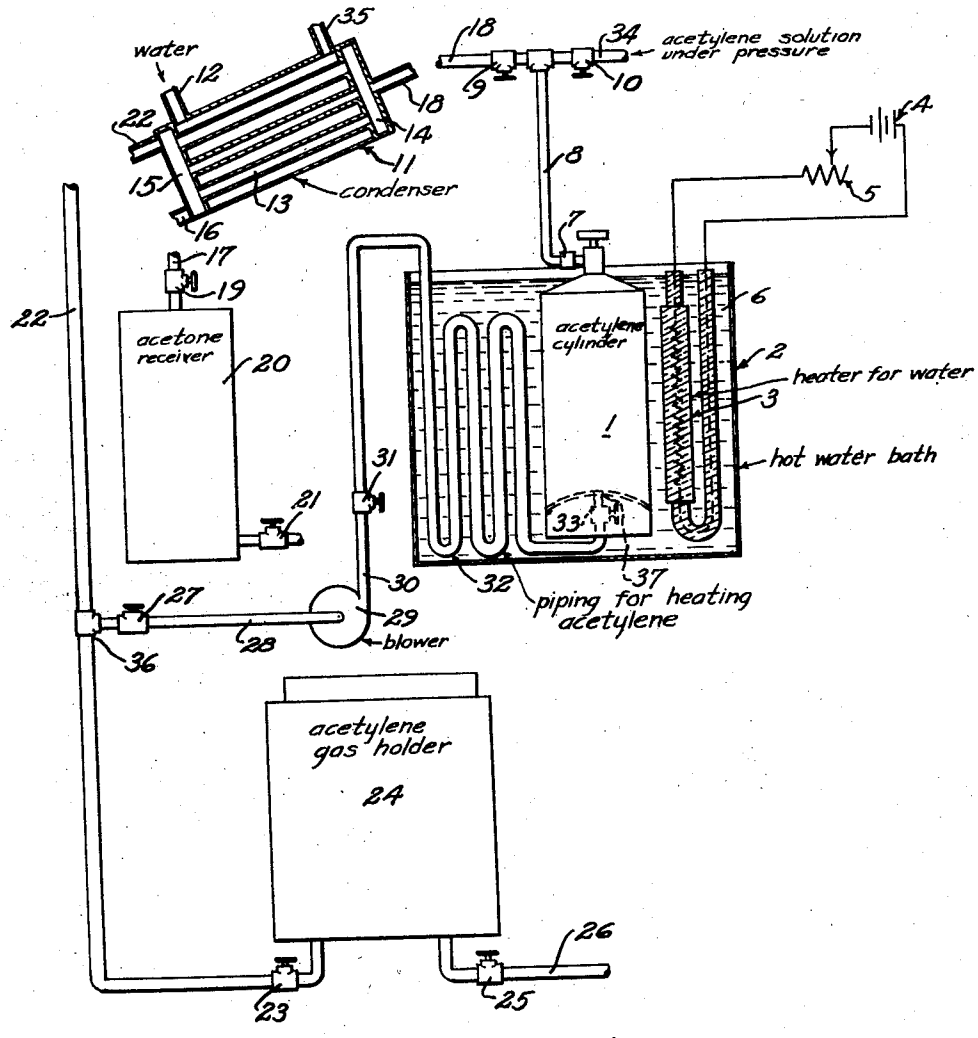
April 25, 1933. R. G. WULFF 1,906,035

METHOD OF CHARGING COMPRESSED GAS CYLINDERS

Filed Sept. 22, 1928




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Patented Apr. 25, 1933

1,906,035

# UNITED STATES PATENT OFFICE

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## METHOD OF CHARGING COMPRESSED GAS CYLINDERS

Application filed September 22, 1928. Serial No. 307,766.

This invention relates to a method and apparatus for filling ordinary commercial steel cylinders with gas and an appropriate solvent for the latter.

The object of the invention is to provide such a method and apparatus whereby the aforesaid filling operation can be conducted with more rapidity, accuracy, economy and continuity of operation than has been possible heretofore.

The accompanying drawing illustrates, more or less diagrammatically, the apparatus which is part of my invention and which I prefer to use in order to operate my method. Other apparatus may be utilized for this purpose, however.

