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(54) CONTAINER AND METHOD FOR MAINTAINING STABILITY OF GAS MIXTURES

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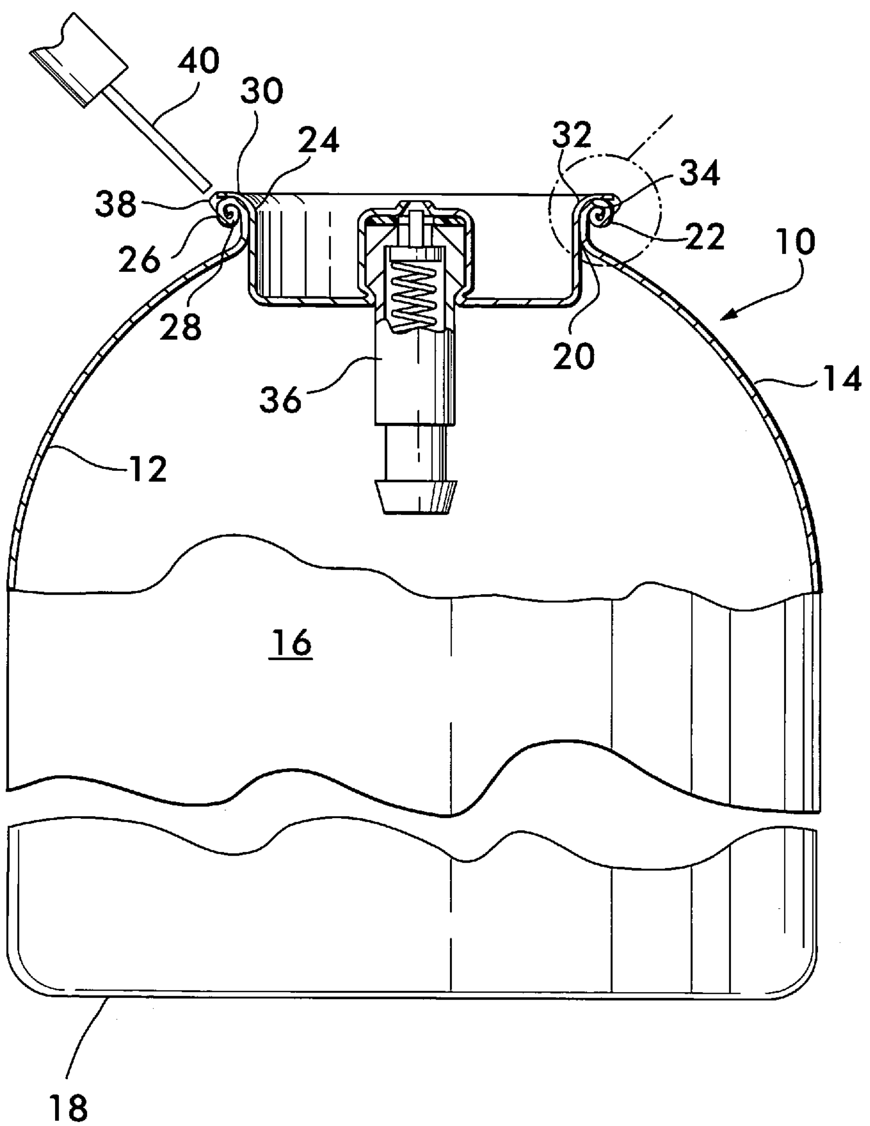
(57) ABSTRACT

A container for holding a single constituent gas or a mixture of constituent gases is disclosed. The container has a wall structure defining an internal space. An opening in the wall structure permits access to the internal space. A closure is positioned over the opening. A seal attaches the closure to the wall structure maintains the single constituent gas or the constituents of the gas mixture at a predetermined concentration over a predetermined time period. A valve is mounted on the closure for permitting controlled release of the mixture. A method of maintaining gas concentration stability by forming a seal between the closure and the wall structure is also disclosed.



