resistant aerosol ISBM containers having the required mechanical properties to withstand the typical high internal pressure, especially for making pressure-resistant aerosol ISBM containers of small volume with low stretch ratios, because the NSR of the copolyester would be too high.

[0024] The invention removed this prejudice by increasing the intrinsic viscosity of the isosorbide containing copolyesters, for example by carrying out a Solid State Polymerization (SSP) of the copolyester for a period sufficient to achieve an IV of at least 0.7 dL/g. This increase of IV advantageously decreases the NSR of the copolyester and enables to obtain a copolyester combining a high level of isosorbide and a lower NSR and suitable for making pressure-resistant ISBM aerosol containers.

[0025] More particularly, the preform or ISBM aerosol container of the invention can have any one of the following optional characteristics:

[0026] The preform or container is essentially made from a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least 0.7 dL/g.

[0027] The copolyester is Polyethylene Terephthalate containing Isosorbide (PETT).

[0028] The copolyester includes at least 3 mole (%) of isosorbide as comonomer, and more particularly at least 5 mole (%) of isosorbide as comonomer.

[0029] The copolyester includes not more than 15 mole (%) of isosorbide as comonomer, and preferably not more than 8 mole (%) of isosorbide as comonomer.

[0030] The intrinsic viscosity of the copolyester is at least 0.8 dL/g, more particularly at least 0.9 dL/g.

[0031] The intrinsic viscosity of the copolyester is not more than 2.0 dL/g.

[0032] The injection stretch blow molded aerosol container has an axial stretch ratio ($S_a$) of not more than 3, and preferably not more than 2.55.

[0033] The injection stretch blow molded aerosol container has a radial stretch ratio ($S_r$) of not more than 2.5, and preferably of not more than 2.4.

[0034] The injection stretch blow molded aerosol container has an overall stretch ratio ($S$) of not more than 9, and preferably of not more than 7.

[0035] The injection stretch blow molded plastic container has a concave sidewall gripping portion.

[0036] The injection stretch blow molded plastic container has a volume of not more than 750 ml, and preferably of not more than 500 ml.

[0037] The invention also relates to an aerosol dispenser comprising the aforesaid injection stretch blow molded aerosol container and a valve dispensing device suitable for dispensing an aerosol contained in the aerosol container.

BRIEF DESCRIPTION OF THE DRAWINGS

[0038] Other technical characteristics and advantages of the invention will appear more clearly on reading the following detailed description which is made by way of non-exhaustive and non-limiting example, and with reference to the appended drawings, as follows:

[0039] FIG. 1 is a longitudinal cross-section view of preform suitable to be stretch blow molded in order to form a pressure-resistant aerosol plastic container.

[0040] FIG. 2 is a longitudinal cross-section view of an aerosol dispenser comprising an ISBM aerosol container that has been obtained by biaxially stretch blow molding the pre-
form of FIG. 1, and that is fitted with a valve dispensing device suitable for dispensing an aerosol contained in the aerosol container.

DETAILED DESCRIPTION

[0041] In reference to FIG. 2, the aerosol dispenser 2 comprises a pressure resistant aerosol container 20 which is knowingly hemispherically closed by a valve dispensing device 21. Said valve dispensing device 21 comprises a closure 210 that is covering the top opening 200 of the aerosol container 20, and that is sealingly attached to the neck 201 of the aerosol container 20. Said closure 210 includes a valve member 211 having an axially extending valve stem 212 which can be either depressed or tilted to release the aerosol contained within the container 20. The structure and functioning of the valve dispensing device 21 are well known in the art and will not be described in details. One skilled in the art can besides refer to the disclosure of US 2004/0149781.

[0042] Pursuant to the invention, the pressure resistant plastic aerosol container 20 is an injection stretch blow molded container.

[0043] FIG. 1 shows a plastic preform 1 suitable to be stretch blow molded in order to form a pressure-resistant aerosol plastic container

[0044] This preform 1 is made of a substantially tubular body of axial length \( L_1 \), which is closed at its bottom end and has a pouring opening at its upper end. More especially, said preform 1 comprises a neck portion 10 terminated by a pouring opening 100, a so-called gate portion 12 forming a closed bottom end, and a body portion 11 that is extending between said gate portion 12 and said neck portion 10. The neck portion 10 comprises a protruding neck support ring 101 of bigger diameter. The body portion 11 comprises a main cylindrical portion 110 of substantially constant wall thickness WT and an upper transition portion 111. The inner face of the main portion 110 can be also conical.

