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REPLY TO ATTN OF: GP

TO: USI/Scientific & Technical Information Division
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned U. S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code USI, the attached NASA-owned U. S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U. S. Patent No. : 3,565,584

Government or Corporate Employee : Rocketdyne/A Div. of North American Rockwell Corporation Canaga Park, Cal 91304

Supplementary Corporate Source (if applicable) : _____

NASA Patent Case No. : NPO-10070

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:

Yes ☒ No ☐

Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of Column No. 1 of the Specification, following the words ". . . with respect to an invention of . . . "

Elizabeth A. Carter

Elizabeth A. Carter
Enclosure
Copy of Patent cited above

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Feb. 23, 1971 T. O. PAINE, ACTING 3,565,584
ADMINISTRATOR OF THE NATIONAL AERONAUTICS
AND SPACE ADMINISTRATION
GAS LIQUEFICATION AND DISPENSING APPARATUS
Filed Nov. 29, 1968

FIG. 1

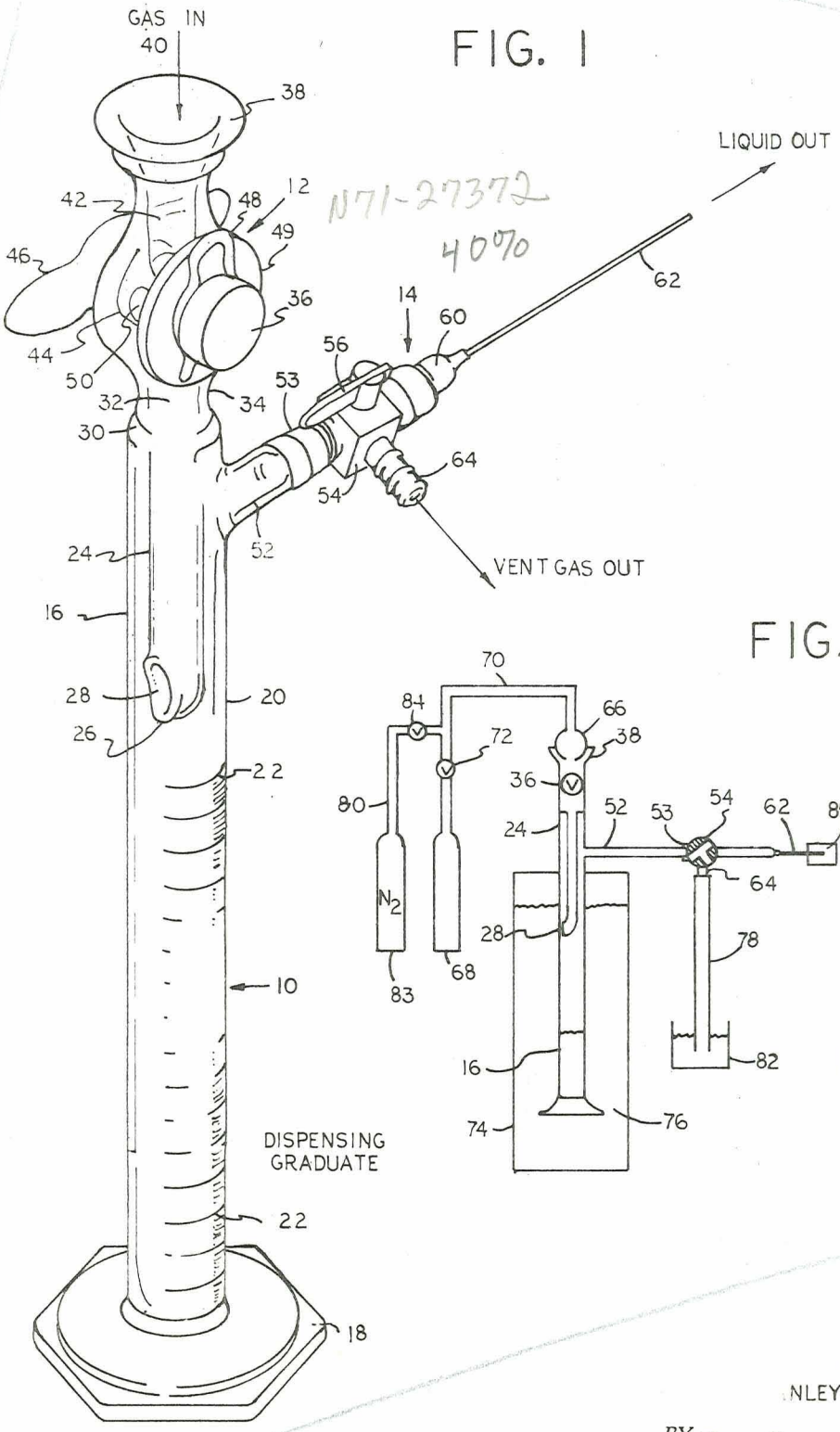




FIG. 2






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United States Patent Office 3,565,584
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3,565,584

# GAS LIQUEFICATION AND DISPENSING APPARATUS

T.O. Paine, Acting Administrator of the National Aeronautics and Space Administration, with respect to an invention of Stanley M. Hirshfield, Canoga Park, Calif.