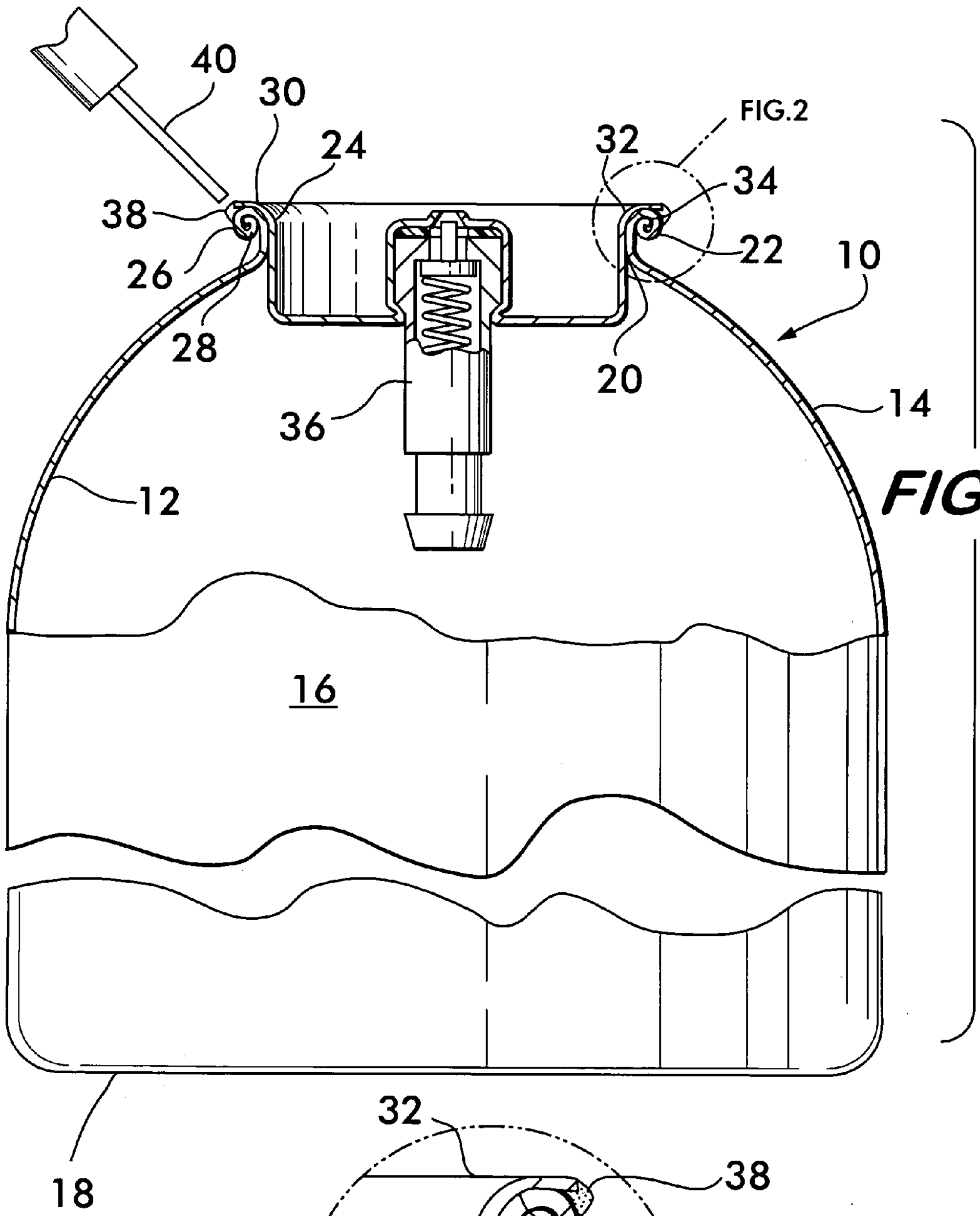


FIG. 1

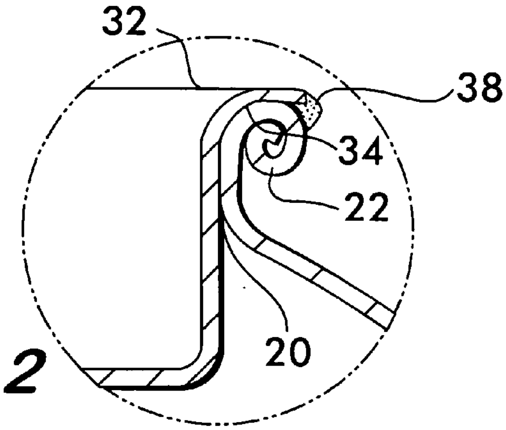


FIG. 2

CONTAINER AND METHOD FOR MAINTAINING STABILITY OF GAS MIXTURES

FIELD OF THE INVENTION

[0001] This invention concerns a method and a container adapted to maintain a single gas or constituent gases of a gas mixture within a predetermined concentration range over a predetermined time period.

BACKGROUND OF THE INVENTION

[0002] Gas mixtures having precise concentrations of constituent gases are used extensively in many industrial and laboratory processes. For example, mass spectrometers for identifying chemical compounds use a gas mixture comprising bromopentafluorobenzene, trifluoromethyl benzene and VOC-free nitrogen for calibration purposes. These constituent gases are present in the mixture at predetermined concentrations appropriate for the calibration purposes. Proper operation of the mass spectrometer depends upon its accurate calibration, which, in turn, depends upon the accuracy of the relative concentration of the constituent gases in the calibration gas mixture.

[0003] When gas mixtures are held under pressure in containers, the relative concentration of the constituent gases may change over time, rendering the mixture useless for its intended purpose. The mixture is said to suffer from "instability", which is defined as the tendency of the constituent gases to change in concentration (either rise or fall) over time. Long term instability of a gas mixture occurs when the concentration of one or more constituent gases in the mixture changes after several (4-6) months, while short term instability is said to occur when the concentration of the constituent gases changes over a period of less than three months.

[0004] Instability is an especially acute problem for gas mixtures stored at relatively low pressures and having relatively low concentrations of reactive components. For example, the aforementioned gas mixture for mass spectrometer calibration contains 50 ppmv of bromopentafluorobenzene and 100 ppmv of trifluoromethyl benzene in 5 liters of VOC-free nitrogen at 700 kPa at 21° C. In view of the small amount of the active constituent gases in the mixture, any characteristic of the container that affects the concentration of these constituents will have a significant effect on both the long and short term stability of the mixture.