[0045] In this particular example of FIG. 1, the gate portion 12 is made of a convex portion having substantially a hemispherical shape, and terminated by a small central protruding injection point 20. The size of this injection point 20 corresponds to the size of the output orifice of the hot runner nozzle that is being used for injecting the plastic material in the mould. The shape of the gate portion 12 is not necessarily hemispherical, but the gate portion 12 can have any other shape, and in particular can be for example conical.

[0046] When this preform 1 is biaxially stretch blow molded in a mould, the neck portion 10 is used for maintaining the preform in the blowing mould, and is thus not stretched. The body portion 11 is biaxially stretched (in a longitudinal direction X and in a radial direction Y) in order to form the container body of higher volume. The gate portion 12 is also biaxially stretched in order to form typically the bottom base of the injection stretch blow molded container.

[0047] Within the scope of the invention a “one stage process” or a “two stages process” can be used. In the one stage process, the stretch-blow moulding step of the preform is performed in line immediately after the first injection step (preform injection). In the “two stages process”, the stretch-blow moulding step of the preform is postponed, and a reheating of the preform is performed prior to this stretch-blow moulding step.

[0048] The final shape and size of the container will depend of the blow mold that is being used and of the stretch ratios that are practiced. For example but not only, the preform 1 can thus be stretch blow molded in order to make the pressure-resistant aerosol container 20 of FIG. 2.

[0049] The invention is however not limited to the particular shape or dimensions of the aerosol container 20 of FIG. 2.

[0050] In particular, the base 202 (FIG. 2) of the aerosol container 20 is not necessarily spherical like the container depicted on FIG. 2, but can be of any shape. More especially, the base 202 of the aerosol container 20 can also be a base including an inwardsly-oriented central dome, also commonly called "champagne" base, or can be a "petri-lid" base like for example the base of the container of FIG. 8D of WO 2007/140407.

[0051] In the particular aerosol container 20 of FIG. 2, the sidewall of the container comprises a main central portion 203 which is concave and forms a kind of hyperboloid configuration, which provides a very ergonomic structure that can be easily handled by a user. In other variants, the sidewall of the container 20 can have any other shape, including straight wall portion, convex wall portions, etc. . .

[0052] Pursuant to the invention, in order to obtain a pressure-resistant injection stretch blow molded aerosol container 20 that can withstand high internal pressure, the polymeric material used for making the preform 1 or container 2 is a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least 0.7 dl/g, more preferably of at least 0.8 dl/g, and even more preferably of at least 0.9 dl/g.

[0053] For achieving said required minimum IV of at least 0.7 dl/g, the intrinsic viscosity of the Isosorbide containing copolyester has generally to be increased for example by carrying out a Solid State Polymerization (SSP) of the copolyester for a period sufficient to achieve the required minimum IV. This increase of IV enables favorably to lower the NSR of the isosorbide containing polyester and to at least partially compensate the increase of the NSR caused by the incorporation of isosorbide in the copolyester. The required IV level can however also be obtained directly, i.e. without a SSP post-treatment, by carrying out a suitable polymerization process.

[0054] More preferably, the isosorbide containing polyester comprises a copolyester that includes at least 5 mole % of isosorbide as comonomer.

[0055] More particularly, the isosorbide containing polyester comprises a copolyester that includes not more than 15 mole % of isosorbide as comonomer, and even more preferably not more than 8 mole % of isosorbide as comonomer.

[0056] The isosorbide containing copolyester may be formed by any method known in the art. Preferably, however, the polyester is formed by solvent or melt polymerization.

[0057] Preferably, the isosorbide containing copolyester is Polyethylene Terephthalate containing Isosorbide (PETI), although other polyesters are also suitable for practicing the invention.

[0058] Preferably, the isosorbide containing copolyester comprises terephthaloyl moiety; optionally, one or more other aromatic diacid moieties; ethylene glycol moieties; isosorbide moieties; and, optionally one or more other diol moieties.

[0059] More particularly, said terephthaloyl moieties can be derived from terephthalic acid or dimethyl terephthalate.

[0060] More particularly the isosorbide containing copolyester can further comprise diethylene glycol moieties.

