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United States Patent	12392696
Kind Code	B2
Date of Patent	August 19, 2025
Inventor(s)	Markle; Doug

Gas phase standard preparation device and method of use

Abstract

A gas phase standard preparation device and method of use for creating a final reference material is disclosed. The final reference material is used to quantitate certain gaseous compounds in collected air samples. This is accomplished by providing a sealed ampule tube with an ampule containing reference material that is eventually breached by the gas preparation device. Once the ampule is breached, the reference material within the ampule is blended with a diluent gas and forced into a reference material cylinder. The concentration of the final reference material within the reference material cylinder can then be verified for use in testing collected air samples. Accordingly, this device allows for creating a custom final reference material onsite in a laboratory without the need for shipping in pre-made compressed gas cylinders for air quality testing.

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Family ID: **1000008767878**

Appl. No.: **17/839434**

Filed: **June 13, 2022**

Prior Publication Data

Document Identifier	Publication Date
US 20220307954 A1	Sep. 29, 2022

Related U.S. Application Data

continuation-in-part parent-doc US 15999506 20180820 US 11360066 20220614 child-doc US 17839434

Publication Classification

Int. Cl.: **G01N1/44** (20060101)

U.S. Cl.:

CPC **G01N1/44** (20130101);

Field of Classification Search

USPC: None

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Background/Summary

CROSS-REFERENCE TO RELATED APPLICATIONS (1) This application is a continuation-in-part of U.S. non-provisional application Ser. No. 15/999,506, filed Aug. 20, 2018, the contents of which are incorporated by reference herein.

BACKGROUND OF THE INVENTION

(1) A reference material or a certified reference material may be prepared for use for identifying and quantifying certain gaseous compounds in collected air samples. Commercial companies (such as Linde, Inc. and Airgas, Inc.) and National Institute of Standards and Technology (NIST) provide premade standardized compounds, also known generally as “reference materials” or “certified reference materials,” that are used for comparison with an air sample. In the art and hereinafter, the phrase “Reference Materials” may be used to refer generally to either or both of “uncertified” reference materials or certified reference materials. Once the air sample is compared to the Reference Material, the tested-for volatile compounds are then quantitated accordingly. However, the current system for providing standardized testing in laboratories becomes problematic when a lab requires a different concentration than those provided by Air Gas, Linde, NIST, or other commercial company. Additionally, the current system requires shipment of high-pressure cylinders which requires special shipping methods. In the current system, when the pressurized cylinders have reached their expiration date, they either need to be shipped back to the supplier for recertification or disposed of, both of which creates complications for laboratories. In other words, as technology advances and labs require different concentrations of Reference Materials, the invention described and claimed herein allows such labs to create a desired reference material at the desired concentration onsite and avoid the complications of sending and receiving certified pressurized cylinders.

BRIEF SUMMARY OF THE INVENTION

(2) The gas phase standard preparation device described herein allows laboratories to request a custom reference material or a custom certified reference material for the requested air quality analysis. This is accomplished by providing a premade sealed ampule that contains the requested ampule reference material, which is comprised of a chemical cocktail at a pre-determined concentration. The ampule containing the ampule reference material is then placed into a steel sleeve and the sleeve is sealed. The sleeve is then placed into a gas phase standard preparation station, which may include separate compartments.

(3) In one embodiment a gas phase standard preparation device includes a housing frame, a sealed ampule tube, a gas/vacuum supply line, a pressure sensor, a vacuum pump valve, a diluent gas valve, a crusher assembly, and an electronics control board. The sealed ampule tube further includes an ampule, an ampule sleeve, a first connector and a second connector. The ampule sleeve has a first end and a second end. The gas/vacuum supply line has a first end and a second end, wherein the first end of the gas/vacuum supply line is connected to the pressure sensor and the second end of the gas/vacuum supply line is connected to the first end of the ampule sleeve. The vacuum pump valve and the diluent gas valve are fluidly coupled to the pressure sensor. The crusher assembly is configured to provide a uniform deformation of the ampule sleeve, thereby causing a breaching of the ampule. The electronics control board electronically couples to the crusher assembly, pressure sensor, the vacuum pump valve, and the diluent gas valve. The second end of the ampule sleeve is fluidly coupled to a reference material cylinder.