[0005] The container characteristics which have the greatest effect on stability include adsorption, absorption and leakage. Practical containers typically comprise a pressure vessel, for example, a thin-walled aluminum cylinder, with an opening that receives a cap having a valve. For low pressure applications, the cap is crimped or swaged onto an outwardly curled lip that surrounds the opening. An elastomeric gasket, such as an O-ring or washer, is positioned between the cap and the lip to provide a fluid tight seal. The valve may be spring biased, and provides for controlled filling and discharge of the gas mixture.

[0006] Gas leakage occurs between the valve cap and the container lip because the swaging or crimping process does not form a continuous, fluid tight connection between the cap and the lip. The use of the gasket between the cap and the lip is intended to provide a fluid tight seal, but leaks can still occur between the gasket and the lip or the valve cap because it is not always possible to control the surface finish of the vessel or the cap, and the gasket cannot conform and block the small surface irregularities that provide unacceptable leakage paths from the container.

[0007] A leaky seal will not only allow gas to escape (thereby changing the relative concentration of the constituents), it may also allow contaminant gases, such as oxygen, to diffuse into the vessel against the gas pressure. The oxygen combines with the reactive components, effectively removing them from the mixture and changing the constituent concentration.

[0008] Even when the leakage rate is acceptable (i.e., the gas mixture remains stable within acceptable limits), the use of elastomeric gaskets can cause instability by absorbing or adsorbing the more reactive constituent gases, effectively removing them from the mixture and thereby changing the relative concentration of the components. It is clear that the effects of leakage and absorption or adsorption will have a proportionally greater effect for smaller concentrations of the constituent gases.