[0061] Aforementioned one or more other diol moieties can be derived from aliphatic alkyleneglycols or branched aliphatic...
glycols having from 3-12 carbon atoms and having the empirical formula HO-CnH2n-OH, where n is an integer from 3-12; including branched diols such as 2,2-dimethyl-1,3-propanediol; cis or trans-1,4-cyclohexanediol and mixtures of the cis and trans isomers; triethylene glycol; 2,2-bis[4-(2-hydroxyethoxy)phenyl]propane; 1,1-bis[4(2-hydroxyethoxy)phenyl]hexane; 9,9-bis[4-(2-hydroxyethoxy)phenyl]fluorene; 1,4,1,4,3,6-triacyldromannitol; 1,4:1,4:3,6-dianhydrocoronitol; and 1,4-anhydroerythritol.

[0062] Preferably, but optionally, the number of terephthaloyl moieties in the polymer is in the range of about 25% to about 75 mole% (mole % of the total polymer).

[0063] In a preferred embodiment, ethylene glycol monomer units are present in amounts of about 5 mole % to about 49.75 mole %. The polymer may also contain diethylene glycol moieties. Depending on the method of manufacture, the amount of diethylene glycol moieties is for example in the range of about 0.0 mole % to about 25 mole %.

Experiments

[0064] Three different batches (Ref. A, Ref. B, Ref. C) of ISBM monolayer aerosol containers having similar shapes and dimensions, and more especially having a sidewall hyperboloid configuration similar to the container of FIG. 2, have been manufactured with three different polymeric compositions, by injecting preforms I having a weight of 35 g and by biaxially stretch blow molding said preforms into ISBM aerosol containers having a volume of 335 ml. The wall thickness (WT) of the main cylindrical body portion II of the preform I was about 4.5 mm (+/-5%).

[0065] The mold blowing step was performed with an axial stretch ratio Sx around 2.4 mm.

[0066] This axial stretch ratio (Sx) is knowingly defined in a standard way by formula:

\[ S_x = \frac{L}{l} \]

wherein: L is the container developed length (FIG. 2) and l is the preform neutral fibre developed length (FIG. 1).

[0067] The radial stretch ratio (Sr) was around 2.55 mm. This radial stretch ratio (Sr) is knowingly defined in a standard way by formula:

\[ S_r = \frac{D}{d} \]

wherein D is the maximum container outside diameter (see FIG. 2) and d is the maximum preform outside diameter Dp (FIG. 1) minus the wall thickness WT (d=Dp−WT).

[0068] The overall stretch ratio (S) of the aerosol containers was around 6. This Overall stretch ratio (S) is knowingly defined in a standard way by formula:

\[ S = S_x \times S_r \]

[0069] The first batch (Ref. A) of ISBM aerosol containers was made from Polycethylene Terephthalate containing Isosorbide (PET) [i.e. Poly(ethylene-co-isosorbide)terephthalate]. More particularly, said PET was containing 5.8 mole % of Isosorbide (5.8 mole % PET). This 5.8 mole % PET was obtained in a known way by melt polymerization. This 5.8 mole % PET issued from the melt polymerization was subjected to a SSP during a period of time sufficient to raise the IV of the Isosorbide containing copolyester up to about 0.95 dl/g.

[0070] The second batch (Ref. B) of ISBM aerosol containers was made from a PET resin of standard grade having an intrinsic viscosity of about 0.86 dl/g.

[0071] The third batch (Ref. C) of ISBM aerosol containers was made from a PET resin having an intrinsic viscosity of about 0.95 dl/g. Said PET resin was a copolymer PET commercialized by Artenius Tech Polymers under commercial reference “Artenius HOT”.

[0072] For all the three batches A, B and C, the intrinsic viscosity (IV) of the resin before injection was measured pursuant to following method based on the ISO 1628 standard:

[0073] 10 g of material is dried for 3 h under vacuum at 120°C.

[0074] 10 g of material is ground to a mesh size of 0.5 mm.

[0075] 0.5 g of ground material is weighed into a volumetric flask of 100 ml.

[0076] Solvent is added to dissolve the sample. The solvent is DCA (dichloroacetic acid).

[0077] The volumetric flask is heated and stirred until everything is dissolved.

[0078] The solution is stabilized at 25°C. and filled with solvent until a volume of exact 100 ml.

[0079] The solution is measured in a dedicated capillary viscometer and the IV is calculated according to the ISO 1628 standard, taking into account the solvent that is used.