(4) In one embodiment a method of using the gas phase preparation device described above further includes placing a clean and evacuated reference material cylinder into the housing frame, connecting the first end of the ampule sleeve to the gas/vacuum supply line, connecting the second end of the ampule sleeve to the reference material cylinder, evacuating the ampule sleeve, leak-checking the ampule sleeve, opening a shutoff valve of the reference material cylinder, starting a trickle flow of a diluent gas into the gas phase standard preparation device, energizing a motor of the crusher assembly to provide linear movement of a carriage to provide a breaching of the

ampule, breaching the ampule and releasing ampules reference material, and starting a preparation program. The preparation program includes filling the reference material cylinder with the diluent gas and the ampule reference material, thereby forming a final reference material, and closing the shutoff valve of the reference material cylinder. The method may further include verifying the concentration of the final reference material within the reference material cylinder.

Description

BRIEF DESCRIPTION OF THE DRAWINGS

(1) FIG. **1** shows the ampule sealed within the ampule sleeve prior to being placed into the gas phase standard preparation station.

(2) FIG. **2**A is a schematic diagram of the preparation station.

(3) FIG. **2**B is a schematic diagram of the electric/electronic components of the preparation station.

(4) FIG. **3** shows a first non-limiting embodiment of the preparation station with the first lid in an open position.

(5) FIG. **4** shows the first non-limiting embodiment of the preparation station with the first lid in a closed position.

(6) FIG. **5** shows a second non-limiting embodiment of the preparation station with the first lid in an open position.

(7) FIG. **6** shows the second non-limiting embodiment of the preparation station with the first lid in a closed position.

(8) FIG. **7** shows the compartment of the first and second non-limiting embodiments holding the cylinder having a formed adapter, load sensor and IR sensor.

(9) FIG. **8** shows a side elevation view of a third non-limiting embodiment of the preparation station.

(10) FIG. **9** shows a side elevation perspective view of the preparation station of FIG. **8**.

(11) FIG. **10** shows a side elevation perspective view of the crusher assembly of the preparation station of FIG. **8**.

(12) FIG. **11** is a schematic diagram of the electric/electronic components of the preparation station of FIG. **8**.

NUMBER REFERENCES

(13) **5**—Sealed ampule tube **10**—Ampule **15**—Ampule sleeve **20**—Spacers **25**—Connectors **40**—Reference material cylinder **42**—Shutoff valve **50**—Preparation Station (First Embodiment) **50**A—Housing **51**—First lid **52**—Second Lid **53**—Clasp **54**—Hinge **55**—Vacuum pump **56**—Vacuum pump valve **60**—Diluent gas cylinder **61**—Diluent gas valve **64**—Tube **65**—Pressure sensor **66**—gas/vacuum supply line **70**—Mandrel **75**—Lid Mandrel **80**—Electronics control board **82**—Power Source **83**—AC Mains **84**—Battery **85**—Insulation wall **87**—Bulkhead **90**—Heat sink **100**—Preparation Station (Second embodiment) **100**A—Housing **105**—Bearing **110**—Load sensor **115**—IR Sensor **120**—Formed adapter **122**—Fulcrum point protrusion **200**—Preparation Station (Third embodiment) **202**—Housing frame **203**—Shroud compartment **204**—Crusher assembly **205**—Support brace **206**—Motor **207**—Motor power source **208**—Electronics support wall **210**—Drive assembly **218**—Lower support bracket **220**—Screw shaft **228**—Guide tracks **230**—Carriage **231**—Fasteners **232**—U-brace **233**—Support arms **234**—Nut bracket **235**—Slides **236**—Clamping assembly **237**—Crusher wheel **238**—Crusher wheel assembly **239**—Wheel mountings **240**—Clamp brackets **250**—Upper support bracket **260**—Mandrel base

LETTER REFERENCES

(14) A—Longitudinal central axis of crusher wheel **237** B—Longitudinal central ampule mounting axis of mandrel base **260** C—Vertical axis of support arms **233**