[0009] In addition to containers of gas mixtures, containers having a single gas constituent, for example, pure nitrogen for purging or calibration purposes, may also suffer from instability. In the case of a single gas constituent, the diffusion of contaminants into the container, such as oxygen or moisture, must be mitigated so that the concentration of the impurities does not increase.

[0010] There is clearly a need for a container and a method of maintaining the stability of gas mixtures as well as single constituent gases, and especially for gas mixtures stored at relatively low pressures (up to 18 bar) and at relatively small concentrations (in the parts per million by volume range).

SUMMARY OF THE INVENTION

[0011] The invention concerns a container for holding a mixture of a plurality of constituent gases. The gases have respective predetermined concentrations within the mixture. The container comprises a vessel having a wall structure defining an internal space for containing the gas mixture. The wall structure has an opening therein for access to the internal space. A closure is positioned in overlying relation with the opening. A seal is positioned between the closure and the wall structure for attaching the closure to the wall structure so as to maintain a first one of the gases at a concentration within a range of about +1% to about −5% of the predetermined concentration of the first gas over about a one month period. A valve is mounted on the closure. The valve is openable and closable for permitting controlled release of the gas mixture from the vessel.

[0012] The first gas may be maintained at a concentration within a range of about +1% to about −7% of the predetermined concentration of the first gas over about a one month period. The first gas may be maintained at a concentration within a range of about +1% to about −5% of the predetermined concentration of the first gas over about a one month period. A second one of the gases may be maintained at a concentration within a range of about +3% to about −15% of the predetermined concentration of the second gas over about a one month period. The second gas may be maintained at a concentration within a range of about +3% to about −30% of the predetermined concentration of the second gas over about a one month period. A third one of the gases may be maintained at a concentration within a range of about +3% to about −15% of the predetermined concentration of the third gas over about a one month period. The third gas may be maintained at a concentration within a range of about +3% to about −30% of the predetermined concentration of the third gas over about a one month period.

[0013] Preferably, the seal comprises a weld integrally joining the closure to the wall structure. The weld is preferably formed by means of an electron beam.

[0014] The invention further encompasses a container for holding a mixture of a plurality of constituent gases. The gases have respective predetermined concentrations within the mixture. The container is adapted to maintain the concentrations of the gases within a predetermined concentration range over a predetermined time period. The container comprises a vessel having a wall structure defining an internal space for containing the gas. The wall structure comprises a metal having the property of being inert with respect to the constituent gases and has an opening therein for access to the internal space. A closure is positioned in overlying relation with the opening. A seal for attaching the closure to the wall structure is positioned between the closure and the wall structure. The closure and the seal have the same properties of being inert to the constituent gases as the wall structure. A valve is mounted on the closure. The valve is openable and closable for permitting controlled release of the gas mixture from the vessel.

[0015] The invention also includes container for holding a single constituent gas. The single constituent gas has a predetermined concentration of impurities upon filling. The container comprises a vessel having a wall structure defining an internal space for containing the gas. The wall structure has an opening therein for access to said internal space. A closure is positioned in overlying relation with the opening. A seal for attaching the closure to the wall structure so as to maintain the impurities at a concentration within a range of about +1% to about −1% of the predetermined concentration of the impurities upon filling over about a one month period. A valve is mounted on the closure. The valve is openable and closable for permitting controlled release of the gas mixture from the vessel.

[0016] The invention also includes a method of maintaining gas concentration stability among constituent gases of a gas mixture within a predetermined concentration variation range and over a predetermined time period. The method comprises the steps of:

[0017] (a) providing a vessel for holding the gas mixture under pressure, the vessel having an opening therein;

[0018] (b) providing a closure adapted to engage the vessel in overlying relation with the opening, the closure having a valve mounted thereon for permitting controlled filling and release of the gas mixture to and from the vessel;

[0019] (c) forming a seal between the closure and the vessel, the seal, the closure and the vessel having the property of being inert with respect to the constituent gases, the seal providing a fluid tight joint between the closure and the vessel that maintains a first one of the gases at a concentration of about +1% to about −5% of a predetermined concentration of the first gas over about a one month period.