[0080] The intrinsic viscosity (IV) of the preforms of the three batches was also measured by carrying out the aforesaid method with m-cresol as solvent. The results showed an IV drop due to a degradation of the polymer during the injection process. The IV of the preforms of the first batch (Ref. A) was around 0.8 dl/g. The IV of the preforms of the second batch (Ref. B) was around 0.73 dl/g. The IV of the preforms of the third batch (Ref. C) was around 0.78 dl/g.

[0081] For all the three batches A, B and C, the glass transition temperature (Tg) of the resins, was knowingly measured by Differential Scanning calorimetry (DSC) with an equipment DSC 821e from Mettler Toledo. The Tg of the 5.8 mole % PET (Ref. A) was around 89.6°C. The Tg of the PET resin of the second batch (Ref. B) was around 79.6°C. The Tg of the PET resin of the third batch (Ref. C) was around 79.5°C.

[0082] The following mechanical and thermal tests have been carried out on the three batches A, B and C of aerosol containers.

Hydraulic Test

[0083] The goal of this test is to evaluate the container dimensions stability after pressurizing the aerosol container. Test pressure needs to be 50% higher than the internal pressure in the container at 50°C.

[0084] Calculation for the pressure (P) used for the hydraulic test: The pressure at 50°C is defined, using the perfect gas law.

[0085] We take the assumption that we have no creep on the container, this means that the volume (V) at 20°C is the same as the volume (V) at 50°C. (which is more severe than reality).

\[ P = \frac{V \cdot T}{k \cdot T} \]
If V is constant, then: 

\[ P_{e_0} \cdot c_f / T_{e_0} \cdot c_f = P_{e_{50}} \cdot c_f / T_{50} \cdot c_f \]

Pressure = Absolute pressure

\[ T^* = \text{Absolu} T^* (\text{° K}) \]

\[ P_{e_{20}} \cdot c_f / P_{e_{30}} \cdot c_f / T_{e_{20}} \cdot c_f > 0.8 \text{ bar (abs)} / 0.32 \text{°C} \]

\[ K/293^* = 8.8 \text{ bar (abs)} / 7.82 \text{ bar (relative)} \]

Conclusion 7 bar at 20°C give a pressure of 7.82 bar at 50°C (considering no volume expansion)

For the hydraulic test, the internal pressure in the container at 50°C is thus 7.82 bar.

Hydraulic pressure = Pressure at 50°C + 5%/> 7.82 + 50% / 11.73 bar > Test pressure / 12 bar

The test is then performed as follows:

Take 10 containers ad random

Measure the container at the start: height, diameter shoulder-label-base area, base clearance, overflow volume.

Pressurize the container for 25 sec at 12 bar (= calculated pressure of container at 50°C + 50%) using the SOMEX Delta 3000 PET bottle Pressure Tester.

Re-measure the container on an identical way as measured at the start

Evaluate the container visually. A slight symmetrical distortion shall be allowed provided that the container passes the burst test.

Material Resistance to Temperature—Drop Test

The goal of this test is to evaluate the container when it is dropped from a height of 1.8 m to a concrete floor and this at different temperatures. Ensure that the orientation, of the test container at drop is statistically random, but that direct impact on the valve or valve closure is avoided. Aerosol containers must be designed that it shall not break or leak.

Aerosol containers needs to be tested at 3 different temperature conditions:

- 18°C for at least 24h
- Room temperature 20-22°C for at least 1 h
- 55°C for at least 6 h (dry air)

Take at random 25 aerosol container for each group of temperature testing

Filling of the container 85% of the overflow volume with water. For the containers that need to be tested at -18°C fill the container with a mixture of 50/50 water/antifreeze

Closing of the container with metal crimp valve

Pressurize the container with compressed air at 8.0 bar relative. (this is the calculated pressure at 50°C.)

Condition the 25 containers for the defined time at the 3 different temperatures.

Drop the containers random on the concrete floor from a height of 1.8 m directly after removing them from the climate chamber

Evaluate the aerosol containers after drop: no leak or break is allowed on the 3 different sets of containers.

Burst Test

The goal of this test is to evaluate the ability of an aerosol container to withstand to a certain internal pressure. Pressure at which the container is bursting needs to be minimum 20% higher than the test pressure. Equipment used for this test is the Delta 3000 PET bottle Pressure Tester from SOMEX. Minimum burst pressure calculation: Test pressure of 12 bar (relative)+20% = 14.4 bar (relative)

Take 10 aerosol containers ad random

Container is filled brinful with water, and the initial pressure on the container is 4 bar for a hold time of 13 sec. After this the pressure is built up with a ramp of 0.69 bar/sec until the container bursts.

