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Geatz

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[54] **APPARATUS AND METHOD FOR THE TRANSFER AND DELIVERY OF HIGH PURITY CHEMICALS**

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[22] Filed: **Sep. 17, 1990**

[51] **Int. Cl.**[5] **B67D 5/08**

[52] **U.S. Cl.** **222/1;** 222/59; 222/61; 222/71; 222/135; 222/152; 222/189; 222/318; 222/399; 137/205; 137/208; 137/209

[58] **Field of Search** 137/205, 208, 209, 545; 222/1, 43, 135, 152, 309, 318, 399, 59, 61, 71

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Assistant Examiner—Kenneth Bomberg
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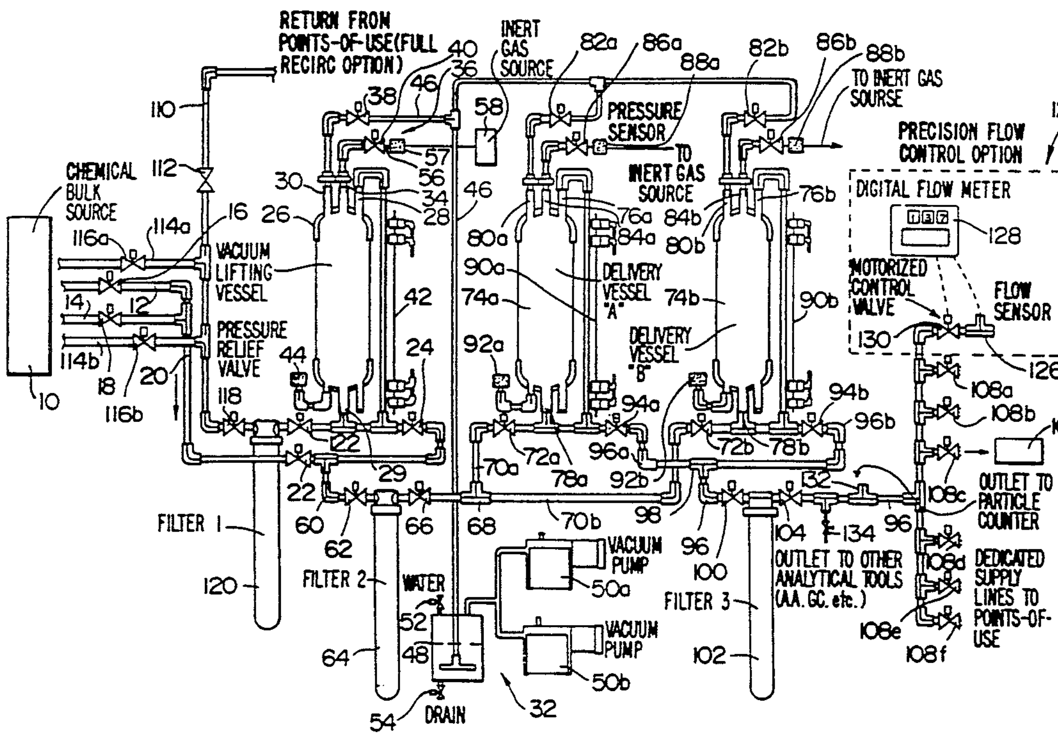
[57] **ABSTRACT**

The present invention provides improved method and apparatus for the transfer and delivery of very high purity chemicals for use in semiconductor production and similar processes. By employing multiple alternating pressure vessels, chemicals are drawn from virtually any bulk source and delivered to one or more end-users. The use of a vacuum system to draw chemicals through sealed conduits eliminates the need for pumps which are a source of both maintenance problems and contamination in the system. Multiple vessels provide for a variety of flow options, which include continuous chemical delivery to the end-users, recirculation and regular filtration during periods of low use, and built-in redundancy to avoid system shut down if there is a component failure.