DETAILED DESCRIPTION OF THE EMBODIMENTS

(15) This device and method of use for a gas phase standard preparation device (the "Device") allows for the Reference Material to be created in a laboratory instead of ordering premade, high-pressure cylinders that are to be utilized in the lab or at an off-site location. This Device additionally allows for the Reference Material to be tailored or customized for the lab that it is to be used in. This is beneficial when the companies or the National Institute of Standards and Technology (NIST) that produce such reference materials are incapable of providing a premade standardized reference material. This is also beneficial for the laboratory and the environment as this Device may contribute to the reduction of waste cylinders because the high-pressure cylinders may be reused. This Device would also result in drastically lower shipping costs due to the fact that a sealed ampule would be shipped as compared to a premade, high-pressure cylinder.
(16) As shown in FIG. **1**, a sealed ampule tube **5** includes an ampule **10**, an ampule sleeve **15**, and connectors **25**. Although connectors **25** are shown as compression fittings (with plugs), they may be any suitable connector utilized for joining piping or tubing and which is configured to provide airtightness and compatibility. The ampule **10** may be formed as a glass sealed tube and the tube may undergo Non-Destructive Testing (NDT) to meet certain quality standards. The ampule **10** contains a mixture of chemicals at predetermined concentrations, which collectively are referred to as ampule reference material. This ampule reference material will be blended with diluent gas to form a final reference material which will be verified for concentration. The ampule **10** is placed within the ampule sleeve **15**. The ampule sleeve **15** should be made of airtight and inert or passivated materials. This can be accomplished by providing a stainless-steel sleeve that is internally coated with a passivation material such as a silica ceramic. The ampule sleeve **15** includes a first end and a second end with each end having a connector **25**, which can be seen in FIG. **1**. While the ampule **10** is typically made of glass, it may be made of any suitable material compatible with the intended chemicals and pressures. It is anticipated that the sealed ampule tube **5** may also include one or more spacers **20** for maintaining the ampule **10** in a specific location within the ampule sleeve **15**.
(17) The ampule sleeve **15**, which contains the ampule **10**, is placed within a preparation station. The preparation station is described herein as various embodiments: a first embodiment for preparation station **50**, a second embodiment for preparation station **100**, and a third embodiment for preparation station **200**.
First Embodiment
(18) The first non-limiting embodiment for a preparation station **50** includes a housing **50**A, sealed ampule tube **5**, a first lid **51** with a clasp **53**, a second lid **52**, and a hinge **54** that the first lid **51** and second lid **52** are attached to and may pivot about, a heated mandrel **70**, a lid mandrel **75**, and an electronics control board **80**. The electronics control board **80** of the gas phase standard preparation device is coupled to a power source **82**, such as AC (alternating current) mains **83** or a battery **84** (see FIG. **2**B). This embodiment further includes a load sensor **110**, an IR sensor **115**, and a formed adapter **120**. Although, the first embodiment for preparation station **50** is shown and described as having four separate compartments, it is anticipated that the location, number, and orientation of the compartments may be altered without departing from the novel aspects of this non-limiting embodiment and the other non-limiting embodiments being claimed and described herein.
(19) The first compartment includes a first connection and a second connection in the exterior side of the housing for coupling a vacuum pump **55** and diluent gas cylinder **60**, respectively. Diluent gas cylinder **60** contains a suitable diluent gas, such as nitrogen gas, zero air, argon gas, or helium gas. The vacuum pump **55** is fluidly coupled to a vacuum pump valve **56** and the diluent gas cylinder **60** is fluidly coupled to a diluent gas valve **61**. A set of tubes extend from the vacuum pump valve **56** and the diluent gas valve **61** and converge into a tube **64** as seen in FIG. **3** and FIG. **4**. The vacuum pump valve **56** and the diluent gas valve **61** are each preferably proportional valves, but either may alternatively be comprised of a servo valve. The tube **64** connects to a pressure sensor **65**. The tube **64** then extends from the pressure sensor **65** and fluidly couples to a gas