Burst pressure for all the containers needs to be above 14.4 bar.

Material Resistance to Temperature—Hot Air

The goal of this test is to indicate the temperature where the deformation of the container is induced. Temperature for this test is 7°C, lower than Tg with a max. test temperature of 75°C and min temperature of 65°C. It is allowed that the containers deform, but without breakage or leakage creating a hazardous environment.

Take 25 aerosol containers at random

Fill the containers with water up to a headspace volume of 15% of the overflow volume of the container

Close the container with metal crimp valve

Pressurize the container with compressed air at 8.0 bar (relative)

Condition the 25 containers for the min. 5 H at 75°C dry air in the climate cupboard.

Evaluate the containers on leakage and breakage when the hot air test is finished

Top Load

The goal of this test is to evaluate the aerosol containers resistance to a vertical load before its first deformation. Equipment used for this test is the INSTRON 3366 Top load tester with a load cell of 5000N. The result is the maximum compressive load (in kgf) a container can withstand before it loses 1% of the compressive load applied. Test is done with a speed of 50 mm/min.

Take at random 10 aerosol containers

Test each container separately

Note the place of deformation at the end of the test and the max compressive load (end of test: 1% rate of compressive load)

Accelerated Stress Cracking

The objective of the accelerated stress cracking is to simulate stress experienced by containers during pressure filling, shipping and storage.

The test is performed as follows:

Take the containers (normal sample size: 10) at random from the samples to be tested.

Fill each container with carbonated water at the requested volume of CO₂ and cap it.

Pour 0.2% NaOH solution into cut bases.

Complete

Submerge the base of the bottles in the caustic solution into the cut bases and start the chronometer

Measure the immersion time until the bottle base bursts or leaks using the chronometer (stop the experiment in case the bottle does not show any failure after 30 minutes)

The permeation of the aerosol containers of the first batch (Ref. A) has been also measured pursuant to the following permeation test.
Permeation

[0132] The goal of this test is to measure the Oxygen transmission rate of the aerosol container. Equipment used to determine the permeation is the MOCON OXtran 2/20 that uses a Coulometric Sensor (Coulox) to determine the O2 concentration. Test method used is derived from the ASTM D 3985 and the ASTM F 1307. The Aerosol container is mounted on a metal fixture and flushed with N2 to purge the air out of the bottle. As the outside of the bottle is in contact with ambient air (20.9% O2), O2 permeates through the bottle wall and is transported with the N2 flow to the Coulox Sensor.

[0133] The test is performed as follows:

[0134] Take 2 aerosol containers at random.

[0135] Glue the containers on the metal fixture with the 2 component glue.

[0136] Condition the bottle for 50 h with N2.

[0137] Measurement is finished when the O2 concentration is stable (less than 1% difference with concentration of 10 h before).

[0138] Result is given in cc/pack/day—22° C.—atmospheric pressure—ambient air.

[0139] ppm/day or ppm/year is calculated.

Test Results

Hydraulic Test

[0140] The results of the hydraulic test are summarized in tables IA, IB and IC.

<table>
<thead>
<tr>
<th>TABLE IA</th>
<th>Hydraulic Test- Ref. A.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial</td>
</tr>
<tr>
<td>total height max (mm)</td>
<td>181.18</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) P/L</td>
<td>56.46</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) 90°</td>
<td>56.77</td>
</tr>
<tr>
<td>label diameter (80.0 mm) P/L</td>
<td>47.77</td>
</tr>
<tr>
<td>label diameter (80.0 mm) 90°</td>
<td>47.79</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) P/L</td>
<td>56.92</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) 90°</td>
<td>56.62</td>
</tr>
<tr>
<td>base clearance (mm)</td>
<td>10.45</td>
</tr>
<tr>
<td>overflow volume (ml)</td>
<td>333.92</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TABLE IB</th>
<th>Hydraulic Test- Ref. B.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial</td>
</tr>
<tr>
<td>total height max (mm)</td>
<td>181.11</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) P/L</td>
<td>56.44</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) 90°</td>
<td>56.55</td>
</tr>
<tr>
<td>label diameter (80.0 mm) P/L</td>
<td>47.88</td>
</tr>
<tr>
<td>label diameter (80.0 mm) 90°</td>
<td>47.55</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) P/L</td>
<td>57.04</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) 90°</td>
<td>56.63</td>
</tr>
<tr>
<td>base clearance (mm)</td>
<td>10.19</td>
</tr>
<tr>
<td>overflow volume (ml)</td>
<td>332.48</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TABLE IC</th>
<th>Hydraulic Test- Ref. C.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial</td>
</tr>
<tr>
<td>total height max (mm)</td>
<td>181.13</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) P/L</td>
<td>56.55</td>
</tr>
<tr>
<td>shoulder diameter (135.0 mm) 90°</td>
<td>56.53</td>
</tr>
<tr>
<td>label diameter (80.0 mm) P/L</td>
<td>47.97</td>
</tr>
<tr>
<td>label diameter (80.0 mm) 90°</td>
<td>47.40</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) P/L</td>
<td>57.03</td>
</tr>
<tr>
<td>heel diameter (20.0 mm) 90°</td>
<td>56.49</td>
</tr>
<tr>
<td>base clearance (mm)</td>
<td>10.04</td>
</tr>
<tr>
<td>overflow volume (ml)</td>
<td>332.70</td>
</tr>
</tbody>
</table>