My method is based upon the procedure of filling a gas cylinder such as an acetylene cylinder which may be entirely or partially emptied of any residual acetylene or solvent therefor, with a solution of acetylene in a solvent under suitable conditions of temperature and pressure. The solvent which I prefer to use is acetone, and such temperatures, pressures and other details as are herein given are recommended with reference to that solvent. Other acetylene solvents such as acetaldehyde, paraldehyde, pyridine, ethyl-methyl ketone, and others may also be used.

Skilled chemical engineers, when in possession of this invention, will know how to vary the temperatures and pressures to suit the various solvents.

In the operation of a filling plant, I prefer to form a large supply of charged acetone (acetone containing the dissolved acetylene), and to keep this supply in a tank cooled by mechanical refrigeration. I find that a large supply tank is a good means for providing for the continuous operation at constant rates, of all of the divisions of a filling plant.

The acetone solution of acetylene may be prepared in an apparatus similar to or identical with a modern carbonator. That is to say, that there should be provided a closed space in which intimate contact between gas and liquid may occur at a low temperature and preferably under pressure. If the acetylene has been made according to the process disclosed in my copending application Serial No. 281,406, filed May 29, 1928, the acetone solution of acetylene may be derived from the scrubbing of the partially purified (detarred) gaseous products of this process with cold acetone, with or without pressure. The scrubbing is done preferably at about −30° F. and at from 150 to 300 lbs. per square inch pressure. Either low temperatures or high pressures, or both, are also recommended for use in the carbonator type of apparatus above referred to.

In compressing the acetylene, it should always be done slowly, since rapid compressing will heat the gas unduly, thereby decomposing the acetylene. In no case should the temperature attained by compression be higher than about 350° F. and even lower temperatures should not be maintained longer than is necessary.

We have then, provided a large tank full of charged acetone, preferably refrigerated to from −20° F. to −40° F. and being saturated with acetylene at these temperatures under some suitable pressure. If less or no refrigeration is used, as is permissible, the tank must be made to resist more pressure and the necessary piping must also be mechanically strong and tight under pressure.

In the drawing, numeral 1 indicates a commercial acetylene cylinder. 2 is a tank containing water, which may be heated by an electric heater 3 supplied from a source of electricity 4 and regulated by rheostat 5. 6 is an electrically insulating water-tight protector for the heating coil 3. A coupling 7 connects the neck of the cylinder with a pipe 8, which joins a pipe 18 and is controlled by a valve 9. The junction of these pipes and the pipe 34, which leads to the charged ace-

tone tank (not shown) takes the form of a T. A valve 10 is provided to control the charged acetone supply.

Along the pipe 18 there is a condenser 11, inclined nearly to 45°, which comprises an outer shell, vestibule spaces 14 and 15, a plurality of tubes 13 connecting the vestibule spaces, a cooling water inlet 12, a cooling water outlet 35, a gas outlet 22 and an acetone outlet 16. The latter connects with a pipe 17 leading to an acetone receiver 20. A valve 19 controls the flow in the pipe 17. A suitably controlled outlet 21 is provided for the receiver 20. The gas outlet 22 continues as a pipe to an acetylene gas-holder 24, a valve 23 being provided to control the flow. A pipe 28 connects with the pipe 22 at 36. In the pipe 28 there is a valve 27 allowing ingress of gas into the pipe and into a blower 29, the exit of which is a pipe 30. The latter connects with a coil 32, located in the water bath 2, by which it may be heated. A valve 31 controls the output of the blower. The coil 32 is in communication with the interior of the cylinder 1 through an opening 37 in the bottom thereof. A regulating valve 33 is provided at the bottom of the cylinder to throttle the flow of gas into said cylinder.

From the gas-holder 24 a pipe 26, having thereon a valve 25, leads to a suitable apparatus in which the solution of acetylene in acetone may be made.