vacuum supply line **66**, which is provided in a second compartment as shown in FIG. **3** and FIG. **4**.
(20) The gas/vacuum supply line **66** is used to supply diluent gas to the ampule sleeve **15** of sealed ampule tube **5** and additionally to remove any room air from within the ampule sleeve of the sealed ampule tube prior to supplying the diluent gas. The gas/vacuum supply line **66** has a first end and a second end. The first end of the gas/vacuum supply line **66** is connected to the tube **64** and the second end of the gas/vacuum supply line **66** is connected to the first end of the ampule sleeve **15** which contains the ampule **10**. It is anticipated that the gas/vacuum supply line **66** may be bendable or flexible.
(21) The ampule sleeve **15** extends into a third compartment within the preparation station **50**. The third compartment includes insulated walls **85** and the heated mandrel **70**. The heated mandrel **70** provides heat at the location, or substantially at the location, of the ampule **10** within the ampule sleeve **15**. The heated mandrel **70** is electrically coupled to and controlled by the electronics control board **80** in the first compartment. The second and third compartments within the preparation station **50** are adjacent to one another and are provided above the first compartment. The first lid **51** is provided above the second and third compartments, which is shown in FIG. **4**. The first lid **51** provides access to the sealed ampule tube **5** for removal and installation.
(22) The second end of the ampule sleeve **15** is connected to an evacuated reference material cylinder **40** with a formed adapter **120**. The gas phase standard preparation device includes an interior bulkhead **87** attached to the housing that forms a fourth compartment. The reference material cylinder **40** is included in the fourth compartment, receives the Reference Material, and includes a shutoff valve **42**. A heat sink **90** is included in the fourth compartments, which is accessible when the second lid **52** is opened. The heat sink **90** provides added containment security in the event that the integrity of the reference material cylinder **40** is compromised. The heat sink **90** also assists with absorbing heat as gas is compressed into the reference material cylinder **40**. The insulation walls **85** are provided proximate to the heated mandrel **70**, as shown in FIGS. **3-6**, for conserving energy due to heat loss. When an individual desires to remove a reference material cylinder **40** or place a reference material cylinder **40** in the fourth compartment, the second lid **52** should be lifted.
(23) A lid mandrel **75** is provided on the first lid **51**. The lid mandrel **75** and heated mandrel **70** include arcuate surfaces, which are shown in FIG. **3** and FIG. **4**. The arcuate surface of the lid mandrel **75** compliments the arcuate surface of the heated mandrel **70**. Accordingly, when the sealed ampule tube **5** is installed, the arcuate surface of the lid mandrel **75** will bend the ampule **10** within the sealed ampule tube **5** against the heated mandrel **70**, thereby breaking the ampule and releasing the ampule reference material within it.
(24) The electronics control board **80** is electronically coupled to various electrical components within the gas standard preparation device as well as includes thermocouple control (T.C.), pressure sensor control (P.S.), valve control (V.C.), and heater control (H.C.). In this first non-limiting embodiment, the electronics control board **80** controls the IR sensor **115** thermocouple included in the compartment housing the reference material cylinder **40**. The electronics control board **80** also controls the pressure sensor **65**, the vacuum pump valve **56**, the diluent gas valve **61**, and the heater control which is connected to the heated mandrel **70**. In addition, it is anticipated that the lid mandrel **75** may include heating elements and be electronically coupled to the electronics control board **80**. The heating elements would be configured to heat the mandrel to a temperature of up to about 240 degrees maximum. The desired temperature would depend on what is needed for each application, which would depend on the compounds/chemicals contained within ampule **10**. Electronics control board **80** is electronically coupled to with IR sensor **115** to allow for monitoring of the temperature of reference material cylinder **40** while being filled with gas, to prevent overheating, and with load sensor **110** for monitoring the weight of reference material cylinder **40** while it is being filled with gas, to determine the mass of gas being added.
(25) The infrared ("IR") sensor **115** is included to detect the temperature of the reference material

cylinder **40** as it is being filled with gas. It is anticipated that the IR sensor **115** is electronically coupled to the electronics control board **80** and assists with ensuring that the reference material cylinder **40** is not overheating due to compression of the gas filling the reference material cylinder.

(26) The load sensor **110** is placed under the formed adapter **120**. The formed adapter **120** fluidly couples the sealed ampule tube **5** to the reference material cylinder **40**. The formed adaptor **120** includes a fulcrum point protrusion **122** for positioning above the load sensor **110**. As gas is added to the reference material cylinder **40**, the fulcrum point protrusion **122** moves down and contacts the load sensor **110** to measure the mass of gas added to the reference material cylinder. The formed adapter **120** is held in place by the second lid **52**, thereby causing the reference material cylinder **40** to pull down as a cantilevered weight at the fulcrum point protrusion **122**. Accordingly, an accurate weight can be observed as the reference material cylinder **40** is filled. In other embodiments, the fulcrum protrusion **122** may also be utilized with load sensor **110**, although not shown in the drawing figures.

(27) The use of the formed adapter **120** and load sensor **110** in combination with the IR sensor **115** avoids having to provide a weight sensor directly on each reference material cylinder **40** placed into the gas standard preparation device.