BRIEF DESCRIPTION OF THE DRAWINGS

[0020] FIG. **1** is a longitudinal sectional view of a container for stable storage of gas mixtures and single constituent gases according to the invention; and

[0021] FIG. **2** shows a portion of FIG. **1** designated within a broken circle and on an enlarged scale.

DETAILED DESCRIPTION OF THE EMBODIMENTS

[0022] FIG. **1** shows a container **10** for holding single constituent gases or gas mixtures comprising a plurality of constituent gases having predetermined concentrations within the mixture. The container is adapted to maintain the concentrations of the gases within a predetermined concentration range over a predetermined time frame and thereby provide for the stable storage of the gas mixtures, both long and short term.

[0023] Container **10** comprises a pressure vessel **12**, preferably formed of aluminum and having a thin sidewall structure **14** that surrounds an internal space **16**. Aluminum is a preferred material because it is self-passivating and inert, and will not react with the constituent gases and change their concentration. Vessel **12** is preferably cylindrical in cross section and has a bottom **18** at one end and an opening **20** at the opposite end for access to the internal space **16**.

[0024] A rim **22**, best shown in FIG. **2**, surrounds opening **20**, the rim projecting outwardly away from the central space. The rim provides a mounting feature for a closure **24**, and preferably comprises a curved lip **26** (see FIG. **1**). Lip **26** is formed by outwardly turning the sidewall **14** around the perimeter of the opening **20**. Turning the sidewall to form the lip results in a substantially spiral cross section **28** and provides a rim **22** having significant bending stiffness and strength, thereby reinforcing an inherently weak part of the vessel.

[0025] Closure **24** is preferably a circular aluminum cap **30** positioned in overlying relation with the opening **20**. Aluminum is preferred for the closure because it is advantageous that the closure also have the property of being inert with respect to the constituent gases. Cap **30** has a circumferential flange **32** adapted to engage the rim **22**. Preferably, flange **32** has a curvature **34** that is substantially matched to the curvature of lip **26**. The curvature of the flange allows the closure to center itself within the opening **20**.

[0026] Closure **24** also includes a valve **36** mounted on the cap **30**. Valve **36** is preferably a spring biased valve and provides for the controlled filling and release of gas from the internal space **16**.

[0027] Closure **24** is welded to the lip **26** by a weld **38** extending between the flange **32** and the lip. The weld is preferably formed by an electron beam **40** applied to the interface between the flange and the lip while rotating the vessel around its longitudinal axis within an evacuated chamber (not shown). Electron beam welding is preferred because the technique allows for significant heat energy to be precisely concentrated over a very small area with great accuracy, thereby allowing the weld **38** to be formed joining the closure to the vessel across the relatively small target that is the flange **32**. Excess heat is avoided, thereby protecting the polymer components of valve **36** from damage. If more thermal protection is required, a heat sink (not shown) may be attached to the valve during welding.

[0028] The weld **38** thus formed provides a substantially continuous fused metal seal between the closure and the vessel which does not permit any significant leakage or diffusion, thereby promoting stability of the gas mixture. The weld **38**, being the same material as the closure and the vessel also has the property of being inert with respect to the constituent gases. Use of the continuous weld as a seal obviates the need for a gasket, thereby eliminating a source of absorption or adsorption that can adversely affect constituent gas concentration.

[0029] Gas containers according to the invention have been tested for stability of the mixtures. Specifically, containers having a mixture of 50 ppmv bromopentafluorobenzene, 100 ppmv trifluoromethyl benzene in 5 liters of VOC-free nitrogen at 700 kPa at 21° C. were analyzed for stability over time. It was found that the concentration of trifluoromethyl benzene varied between about +1% and about −5% over a three month period, and between about +1% and about −7% over a six month period. In the analysis, the concentration of bromopentafluorobenzene was found to vary between about +3% to about −15% over a three month period and between about +3% and about −30% over a six month period. These results show improved stability over prior art containers that use elastomeric seals. In view of such test results, it is expected that the stability of gases, with as many as three or more constituents, should be readily achievable within the aforementioned concentration levels and within about a one month time period.