37 Claims, 2 Drawing Sheets



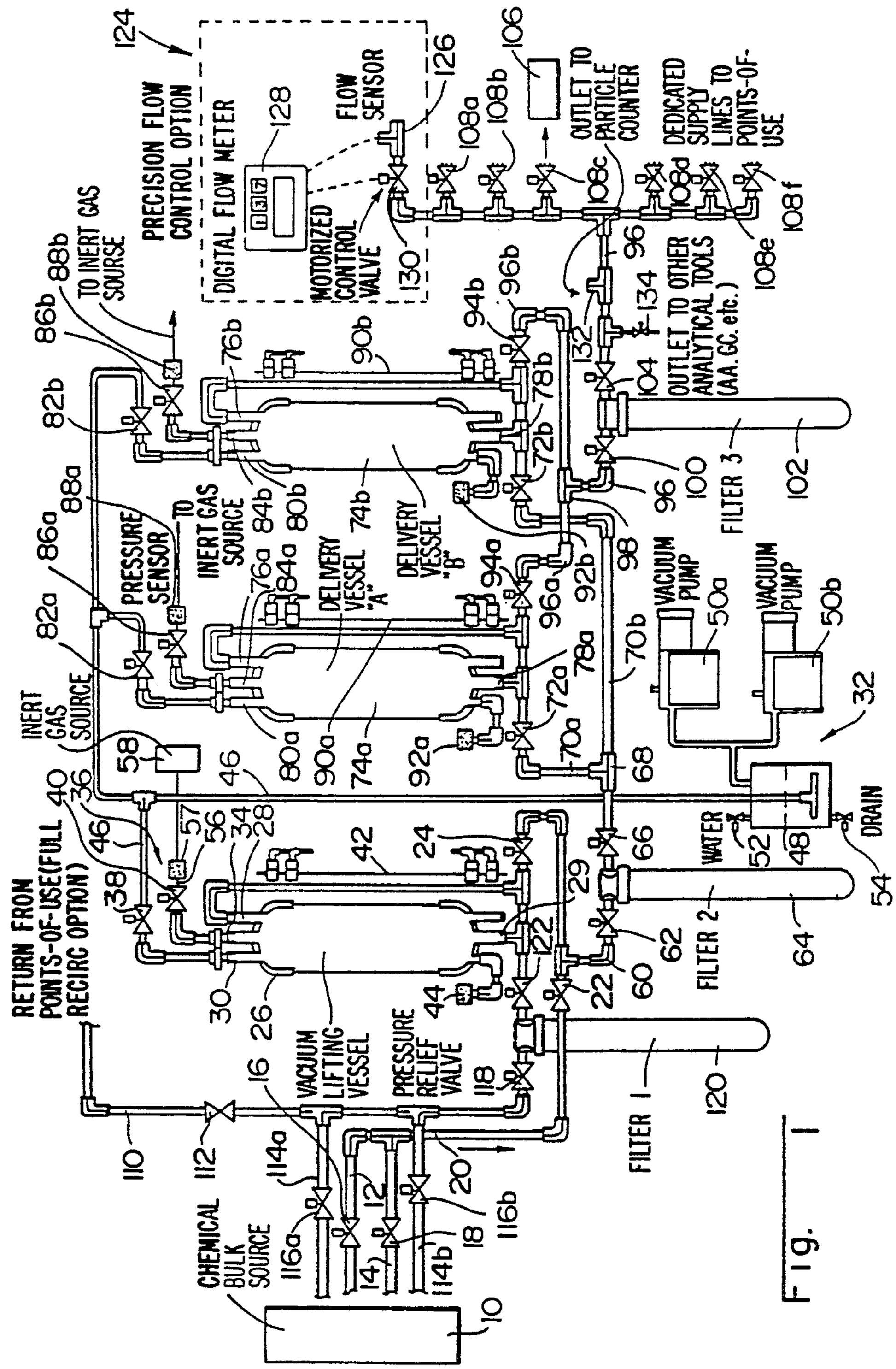