(28) Prior to creating a Reference Material with the preparation station **50**, the second lid **52** is lifted, the reference material cylinder **40** is placed in the fourth compartment, the first lid **51** is lifted, and the sealed ampule tube **5** is installed in the preparation station. The sealed ampule tube **5** is installed by connecting the first end of the ampule sleeve **15** of sealed ampule tube **5** to the second end of the gas/vacuum supply line **66** and joining the second end of the ampule sleeve **15** of sealed ampule tube **5** to the formed adaptor **120** of reference material cylinder **40**. A vacuum pump **55** is fluidly coupled to the vacuum pump valve **56** and a diluent gas cylinder **60** filled with diluent gas is fluidly coupled to the diluent gas valve **61**. The vacuum pump **55** is used to evacuate (remove any room air from) the tubes and sealed ampule tube **5** within the preparation station **50** and a leak check is performed. Once the room air is removed, the shutoff valve **42** of the reference material cylinder **40** is opened and a trickle flow of the diluent gas from the diluent gas cylinder **60** is released into the preparation station **50**. Accordingly, the first lid **51** is closed, thereby breaking the ampule **10** and releasing the ampule reference material within the ampule **10** into the ampule sleeve **15**. A preparation program is then started.

(29) The preparation program is comprised of: (1) Heating the mandrel **70** to a desired temperature up to 240 degrees Celsius; (2) Flowing the diluent gas through the ampule sleeve **15** containing the broken ampule **10** to mix the diluent gas with the ampule reference material as they flow into the reference material cylinder; (3) Monitoring temperature on the reference material cylinder **40**; (4) Pressurizing the cylinder to a final predetermined pressure; and (4) Closing the shutoff valve **42**.

(30) Once the preparation program is complete, then the concentration of the final reference material within the reference material cylinder **40** may be verified.

Second Embodiment

(31) The second non-limiting embodiment for a preparation station **100** shares many components of the first embodiment for a preparation station **50**, however this second embodiment for a preparation station **100** additionally utilizes a plurality of bearings **105**. At least one bearing of the plurality of bearings **105** is placed on the lid mandrel **75** and at least one bearing of the plurality of bearings **105** is placed proximate to the heated mandrel **70**, which is shown in FIG. **5** and FIG. **6**. The second embodiment for a preparation station **100** also comprises a housing **100**A and anticipates providing the gas/vacuum supply line **66** on the exterior of the housing.

Third Embodiment

(32) The third non-limiting embodiment for a preparation station **200** shares many components of the first embodiment for a preparation station **50**, which are disposed within or adjacent to a housing frame **202**, as shown in FIGS. **8** and **9**. Instead of four compartments, the preparation station **200** includes at least a separate metal shroud compartment **203** for containment of the

reference material cylinder **40**. Shroud compartment **203** includes one or more walls configured to surround at least the sides of the reference material cylinder **40**. The shroud compartment **203** also houses the shutoff valve **42** and may include the load sensor **110**. The preparation station **200** includes the housing frame **202**, the shroud compartment **203**, the sealed ampule tube **5**, the gas/vacuum supply line **66** connected to the first end of the sealed ampule tube, the pressure sensor **65**, the vacuum pump valve **56**, the diluent gas valve **61**, a crusher assembly **204**, and the electronics control board **80**.

(33) The preparation station **200** utilizes the crusher assembly **204** to provide a uniform deformation of the ampule sleeve **15**, thereby causing a controlled uniform load to be imparted to and thereby result in a breaching of ampule **10**. Such breaching may include breaking of portions and/or crushing of portions of ampule **10**, or breaking the ampule to the extent that it is breached. In this embodiment, the breaching may occur along the entire length of ampule **10**. Such breaching of ampule **10** causes a substantially complete release of the ampule reference material within it, and dispersion of the ampule reference material into the ampule sleeve **15**. Such a substantially complete release of the ampule reference material into the ampule sleeve **15** promotes a more thorough and complete blending with the diluent gas, once introduced. As shown in FIG. **10**, the crusher assembly **204** includes a motor **206**, a drive assembly **210**, a lower support bracket **218**, a screw shaft **220**, a pair of opposing guide tracks **228**, a carriage **230**, an upper support bracket **250** and a mandrel base **260**.