[0030] When containers according to the invention are used to hold single constituent gases, for example, pure nitrogen, it is recognized that there will be some impurities present in the gas upon filling of the container. For stability of such single constituent gases, it is preferable that the concentration of total impurities reported in the constituent gas on the day of fill does not change by more than +/−1% over a period of about one month. For example, for a container of pure nitrogen, the oxygen content must not change, that is, increase or decrease, by 2 ppm or greater for the gas constituent to be considered stable. Similarly, instability occurs when concentrations of the following impurities change by the following amounts or more: total hydrocarbons +/−0.05 ppm, carbon monoxide +/−0.1 ppm; carbon dioxide +/−0.3 ppm; and water vapor +/−2 ppm.

What is claimed is:

1. A container for holding a mixture of a plurality of constituent gases, said gases having respective predetermined concentrations within said mixture, said container comprising:

a vessel having a wall structure defining an internal space for containing said gas mixture, said wall structure having an opening therein for access to said internal space;

a closure positioned in overlying relation with said opening, a seal for attaching said closure to said wall structure so as to maintain a first one of said gases at a concentration within a range of about +1% to about −7% of said predetermined concentration of said first gas over about a one month period; and

a valve mounted on said closure, said valve being openable and closable for permitting controlled release of said gas mixture from said vessel.

2. A container according to claim **1**, wherein said first gas is maintained at a concentration within a range of about +1% to about −5% of said predetermined concentration of said first gas over about a one month period.

3. A container according to claim **1**, wherein a second one of said gases is maintained at a concentration within a range of about +3% to about −15% of said predetermined concentration of said second gas over about a one month period.

4. A container according to claim **1**, wherein said second gas is maintained at a concentration within a range of about +3% to about −30% of said predetermined concentration of said second gas over about a one month period.

5. A container according to claim **1**, wherein a third one of said gases is maintained at a concentration within a range of about +3% to about −15% of said predetermined concentration of said third gas over about a one month period.

6. A container according to claim **1**, wherein said third gas is maintained at a concentration within a range of about +3% to about −30% of said predetermined concentration of said third gas over about a one month period.

7. A container according to claim **1**, wherein said seal comprises a weld integrally joining said closure to said wall structure.

8. A container according to claim **7**, wherein said weld is formed by means of an electron beam.

9. A container for holding a mixture of a plurality of constituent gases, said gases having respective predetermined concentrations within said mixture, said container adapted to maintain the concentrations of said gases within a predetermined concentration range over a predetermined time period, said container comprising:

a vessel having a wall structure defining an internal space for containing said gas, said wall structure comprising a metal having the property of being inert with respect to the constituent gases and having an opening therein for access to said internal space;

a closure positioned in overlying relation with said opening, a seal for attaching said closure to said wall structure, said closure and said seal having the same properties of being inert to the constituent gases as said wall structure; and

a valve mounted on said closure, said valve being openable and closable for permitting controlled release of said gas mixture from said vessel.

10. A container according to claim **9**, wherein said seal comprises a weld integrally joining said closure to said wall structure.

11. A container according to claim **10**, wherein said weld maintains a first one of said gases at a concentration within a range of about +1% to about −5% of said predetermined concentration of said first gas over about a one month period.

12. A container according to claim **10**, wherein said weld maintains a first one of said gases at a concentration within a range of about +1% to about −7% of said predetermined concentration of said first gas over about a one month period.

13. A container according to claim **11**, wherein said weld maintains a second one of said gases at a concentration within a range of about +3% to about −15% of said predetermined concentration of said second gas over about a one month period.

14. A container according to claim **11**, wherein said weld maintains a second one of said gases at a concentration within a range of about +3% to about −30% of said predetermined concentration of said second gas over about a one month period.