Filed Nov. 29, 1968, Ser. No. 780,064
Int. Cl. B01l 5/00; F28d 1/02
U.S. Cl. 23—259 3 Claims

## ABSTRACT OF THE DISCLOSURE

An apparatus for condensing a reagent gas and volumetrically dispensing the resultant liquid under isolation conditions is disclosed. The apparatus comprises a volumetrically calibrated transparent container such as a graduate having a side branch liquid outlet arm. An inlet gas assembly communicates with an elongated inlet tube having an opening below the outlet arm which serves to isolate the liquid from the inlet valve and introduces the gas below the coolant level and below the outlet arm. The graduate is immersed in coolant during collection of liquid reagent. The outlet terminates in a needle nozzle which pierces a septum placed over the recipient vessel for the dispensed liquid reagent.

## ORIGIN OF THE INVENTION

The invention described herein was made in the performance of work under a NASA contract and is subject to the provisions of Section 305 of the National Aeronautics and Space Act of 1958, Public Law 85–568 (72 Stat. 435; 42 U.S.C. 2457).

## BACKGROUND OF THE INVENTION

### (1) Field of the invention

The present invention relates to an apparatus for condensing and dispensing normally volatile gases. More particularly, the invention relates to an apparatus and process for volumetrically dispensing reagent quantities of volatile chemicals for small batch reactions.

### (2) Description of the prior art

It has been found very difficult to conveniently provide accurate quantities of uncontaminated gaseous reactants for small batch reactions. Critical control of these small quantities is very significant to the collection of meaningful data. For example, in polymer research involving butadiene polymerization, a slight error in the quantity of butadiene introduced can grossly influence both the quantity of homopolymer or copolymer produced as well as the elastomeric properties of the final product which is largely influenced by the ratios of monomers present. Furthermore, the presence of oxygen or water vapor can grossly influence the properties of the final product by forming cross-linking bonds or by activating or deactivating the catalyst.

Presently, small quantities of gaseous reagents are obtained by dispensing gas from heavy pressure vessels while they are being weighed on a large balance. It is extremely difficult to maintain precision between experiments with the use of such equipment since such small differential weights will be obtained from such large and heavy tare weights. Precision within less than 0.1 cc. of the condensed liquid is desired and cannot be readily provided by these techniques.

## OBJECTS AND SUMMARY OF THE INVENTION

It is therefore an object of the invetnion to provide for accurate dispensing of very small quantities of gaseous reagents.



A further object of the invention is the provision of an apparatus suitable for convenient use at cryogenic or near cryogenic temperatures for condensing, collecting and volumetrically dispensing normally gaseous chemicals.

Yet another object of the invention is to provide for collecting and dispensing liquefied reagents in an uncontaminated condition.

These and other objects and many attendant advantages of the invention will become apparent as the description proceeds.

The liquefying and volumetrically dispensing apparatus of the invention comprises an elongated hollow member having side walls and an open top and a closed bottom member. The lower portion of the cylinder is formed into a volumetrically calibrated chamber, at least a portion of the side wall along the chamber is transparent and is marked with visible volumetric indicia. A liquid outlet assembly containing a branch conduit and an orifice outlet member is connected to a side wall of the cylinder adjacent the top thereof through a three-way valve. The outlet orifice is preferably in the form of an elongated small-diameter dispensing nozzle member.

The inlet assembly comprises a socket joint for receiving a mating fitting for forming a gas-sealed joint. A two-way valve is provided below the fitting and communicates with an elongated gas inlet tube which extends downwardly into said cylinder and contains an opening disposed below said outlet assembly but above said calibrated chamber.

In operation of the apparatus of the invention, the chamber, usually in the form of a cylinder, is immersed into a refrigerant above the level of the opening in the gas inlet tube but below the level of the side arm outlet assembly. The apparatus is purged with inert gas, and then reagent gas is introduced and condensed in the calibrated chamber until at least the desired volume has accumulated. The reagent gas feed is then shut off, the cylinder removed from the refrigerant and a measured quantity of liquefied gas is dispensed into a reaction vessel. If desired, the apparatus may be purged before immersion in refrigerant. In one procedure, the cylinder is heated with a heat gun to vaporize moisture during nitrogen purging, and the system is then closed and inserted into refrigerant after cooling to room temperature.