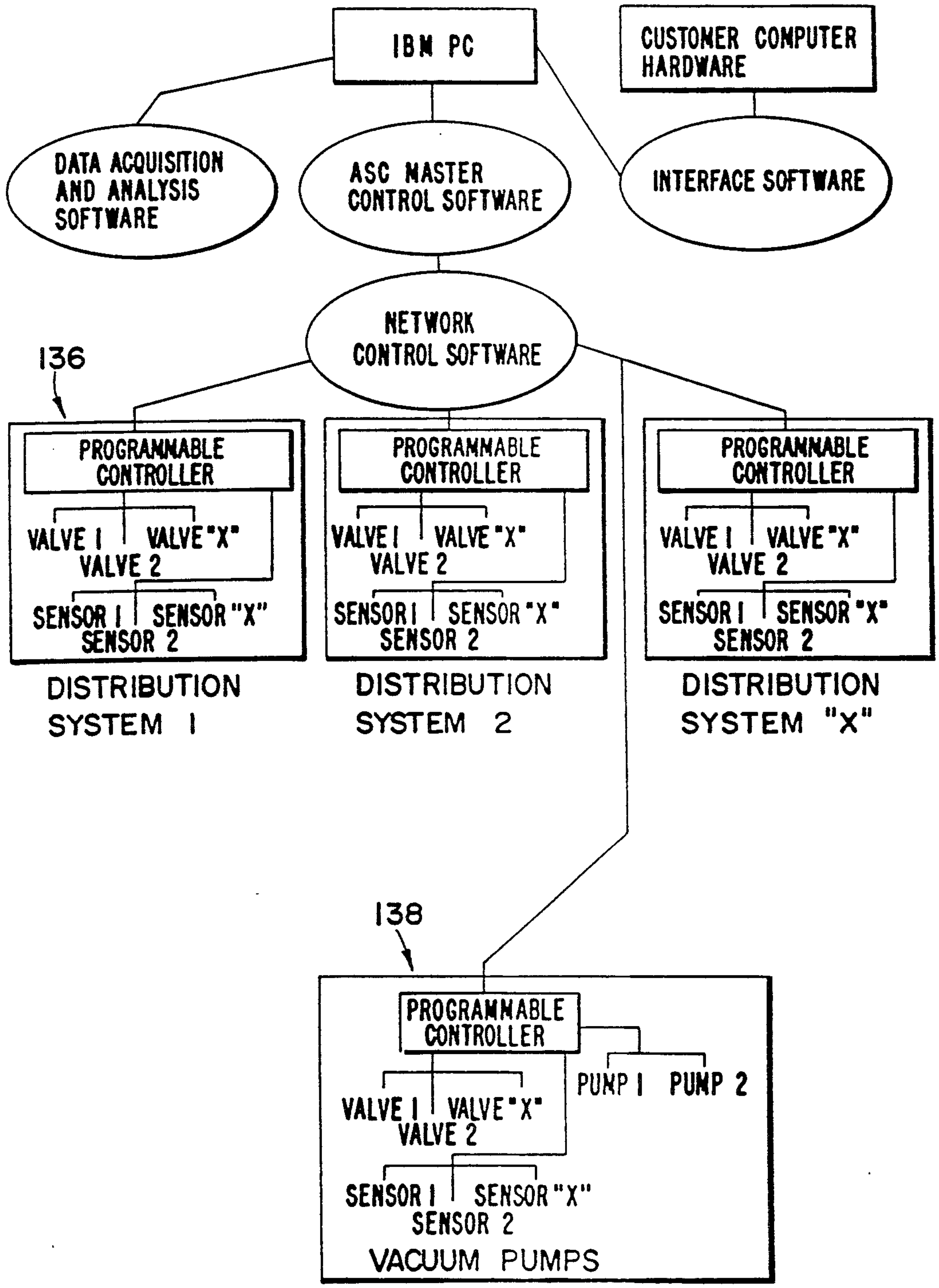