(34) As shown in FIGS. **8**, **9** and **11**, (similar to the first embodiment), the electronics control board **80** is configured to control the pressure sensor **65**, the vacuum pump valve **56**, the diluent gas valve **61**, and the heater control (H.C.) which is connected to the mandrel base **260**. In addition, it is anticipated that the mandrel base **260** includes heating elements and is electronically coupled to the electronics control board **80**, as shown in FIG. **9**. The heating elements would be configured to heat the mandrel base to a temperature of up to about 240 degrees maximum. The desired temperature would depend on what is needed for each application, which would depend on the compounds/chemicals contained within the ampule **10**. The electronics control board **80** may also be electronically coupled with the optional load sensor **110** (L.S., see FIGS. **8** and **9**) for monitoring the weight of the reference material cylinder **40** while it is being filled with gas, to determine the mass of gas being added (mass of gas added is used to determine total mass of gas to be used in calculating the final concentration). The electronics control board **80** is also electronically coupled with the motor **206** for motor control (M.C., see FIGS. **8** and **9**) operations. As such, the electronics control board **80** is electronically coupled with the crusher assembly **204** for H.C. and M.C. Motor control operations may include, without limitation, energizing on/off, speed and direction of rotation of one or more components of the crusher assembly **204**. The motor **206** is coupled to a motor power source **207**, such as AC (alternating current) mains **83** or a battery **84**.

(35) The motor **206** is connected to the drive assembly **210**, which is connected to the screw shaft **220** to provide linear movement of the carriage **230**. Although a pully and belt type transmission is shown for the drive assembly **210**, it may alternatively include gears, chains or other types of transmissions. In other aspects, the motor **206** may be configured for directly driving the screw shaft **220**.

(36) A support brace **205** is mounted on the vertical interior of the housing frame **202** and is configured to provide support for an electronics support wall **208**, the lower support bracket **218**, and the upper support bracket **250**. The electronics support wall **208** provides mounting for at least the electronic control board **80** and is preferably connected to one of its sides, the support brace **205** and on at least one of its other sides is connected to the shroud compartment **203**. The electronic support wall **208** may also be connected at its bottom side to the housing frame **202**. The lower support bracket **218** is connected on one side to the support brace **205** and at the other side to the shroud compartment **203**, and provides connection support for the drive assembly **210** and the pair of opposing guide tracks **228**. The upper support bracket **250** is connected on one side to the

support brace **205** and at the other side to the shroud compartment **203**, and provides connection and support for the mandrel base **260**.

(37) The carriage **230** includes a U-brace **232**, a nut bracket **234**, a clamping assembly **236** and a crusher wheel assembly **238**. The U-brace **232** includes a pair of opposing support arms **233**, which are removably connected at the top end to the clamping assembly **236**. A pair of opposing slides **235** mounted to the underside of the U-brace **232** are configured to slidably engage with the pair of opposing guide tracks **228**. The nut bracket **234** is mounted on the bottom of the U-brace **232** and is configured to threadedly engage with the screw shaft **220**.

(38) In operation, the rotational motion provided by the motor **206** is transmitted via the drive assembly **210** to the screw shaft **220**, and the rotation of the screw shaft advances the nut bracket **234** axially from end to end of the screw shaft and back, depending on the direction of rotation provided by the motor **206**. As the nut bracket **234** axially advances, the U-brace **232** and the crusher wheel assembly **238** are also advanced linearly along a longitudinal central ampule mounting axis B of the mandrel base **260**. The linear movement of the U-brace **232** is stabilized by slides **235** engaging the guide tracks **228**.

(39) The clamping assembly **236** includes a pair of opposing clamp brackets **240** and one or more fasteners **231**. Although four screw bolt and nut type fasteners **231** are shown in the figures (installed via manual tightening), in other aspects, alternative fasteners may be utilizing, such as one or more cam clamping members having actuation levers. In other aspects, alternative clamping members may be utilized whereby a single lever, handle or other means of actuation is used to securely clamp the clamp brackets **240** of the clamping assembly **236** to the opposing support arms **233**.

(40) The crusher wheel assembly **238** includes a crusher wheel **237** removably connected in-between the pair of opposing clamp brackets **240** via a pair of opposing wheel mountings **239**. The crusher wheel **237** is coaxially connected at each end to one of each of the opposing wheel mountings **239**, which allows for removable connection to the clamping assembly **236**. The crusher wheel assembly **238** is configured such that the longitudinal central axis A of the crusher wheel **237** is mounted substantially perpendicular to the vertical axis C of the support arms **233** and mounted substantially perpendicular to the longitudinal central ampule mounting axis B of the mandrel base **260**.