The apparatus of the invention permits critical dispensing of very small volumes of reagent without the complications required with weighing heavy pressure vessels in which gaseous reactants are normally stored. The apparatus can be readily utilized with any of the near cryogenic or cryogenic fluids that are compatible with the materials from which transparent laboratory equipment is fabricated. Furthermore, the provision for inert gas blanketing of the condensed fluid eliminates the probability of contamination in handling or during storage. Furthermore, refrigerants can be tailored to suit the boiling point of the various fluids at least to liquid nitrogen temperature with currently available technology.

The inlet and outlet assemblies are positioned outside of the cryogenic environment and are thus not subject to the stresses encountered when subjected to extremely cold temperatures. Thus, the valve members remain unfrozen and freely removable throughout the operation. Furthermore, the construction of the inlet tube provides for delivery of the reagent gas below the level of the outlet tube and this permits the use of an open system during condensation and thus avoids the danger of implosion or explosion. The side wall opening in the inlet tube also isolates the inlet assembly from the liquid as it is poured out and thus provides for better maintenance of the apparatus and for more accurate measurement of the dispensed quantity of liquid.

The invention will now become better understood by reference to the following detailed description when considered in connection with the accompanying drawings.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a perspective view of a preferred embodiment of the apparatus of the invention; and

FIG. 2 is a schematic view illustrating the manner in which the apparatus is utilized according to the invention.

Referring now to FIG. 1, the apparatus of the invention comprises a liquid collection and measuring device having a collection chamber **10**, an inlet assembly **12** and an outlet assembly **14**.

The collection chamber **10** comprises the lower portion of a graduated cylinder **16** formed of transparent material such as glass having a base member **18** and a side wall **20**. The wall **20** surrounding the chamber **10** is marked with visible volumetric indicia such as scribed lines **22** which are correlated to the calibrated volume increments of the chamber **10**.

An isolation-inlet tube **24** extends downwardly from the top of the cylinder **16** and terminates in a rounded bottom portion **26** disposed above the collection chamber **10**. A gas outlet **28** is provided in the side wall of the inlet tube **24** in the proximity of the rounded bottom **26**. The inlet tube **24** is fixed to the top of the cylinder by a gas-tight glass weld at **30**. The top **32** of the inlet tube is surrounded by a glass sleeve **34**. The sleeve **34** is fixed to the cylinder **16** and contains a female ground glass joint member **38**. The opening **40** of the ground glass joint **38** communicates with a tube **42** which terminates in an arcuate portion which forms a sealing fit engagement with the stem **44** of the pressure stockcock **36**. The stem **44** has a handle **46** attached to one side and a pin **48** secures a washer **49** to the other side thereof. The stem **44** may be revolved by turning the handle **46** to place the through-hole **50** in communication with the tube **42** and the isolation tube **24** when a flow into the cylinder **16** is desired. When a solid portion of the stem **44** is placed in front of the tube **42**, the assembly is sealed.

The outlet assembly **14** comprises an elongated side arm disposed on the side wall **20** of the cylinder **16** at a position facing away from the outlet opening **28** in the isolation tube **24**. A length of glass tubing **52** is fused to the wall **20**, preferably in the proximity of the top of the cylinder **16**. Leur-lock fitting **53** is cemented to tubing **52**. A three-position valve **54** having two outlets and a handle member **56** is connected to the fitting **53**. The first outlet is an externally threaded member to which is attached a hub **60** and a hypodermic needle **62**. The second outlet is in the form of a short length **64** of metal tubing for venting the system as will be subsequently explained.

Referring now to FIG. 2 for a more detailed explanation of the operation of the device, the cylinder **16** is connected to the male-ground glass connector **66** of a reagent gas train. The reagent gas is stored in a pressurized tank **68**. A line **70** containing a valve **72** connects the train to the connector **66**. A branch line **80** containing a valve **84** communicates a pressurized nitrogen tank **83** to the line **70**.

The cylinder **16** is immersed into a cryogenic container **74** filled with cryogenic liquid **76**. The cylinder **16** is immersed to a level at which the cryogenic liquid is above the outlet **28** but below the outlet side arm **52**. A vent tube **78** is immersed in a mercury bubble trap **82** and is connected to the vent tubing **64** on three-way valve **54**.