15. A container according to claim **11**, wherein said weld maintains a third one of said gases at a concentration within a range of about +3% to about −15% of said predetermined concentration of said third gas over about a one month period.

16. A container according to claim **11**, wherein said weld maintains a third one of said gases at a concentration within a range of about +3% to about −30% of said predetermined concentration of said third gas over about a one month period.

17. A container according to claim **10**, wherein said weld is formed by means of an electron beam.

18. A container according to claim **10**, wherein said vessel comprises a thin-walled aluminum cylinder, said opening being positioned at one end thereof.

19. A container according to claim **18**, further comprising an outwardly turned lip surrounding said opening, said closure comprising a flange extending around its perimeter, said weld integrally joining said flange and said lip.

20. A container according to claim **19**, wherein said flange comprises a perimeter having a curvature matched to the curvature of said lip.

21. A container according to claim **10**, wherein said weld maintains a more reactive one of said gases at a concentration within a range of about +1% to about 5% of said predetermined concentration of said more reactive gas over about a one month period.

22. A container according to claim **10**, wherein said weld maintains a more reactive one of said gases at a concentration within a range of about +1% to about −7% of said predetermined concentration of said first gas over about a one month period.

23. A container according to claim **21**, wherein said weld maintains a less reactive one of said gases at a concentration within a range of about +3% to about −15% of said predetermined concentration of said less reactive gas over about a one month period.

24. A container according to claim **21**, wherein said weld maintains a less reactive one of said gases at a concentration within a range of about +3% to about −30% of said predetermined concentration of said less reactive gas over about a one month period.

25. A container for holding a single constituent gas, said gas having a predetermined concentration of impurities upon filling, said container comprising:

a vessel having a wall structure defining an internal space for containing said gas, said wall structure having an opening therein for access to said internal space;

a closure positioned in overlying relation with said opening, a seal for attaching said closure to said wall structure so as to maintain said impurities at a concentration within a range of about +1% to about −1% of said predetermined concentration of said impurities upon filling over about a one month period; and

a valve mounted on said closure, said valve being openable and closable for permitting controlled release of said gas mixture from said vessel.

26. A method of maintaining gas concentration stability among constituent gases of a gas mixture within a predetermined concentration variation range and over a predetermined time period, said method comprising the steps of:

providing a vessel for holding said gas mixture under pressure, said vessel having an opening therein;

providing a closure adapted to engage said vessel in overlying relation with said opening, said closure having a valve mounted thereon for permitting controlled filling and release of said gas mixture to and from said vessel; and

forming a seal between said closure and said vessel, said seal, said closure and said vessel having the property of being inert with respect to the constituent gases, said seal providing a fluid tight joint between said closure and said vessel that maintains a first one of said gases at a concentration of about +1% to about −5% of a predetermined concentration of said first gas over about a one month period.

27. A method according to claim **26**, further comprising the step of forming a seal that maintains said first one of said gases at a concentration within a concentration of about +1% to about −7% of said predetermined concentration of said first gas over about a one month period.

28. A method according to claim **26**, further comprising the step of forming a seal that maintains a second one of said gases at a concentration within a range of about +3% to about −15% of said predetermined concentration of said second gas over about a one month period.

29. A method according to claim **26**, further comprising the step of forming a seal that maintains a second one of said gases at a concentration of about +3% to about −30% of said predetermined concentration of said second gas over about a one month period.

30. A method according to claim **26**, further comprising the step of forming a seal that maintains a third one of said gases at a concentration within a range of about +3% to about −15% of said predetermined concentration of said third gas over about a one month period.

31. A method according to claim **26**, further comprising the step of forming a seal that maintains a third one of said gases at a concentration of about +3% to about −30% of said predetermined concentration of said third gas over about a one month period.

32. A method according to claim **26**, wherein said seal is formed by the step of electron-beam welding said closure to said vessel.

33. A method according to claim **26**, further comprising the step of filling said container with said gas mixture.

34. A method according to claim **33**, wherein said filing step comprises filling said container with a gas mixture comprising bromopentafluorobenzene, trifluoromethyl benzene and nitrogen.

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