PRECISION FLOW CONTROL OPTION
DIGITAL FLOW METER
FLOW SENSOR
MOTORIZED CONTROL VALVE
TO INERT GAS SOURSE
OUTLET TO PARTICLE COUNTER
DEDICATED SUPPLY LINES TO POINTS-OF-USE
OUTLET TO OTHER ANALYTICAL TOOLS (AA. GC. etc.)
PRESSURE SENSOR
TO INERT GAS SOURCE
DELIVERY VESSEL "A"
DELIVERY VESSEL "B"
INERT GAS SOURCE
VACUUM PUMP
FILTER 3
FILTER 2
WATER
DRAIN
RETURN FROM POINTS-OF-USE (FULL RECIRC OPTION)
VACUUM LIFTING VESSEL
PRESSURE RELIEF VALVE
FILTER 1
CHEMICAL BULK SOURCE

Fig. 1

Fig- 2


IBM PC
CUSTOMER COMPUTER HARDWARE
DATA ACQUISITION AND ANALYSIS SOFTWARE
ASC MASTER CONTROL SOFTWARE
INTERFACE SOFTWARE
NETWORK CONTROL SOFTWARE
136
PROGRAMMABLE CONTROLLER
VALVE 1 VALVE "X"
VALVE 2
SENSOR 1 SENSOR "X"
SENSOR 2
DISTRIBUTION SYSTEM 1
PROGRAMMABLE CONTROLLER
VALVE 1 VALVE "X"
VALVE 2
SENSOR 1 SENSOR "X"
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DISTRIBUTION SYSTEM 2
PROGRAMMABLE CONTROLLER
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VALVE 2
SENSOR 1 SENSOR "X"
SENSOR 2
DISTRIBUTION SYSTEM "X"
138
PROGRAMMABLE CONTROLLER
PUMP 1 PUMP 2
VALVE 1 VALVE "X"
VALVE 2
SENSOR 1 SENSOR "X"
SENSOR 2
VACUUM PUMPS

APPARATUS AND METHOD FOR THE TRANSFER AND DELIVERY OF HIGH PURITY CHEMICALS

BACKGROUND OF THE INVENTION

1. Field of the Invention.

The present invention relates to the transfer, storage and delivery of process chemicals. More particularly, the present invention provides improved apparatus and method for the transfer, storage and delivery of ultra-high purity chemicals for use in a variety of industries, such as in the manufacture of semiconductor wafers and similar products.

2. Description of the Prior Art.

In many applications in industry today it is extremely important to maintain process chemicals free of virtually all contaminants. For instance, in the semiconductor industry the purity of chemicals, such as sulfuric acid, hydrogen peroxide, and ammonium hydroxide, used in semi-conductor wafer production must be pure on level of approximately 25 (or fewer) particles per milliliter with a particle size of less than a fraction of a micron. As a result of these purity standards, many conventional methods of chemical transfer and delivery, such as paddled pumps and similar devices, have proven completely unsatisfactory.

Of further concern in these industries is that many of the chemicals employed are toxic and must be carefully handled. In order to assure adequate purity and worker safety, it is extremely important that such chemicals be transferred, stored, and dispensed in a closed system, with minimal contact with the environment or workers.

Generally today one of two methods are employed to effectuate high-purity chemical transfer. The first method is a "pumped delivery." In this method a positive displacement pump, usually an air powered double diaphragm type, is employed to provide both lift at a suction inlet from the bulk source of the chemicals and simultaneous pressure at the output to the enduser. In this system, chemical is lifted from a chemical drum, driven through a pump, and pushed out to the point of use. Although this method is widely employed, it is far from satisfactory.

The deficiencies of the pumped delivery system are manifold. This system is capable of producing only minimal lift from the chemical bulk source--usually on the order of only a few pounds per square inch. Moreover, this system is replete with contamination problems: the rapidly expanding and contracting of the pump diaphragm material (e.g. Teflon®) causes mechanical degradation, with the degradation by-products (many of which being too small to filter with state-of-the-art filtration equipment) entering the chemical process stream; further, the rapid action of the pump (usually greater than 60 cycle per minute) creates massive impulse in the system with a resulting pulsed flow which forces particles through filters—thus rendering the filters ineffective. Finally, the mechanical shock inherent in this system creates constant maintenance problems.