(41) In operation, the reference material cylinder **40** is placed within housing frame **202**. The U-brace **232** is set in its predetermined initial horizontal starting position relative to the screw shaft **220**. The sealed ampule tube **5** is installed in a predetermined position along the longitudinal central ampule mounting axis B, proximate to the mandrel base **260**. The sealed ampule tube **5** is installed by connecting the first end of the ampule sleeve **15** of the sealed ampule tube **5** to the second end of the gas/vacuum supply line **66** and connecting the second end of the ampule sleeve **15** of the sealed ampule tube **5** to the formed adaptor **120** of the reference material cylinder **40**. The vacuum pump **55** is fluidly coupled to the vacuum pump valve **56** and the diluent gas cylinder **60** filled with diluent gas is fluidly coupled to the diluent gas valve **61**. The vacuum pump **55** is used to evacuate (remove any room air from) the tubes and the sealed ampule tube **5** within the preparation station and a leak check is performed. Once the room air is removed, the shutoff valve **42** of the reference material cylinder **40** is opened and a trickle flow of the diluent gas from the diluent gas cylinder **60** is released into the sealed ampule tube **5**.

(42) The clamping assembly **236** is then positioned against the opposing support arms **233** and installed to be securely clamped to the support arms via the one or more fasteners **231**. The clamping assembly **236** is securely clamped to the opposing support arms **233** such that there is substantially zero clearance in-between the bottom of the clamp brackets **240** and the top of the support arms **233**. As the clamping assembly **236** is being clamped down, the bottom of the crusher wheel **237** deforms the top of the ampule sleeve **15** to a predetermined depth at the initial horizontal starting position. Next, the motor **206** is energized and controlled to begin the forward

linear advancement of the crusher wheel **237** from the initial horizontal starting position to a predetermined finishing position relative to the screw shaft **220** as required to breach ampule **10**. Once the crusher wheel **237** reaches the ampule **10**, it begins to breach ampule **10**, to release the ampule reference material into the ampule sleeve **15**. Next, a preparation program is then started.
(43) The preparation program is comprised of: (1) Heating the mandrel base **260** to a desired temperature up to 240 degrees Celsius; (2) Flowing the diluent gas through the ampule sleeve **15** containing the broken ampule **10** to mix the diluent gas with the ampule reference material as they flow into the reference material cylinder **40**; (3) pressurizing the cylinder to a final predetermined pressure; and (4) Closing the shutoff valve **42**.
(44) In this embodiment, it is anticipated during step (3) of the preparation program, the mass of the gas being added to reference material cylinder **40** is determined by the gas pressure, which can be monitored at the pressure sensor **65**. Once the preparation program is complete, an external balance type scale may be used to verify the weight of reference material cylinder **40**.
(45) Once reaching the finishing position, motor **206** is controlled to reverse direction and return crusher wheel **237** to the initial starting position. Crusher wheel **237** is controlled to be moving its course forward and then reverse against ampule sleeve **15** while the mixture of ampule reference material and diluent gas flow into the reference material cylinder **40**. Once the crusher wheel **237** returns to the initial starting position by moving over the ampule **10** for a second time, then the motor **206** is deenergized and controlled to end the linear advancement of the crusher wheel. At this point, the clamping assembly **236** may be removed from the opposing support arms **233**, thus removing the crusher wheel **237** from engagement with the ampule sleeve **15**.
(46) Once the preparation program is complete, then the concentration of the final reference material within the reference material cylinder **40** may be verified. The user may further certify the final reference material onsite, or alternatively, send it out for independent certification. The International Organization for Standardization (ISO) guidelines provide standardization requirements for certified reference materials and reference materials, such ISO 17034 and ISO/IEC 17025, for example.
(47) While the embodiments of the one or more non-limiting embodiments have been disclosed, certain modifications may be made by those skilled in the art to modify the embodiments without departing from the scope of the claims.

Claims

1. A gas phase standard preparation device comprising: a. a housing frame; b. a sealed ampule tube; wherein the sealed ampule tube is further comprised of an ampule, an ampule sleeve, a first connector and a second connector; wherein the ampule is provided within the ampule sleeve; wherein the ampule sleeve has a first end and a second end; c. a gas/vacuum supply line; wherein the gas/vacuum supply line has a first end and a second end; wherein the second end of the gas/vacuum supply line is connected to the first end of the ampule sleeve; d. a pressure sensor; wherein the pressure sensor is connected to the first end of the gas/vacuum supply line; e. a vacuum pump valve; wherein the vacuum pump valve is fluidly coupled to the pressure sensor; f. a diluent gas valve; wherein the diluent gas valve is fluidly coupled to the pressure sensor; g. a crusher assembly; wherein the crusher assembly is configured to provide a uniform deformation of the ampule sleeve, thereby causing a breaching of the ampule; and h. an electronics control board; wherein the electronics control board electronically couples to the crusher assembly, the pressure sensor, the vacuum pump valve, and the diluent gas valve.
2. The gas phase standard preparation device of claim 1 wherein the sealed ampule tube is removable.
3. The gas phase standard preparation device of claim 1 wherein a reference material cylinder is connected to the second end of the ampule sleeve.