The valve **84** in the nitrogen train is opened, as is stopcock valve **36**, and nitrogen sweeps through the system. The three-way valve is turned first to purge the needle which is then covered with a cork stopper **85** and the three-way valve is turned to vent and purge the bubble trap **82**.

The nitrogen valve **84** is then closed and the reagent gas valve **72** is placed in the open position. With the outlet three-way valve **54** turned toward vent, the gas is fed into the system at a level below the refrigerant and condenses and collects in the chamber **10**. After the required amount of liquid has been collected, the stopcock **36** and the gas valve **72** are closed and the gas train removed. The three-way valve **54** is turned to a closed position and the cylinder **16** is removed from the cryogenic container **74**. The cork **85** is removed and the needle **62** inserted into a reaction vessel **88** by piercing a septum **90**. The three-way valve **54** is turned to needle-out-only position and the liquefied reagent flows into the vessel **88**. During nonuse of the apparatus, the valve **54** and needle **62** are disconnected from the Leur-lock fitting **53**.

The following detailed example is presented by way of illustration only and is not intended in any way to limit the invention.

## DESCRIPTION OF THE PREFERRED EMBODIMENTS

The cylinder was connected to a 1,3-butadiene (B.P. −5° C.) dispensing train through the socket joint. The cylinder was immersed in the cryogenic container which contained a Dry Ice-acetone bath maintained at −78° C. The assembly was swept with nitrogen and then 1,3-butadiene gas was introduced into the cylinder below the level of the liquid in the cryogenic container. The flow rate of butadiene gas was adjusted to a level to provide some bubbling in the trap and thus avoid backfeed of mercury into the system. Liquefied butadiene collected in the graduated portion of the cylinder, and after a sufficient quantity of butadiene for batch polymerization had been accumulated, the supply of butadiene was terminated. The stopcock and the three-way valve were closed and the butadiene train removed from the socket joint. A measured quantity of liquefied butadiene was added to a solution of catalyst in a septum closed reaction vessel. Accuracies of less than 0.5 cc. have been readily achieved.

The apparatus of the invention is adapted to the collection, measurement and dispensing of normally gaseous research or laboratory reagents such as chlorine, boron trifluoride, Freon, etc. The cryogenic refrigerant should be selected to quickly liquefy the gas without solidifying it. Available refrigerants such as Dry Ice and liquid nitrogen can be tailored to suit the boiling point of the various fluids to be treated.

The apparatus of the invention can be readily fabricated from available stock laboratory equipment. A graduated cylinder is modified by providing a sealed inlet composed of a stopcock equipped with an isolation tube and a ground glass fitting. A side outlet fitted with a three-position valve and a hypodermic needle is fused to the cylinder. With the use of high pressure calibrated vessels, a two-position valve may be utilized and the system may be closed during reagent collection rather than vented as described.

It is to be understood that the foregoing relates only to preferred embodiments of the invention and that numerous substitutions, alterations and modifications are all permissible without departing from the scope of the invention as defined in the following claims.

What is claimed is:

**1.** An apparatus for liquefying and volumetrically dispensing a gaseous reagent into a piercible septum covered vessel comprising:

a collection and dispensing vessel comprising an elongated container having a side wall and a closed bottom;

a volumetrically calibrated chamber disposed along the lower portion of said container, at least a portion of the side wall of said chamber being transparent and containing visible indicia of volumetric units and said chamber being adapted to collect liquefied gaseous reagent when cooled to a temperature below the condensation temperature of said reagent;

liquid outlet means containing a gas-tight, three-position valve and comprising a rigid, elongated branch arm

member having a first end in sealed communication with said side wall of said container adjacent the top thereof and a second end joined to an elongated small diameter dispensing nozzle means adapted to pierce a septum during dispensing said collected liquid, said three-position valve also connecting a branch to said elongated branch arm; and

gas inlet means containing a gas-tight valve and being in sealed communication with the top of said container including a central gas inlet tube extending downwardly into said container and having an opening disposed in a side wall of said inlet tube facing away from and below said outlet means.

2. An apparatus according to claim 1 in which the inlet terminates in a ground glass fitting for receiving a mating member for forming a gas-tight separable joint.

3. An apparatus according to claim 1 in which said container is formed of glass and said outlet means includes a length of glass tubing fused to an opening in the side



wall of said container, a valve fitting having a threaded outlet is joined to said length of tubing and a threaded hub and hypodermic needle are connected to said threaded outlet.


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U.S. Cl. X.R.

23—292; 62—55.5; 55—269; 73—421.5, 425.4