The other system in general use today addresses only some of these problems. In the "pump/pressure delivery," a positive displacement pump is again employed to provide lift from the bulk source of chemicals. However, the chemicals are delivered to an intermediate vessel from which inert gas pressure is used to motivate chemical to the use areas.

Although the pump/pressure system is better controlled and is more conducive to use of filters to assure chemical purity, it still has serious drawbacks in a sub-micron chemical environment. Again, lift provided by a double diaphragm pump is restricted. Further, such pumps are prone to degradation—with the by-products entering the chemical stream. Finally, the use of a single pressure vessel for delivery means that delivery is not continuous, but is rather constrained to "batch" sizes based on the size of the pressure vessel. If demand exceeds the volume of the pressure vessel, further delivery must be "queued" while the pump refills the pressure vessel. Alternatively, pressure from the pump that is equal to or greater than the pressure of the delivery vessel must be applied to the delivery vessel to supplement or refill it during demand; this further compounds the filtration and maintenance problems.

Accordingly, it is a primary object of the present invention to provide a chemical transfer and delivery apparatus and method which effectively transfers high-purity process chemicals from any bulk source and delivers them accurately and without contamination to end-use stations.

It is an additional object of the present invention to provide such a transfer and delivery system which provides even flow at a consistent velocity so to permit accurate filtration and to minimize mechanical shock in the system.

It is a further object of the present invention to provide such a transfer and delivery system which does not employ pumps or other transfer apparatus which are subject to degradation or maintenance problems and which employs a minimum of any other moving parts which may be subject to degradation.

It is yet another object of the present invention to provide such a transfer and delivery system which has multiple flow paths so to provide virtually unlimited delivery capacity and built-in redundancy to avoid complete system shut down in instances of failure of a component of the system.

SUMMARY OF THE INVENTION

The present invention provides improved apparatus and method for the transfer and delivery of high purity chemicals from any bulk source to multiple end-use stations.

The invention comprises using a vacuum system and a pressure system to alternately decompress and pressurize a chemical storage vessel. By creating a vacuum in the vessel, chemical can be drawn from the bulk source to the vessel; by creating a pressure in the vessel, chemical may then be delivered to the end-use station or to one or more intermediate vessels capable of being pressurized. The use of multiple vessels allow simultaneous delivery of chemical to end-users and refilling of the vessels; this provides essentially unlimited continuous chemical delivery.

By employing multiple alternating pressure vessels and multiple flow paths, the present invention readily lends itself to many options. These include assuring continuous flow to endusers, providing recirculation and re-filtration during periods of low use, and providing redundancy to insure continued delivery of chemicals even when there is a failure of one or more components in the system. Microprocessor control of the system is readily implemented to provide accurate and instantaneous monitoring and control over all facets of chemical transfer and delivery.

The present invention completely eliminates the need for in-line pumps which are prone to degradation, contamination, and maintenance problems. Moreover, the present invention provides even, controlled flow in the system which greatly reduces maintenance problems and is extremely conducive to accurate filtration and fluid delivery.

A completely closed transfer and delivery system is provided with the present invention which vastly reduces the need to handle any chemicals from initial delivery in a bulk source to dispense of the chemicals at the end-use station. This avoids both health problems for workers and further potential contamination problems.

DESCRIPTION OF THE DRAWINGS

The operation of the present invention should become apparent from the following description when considered in conjunction with the accompanying drawings, in which:

FIG. **1** is a schematic representation of the preferred embodiment of the apparatus of the present invention; and

FIG. **2** is a schematic representation of one embodiment of the control apparatus for the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides improved apparatus and method for accurately and effectively transferring any form of high purity chemical from a bulk source to an end-user station.

As is shown in FIG. **1**, chemical is delivered to an industrial site in a bulk source container **10** of a volume from 5 to 800 or more liters. The bulk source **10** may take the form of a drum or other sealed container with a top, side, or bottom opening. Generally the bulk source **10** has one or more openings in its top, making suction removal of chemical from the bulk source in an upright position the most convenient method of transfer. A source conduit **12** or **14** is inserted into and attached to the bulk source **10** in any known manner to provide for the withdrawal of chemical. Naturally, if a top-opening source **10** is employed, the source conduit **12, 14** should be long enough or include adequate extensions to pass to the bottom of the bulk source **10** to assure the complete removal of chemical from the source. Multiple source conduits **12, 14** are preferred to permit alternating between chemical bulk sources **10** to assure continuous transfer. Each of the source conduits **12, 14** are provided with a fluid handling valve **16** and **18**, respectively.