Material Resistance to Temperature—Drop Test

[0142] All the aerosol containers of the first batch (Ref A) successfully passed the drop test for the three temperatures conditions i.e.,

-18° C. for at least 24 h
55° C. for at least 6 h (dry air)

Burst Test

[0146] All the aerosol containers of the three batches (Ref A, B and C) successfully passed the burst test. Burst pressure for all the aerosol containers was above 14.4 bar.

Material Resistance to Temperature—Hot air

[0147] All the aerosol containers of the first batch (Ref A) successfully passed the test. The aerosol containers of the first batch (Ref A) did not exhibit any leakage after 5 h at 75° C.

[0148] In return, the aerosol containers of the second batch (Ref B) and third batch (Ref C) did not successfully pass the test, and were leaking after 5 h at 75° C.

Top Load

[0149] The results of the hydraulic test are summarized in tables IIA, IIB and IIC.

<table>
<thead>
<tr>
<th>TABLE IIA</th>
<th>To load Test- Ref. A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force (Kg)</td>
<td>Average</td>
</tr>
<tr>
<td>70.7</td>
<td>4.96</td>
</tr>
<tr>
<td>Location of failure</td>
<td>Finish pushed into the shoulder</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TABLE IIB</th>
<th>To load Test- Ref. B.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force (Kg)</td>
<td>Average</td>
</tr>
<tr>
<td>74.8</td>
<td>1.05</td>
</tr>
<tr>
<td>Location of failure</td>
<td>Finish pushed into the shoulder</td>
</tr>
</tbody>
</table>
TABLE IIC

<table>
<thead>
<tr>
<th>Location of failure</th>
<th>Average</th>
<th>Standard Deviation</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force (Kg)</td>
<td>83.4</td>
<td>3.22</td>
<td>77.6</td>
<td>88.4</td>
</tr>
<tr>
<td>Finish pushed into the shoulder or deformation of the body</td>
<td>83.4</td>
<td>3.22</td>
<td>77.6</td>
<td>88.4</td>
</tr>
</tbody>
</table>

Stress Cracking

[0150] All the aerosol containers of the three batches A, B and C successfully passed the stress cracking test. No burst and no crack appears after 30 minutes.

Permeation

[0151] The result of the permeation of the aerosol containers of the first batch (Ref. A) are summarized in table III.

TABLE III

<table>
<thead>
<tr>
<th>Permeation- Ref. A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brimfull volume (ml)</td>
</tr>
<tr>
<td>(cm³/d)</td>
</tr>
<tr>
<td>ppm/day</td>
</tr>
<tr>
<td>ppm/year</td>
</tr>
</tbody>
</table>

[0152] The invention is not limited to an injection stretch blow molded aerosol container made from a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least about 0.7 dL/g and another polymer, in particular another polyester.

What is claimed is:

1. - 15. (canceled)

16. A plastic preform adapted to be stretch blow molded to form an injection stretch blow molded container, the preform comprising:

a polymeric material that comprises a copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least about 0.7 dL/g.

17. The preform of claim 16, wherein the preform is comprised of copolyester including at least 1 mole % of isosorbide as comonomer and having an intrinsic viscosity of at least about 0.7 dL/g.

18. The preform of claim 16, wherein the copolyester is polyethylene terephthalate containing isosorbide (PET).