The operation of the apparatus is as follows:

First, if cylinders which have previously been used for acetylene are to be filled, they are emptied of any residual acetone or acetylene which may be present. To do this, a cylinder is connected at 37 and with the pipe 8 by the union 7. The ordinary exit valve of the cylinder and the valve 33 are opened before the cylinder has become as warm as the water bath, which has meanwhile been heated by the heater 3. The heating capacity of the heater 3 is such that the temperature of the cylinder will not rise very much above the boiling point of acetone (56° C. at ordinary temperature and pressure) and will not allow it to drop below 56° C. The valves 10, 21 and 25 are closed and the valves 9, 19, 23, 27, and 31 are opened. The cooling water is turned on through the pipe 12. The blower 29 is set in operation. The warm acetone and acetylene are then carried out of the cylinder through the pipe 8 and 18 into the condenser 11 where the acetone vapor is condensed and the resulting liquid flows out of the pipe 16 into the receiver 20. The acetylene makes its exit from the condenser through the pipe 22 and is partially recirculated through the pipe 28 and the blower 29 to the cylinder and so again to the condenser. The excess, however, enters the gas-holder 24, the valve 23 of which is opened as soon as any material pressure builds up in the circulating system.

The cylinder 1 is now warm, full of acetylene and empty of acetone. The current of the heating coil 3 is cut off and the water bath allowed to cool. In order to hasten the cooling process, a refrigeration of the water bath may be practiced, and a cooling coil, provided for the purpose. The mode of procedure would be to run out the hot water and replace it with cool, and, if convenient, refrigerate the cool water. The hot water may be refrigerated instead of being changed, but such procedure is usually wasteful.

During the cooling, the ordinary valve at the neck of the cylinder is closed and the valve 33 is also closed. The result of this manipulation is that the cylinder, when cool encloses a partial vacuum, which facilitates introduction of the charged acetone into the empty tank. The neck valve is thereupon opened and the valve 10 is opened, the valves 9 and 33 being closed. Charged acetone then flows through the pipes 34 and 8, (in which it may be metered) into the cylinder 1. The latter is then closed, disconnected at 7 and below the valve 33 and then is ready for use or transportation.

It is advantageous in certain cases, for instance, in handling cylinders having badly seating valves, to avoid vacuum formation during or after the cooling of the cylinder. By such avoidance the inward leakage of air into the cylinder is overcome. The operation is accomplished by injecting a few pounds pressure of gaseous acetylene into the cylinder while the latter is still hot from the emptying operation. The pressure so brought about should be approximately sufficient to create only atmospheric pressure in the cylinder after it cools.

I am aware that gas cylinders are usually provided with plugs containing easily fusible metal. If circumstances make it necessary that the temperature of such plugs be kept very low, they may be refrigerated by being sprayed with cold water, whether exposed or submerged in the bath, or by other methods of local refrigeration.

In the usual way of filling acetylene cylinders with gaseous acetylene, the heat of absorption of the acetylene in acetone must be dissipated. Interruption of the filling process is sometimes necessary to allow the cylinders to cool. In my process, no such dissipation or interruption is necessary.

Another great advantage of my method, particularly when I use acetylene derived from the process described in my copending application Serial No. 281,406, previously referred to, is that no drying of the acetylene is necessary. This acetylene is not made from carbide and water is not necessarily used in its production, therefore the latter does not ordinarily have to be eliminated.

In the case where the acetylene from such a process is used, it is an economy to charge the cylinders with the acetone solution of acetylene derived by scrubbing the gas which the process produces. Storage of the gas over water in a gas-holder is eliminated, whereby the necessity for compressing and re-drying the gas is avoided.

There are several modifications of my charging process. The first is as follows:

If the cylinder has not a great enough deficiency of acetone, or if it has no such deficiency, some of the acetone may be drained out and then charged acetone immediately admitted, the latter having such a concentration of dissolved acetylene, that by the time enough acetone has entered to make up the cylinder content of pure acetone to the value desired, it will also be fully charged with acetylene.

By a second modification very concentrated solutions of acetylene in acetone at very low temperatures and high pressures may be used. In my researches I have found that at very low temperatures the solubility of acetylene in acetone increases very sharply. Therefore, it is sometimes entirely feasible to charge an entire cylinder with merely a pound or two of acetone solution which is very cold and which is under high pressure. For instance I may use a solution approaching pure liquid acetylene in composition for this purpose which is at about −80° F. to −112° F. in temperature and under 300 lbs. per square inch pressure.