It should be appreciated that as employed throughout this description, all fluid handling valves may be constructed from any suitable material appropriate for particular design specifications and the chemicals employed. As will become apparent, it is important that such valves are capable of handling both liquid and gas fluids and, preferably, fluid pressures of 0 to 100 psig. Diaphragm-type valves constructed of perfluoroalkoxy (PFA) TEFLON® (i.e. polytetrafluoroethylene) or similar material are preferred.

The source conduits **12, 14** then combine to a main source conduit **20** and pass through valves **22** and **24** and into a lifting vessel **26** via upper port **28** and lower port **29**. The lifting vessel **26** is also provided with an

opening **30** to attach to a vacuum system **32**, and an opening **34** to attach to a pressure system **36**. The vacuum system is controlled by valve **38**, and the pressure system **36** is controlled by valve **40**. For proper operation, a level sensor **42** should be provided to assure accurate monitoring of the fluid level in the vessel **26**. A capacitive-type level sensor is preferred. The lifting vessel **26** should also be provided with a pressure release valve **44**.

Again, it should be appreciated that as employed throughout this description, all pressure relief valves may be constructed from any suitable material and in accordance with the appropriate design specifications for each installation. The preferred pressure relief valves are rupture disc type designed to burst when pressures exceed user specifications. Generally the valves should be designed to open below 100 psig.

The lifting vessel **26** may be constructed of any suitable material and of any required size. Preferably the vessel **26** is a multiple layer construction of a stainless steel tank (e.g. Type **316L** stainless steel, electropolished and passivated), an inner liner of PFA TEFLON or similar material, and an outer pressure tank of polyvinyl chloride (PVC). The vessel **26** should have a pressure operational range of 0 to 100 psig, and a vacuum operational range of 700 to 50 torr. The nominal vessel size should be approximately 20 liters; however, depending on application, vessel size may reach or exceed 900–1000 liters.

The vacuum system **32** comprises fluid handling valve **38**, gas evacuation conduit **46**, water trap/scrubber **48**, and one or more vacuum pumps **50*a*** and **50*b***. Gas is evacuated from the lifting vessel operating a pump **50** to create a negative pressure in the evacuation conduit **46** and then by opening valve **38**.

The pumps **50** may be of any construction and size necessary to create a negative pressure in the lifting vessel of 700 to 50 torr. To adequately evacuate a **20** liter lifting vessel, a vacuum pump with a 5 to 50 CFM capacity should be provided. In order to avoid needless system shut downs, it is preferred that multiple pumps be provided with automatic switching between them in the case of pump failure or need for greater gas evacuation capacity. The water trap/scrubber **48** of known construction is provided as means to remove from the exhaust through conduit **46** any chemical residues which become entrapped in the vacuum stream, and to maintain conduit **46** free of any and all contaminants which may enter the system from the pump **50**. A water inlet **52** and a water drain **54** should be provided on the trap **48** to permit periodic replacement of the scrubber water. Naturally, other filtration or scrubber means may be substituted for trap **48**.

The pressure system **36** comprises fluid handling valve **40**, a pressurized gas conduit **56**, a pressure sensor **57**, and a pressurized gas source **58**. Sufficient gas capacity should be provided to pressurize and maintain the lifting vessel at a pressure of 0 to 100 psig during chemical transfer, and preferably at a pressure of 5 to 15 psig. Although the choice of gas may varying depending on particular applications, generally any noble or inert gas may be employed, such as nitrogen, argon, helium, neon, etc. Nitrogen and argon are preferred for most applications due to cost and availability advantages. Control of the application of pressure to the lifting vessel **26** is controlled by valve **40** in conjunction with pressure sensor **57**.

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