4. The gas phase standard preparation device of claim 3 further comprising a formed adaptor having a first end and a second end, wherein the second end of the ampule sleeve is connected to the first end of the formed adaptor, and the second end of the formed adaptor is connected to the reference material cylinder.
5. The gas phase standard preparation device of claim 4 further comprising a shroud compartment for containment of the reference material cylinder.
6. The gas phase standard preparation device of claim 5 further comprising a load sensor electronically coupled to the electronics control board to allow for monitoring the weight of the reference material cylinder to determine the mass of gas being added.
7. The gas phase standard preparation device of claim 6 wherein the formed adaptor further comprises a fulcrum point protrusion for positioning above the load sensor, wherein as gas is added to the reference material cylinder, the fulcrum point protrusion moves down and contacts the load sensor to measure the mass of gas added to the reference material cylinder.
8. The gas phase standard preparation device of claim 1 wherein the crusher assembly further comprises a motor, a drive assembly, a lower support bracket, a screw shaft, a pair of opposing guide tracks, a carriage, an upper support bracket and a mandrel base; wherein the motor is connected to the drive assembly, which is connected to the screw shaft to provide linear movement of the carriage.
9. The gas phase standard preparation device of claim 8 wherein the mandrel base includes heating elements.
10. The gas phase standard preparation device of claim 8 wherein the carriage includes a U-brace, a nut bracket, a clamping assembly and a crusher wheel assembly; wherein the nut bracket is mounted on the bottom of the U-brace and is configured to threadedly engage with the screw shaft.
11. The gas phase standard preparation device of claim 10 wherein the carriage further comprises a pair of opposing slides mounted to the underside of the U-brace and are configured to slidably engage with the pair of opposing guide tracks.
12. The gas phase standard preparation device of claim 11 wherein the U-brace includes a pair of opposing support arms, which are removably connected at a top end thereof to the clamping assembly.
13. The gas phase standard preparation device of claim 12 wherein the clamping assembly includes a pair of opposing clamp brackets and one or more fasteners.
14. The gas phase standard preparation device of claim 13 wherein the crusher wheel assembly includes a crusher wheel removably connected in-between the pair of opposing clamp brackets via a pair of opposing wheel mountings.
15. The gas phase standard preparation device of claim 14 wherein the crusher wheel is coaxially connected at each end to one of each of the opposing wheel mountings which allows for removable connection to the clamping assembly.
16. The gas phase standard preparation device of claim 15 wherein the crusher wheel assembly is configured such that a longitudinal central axis of the crusher wheel is mounted substantially perpendicular to a vertical axis of the support arms and mounted substantially perpendicular to a longitudinal central ampule mounting axis of the mandrel base.
17. A method of using the gas phase standard preparation device of claim 3 above comprising: obtaining the gas phase standard preparation device of claim 3, then; a. placing the reference material cylinder into the housing frame; b. connecting the first end of the ampule sleeve to the gas/vacuum supply line; c. connecting the second end of the ampule sleeve to the reference material cylinder; d. evacuating the ampule sleeve; e. leak-checking the ampule sleeve; f. opening a shutoff valve of the reference material cylinder; g. starting a trickle flow of a diluent gas into the gas phase standard preparation device; h. energizing a motor of the crusher assembly to provide linear movement of a carriage to provide a breaching of the ampule; i. breaching the ampule and releasing ampule reference material; j. starting a preparation program; k. filling the reference

material cylinder with the diluent gas and the ampule reference material, thereby forming a final reference material; and l. closing the shutoff valve of the reference material cylinder.
18. The method of using the gas phase standard preparation device of claim 17 wherein the preparation program comprising: a. heating a mandrel base to a predetermined temperature; b. flowing diluent gas through the ampule sleeve containing the breached ampule; and c. pressurizing the reference material cylinder to a predetermined pressure.
19. The method of using the gas phase standard preparation device of claim 18 further comprising: heating the mandrel base to a desired temperature up to 240 degrees Celsius.