Of course, as in the first modification, acetone may be drained from the tank to create an acetone deficiency and these highly concentrated solutions may be supplied in larger amounts. I regard a temperature of −20° F. and a pressure of 150 lbs. per square inch as the most moderate temperatures and pressures which it is feasible to employ in this method.

The third modification is as follows:

Knowing the content of acetylene and acetone in the empty or partially empty cylinder according to present methods of determining the same, I can force in the said cylinder a given quantity of acetone charged with acetylene to a greater degree than that usually contained in the standard charged cylinder, then allow time for the diffusion of the acetone admitted and for its complete admixture with the acetone already in the cylinder and for equalization of temperature within the cylinder, and then draw off acetone until the content of acetone in the cylinder on the basis of pure acetone is as desired in the fully charged cylinder. If in this method the quantity of charged acetone forced into the cylinder and the content of acetylene per unit of acetone are balanced properly with the quantity of acetone and acetylene already in the cylinder, the resulting proportion of total acetone and total acetylene in the cylinder can be made to be the same as that wanted in the charged cylinder ready for distribution, so that the additional step of draining said cylinder after the products introduced are thoroughly mixed with what the cylinder already contained to make the total weight desired, will condition the cylinder as to content. When filling is done in this manner, it may be said that the total volume of acetone in a cylinder is ordinarily about one cubic foot when the total fillable space is in the neighborhood of three cubic feet.

There is therefore room for introduction of an appreciable amount of charged acetone in addition to the acetone already in the empty cylinder, even though there is not an initial deficiency of depleted acetone from what is wanted in the filled cylinder on the basis of pure acetone.

The fourth modification is as follows:

I may place an auxiliary valve in the bottom of the cylinder and using the ordinary valve also at the top, flush through said cylinder acetone of such a content of acetylene, so that when a sufficient volume of said charged acetone has been run through the cylinder to wash out or carry out all of the original acetone in the cylinder, I will have in said cylinder charged acetone of the same content of acetylene, substantially as that with which I am flushing. Using acetone, therefore, which is charged to the same degree per unit of acetone as is found in the standard charged cylinder, as a flushing fluid, for said flushing operation it is only necessary to drain said cylinder until the weight it shows will be that of the proper content of acetylene plus acetone, which will be known from this procedure.

The fifth modification is as follows:

I place the empty or nearly empty cylinder in a bath of hot water which, if it is held above the boiling point of acetone, will cause said acetone to boil out. The cylinder is in the meantime connected to the inlet end of a condenser cooled with water to about room temperature. The cylinder valve is opened and the end of the boiling action is awaited. The acetone can be effectively removed without great loss of time by this means. If there is also some acetylene to be recovered from the cylinder, the outlet end of the condenser which recovers the acetone boiled out, besides being connected to a receiver to catch and store said condensate for use, will also be connected to a gas-holder or other suitable receiver for storing said acetylene for further use. This acetone and acetylene will eventually find its way back to another cylinder in any one of several ways. They may for instance be pumped in proper proportion into the storage tank for charged acetone or into the apparatus used to absorb acetylene in acetone( carbonato-). Or, they may first

be purified and then so introduced. Or they may be mixed, that is, the acetylene may be mixed with the gas entering the acetone scrubber for absorption or stripping and the acetone may be mixed with the other acetone being sprayed into the top of said scrubber.

The acetylene cylinder having thus lost all its acetone and acetylene except for a small gaseous residuum thereof, it is then removed from the water, both for closing the valves and disconnecting the cylinder from the condenser. The cylinder is then allowed to stand and cool. Partial condensation of acetone vapor during this cooling would cause a reduced pressure to exist within the cylinder, which is undesirable due to possible entry of air. Therefore, the apparatus may be so arranged as to admit enough acetylene gas under slight pressure into the cylinder before closing the valve and disconnecting for cooling, so that said cooling will not create pressure less than atmospheric. While the cylinder is being allowed to cool other cylinders may of course be processed in the same manner. After the cylinder which is being cooled is sufficiently reduced in temperature, it may be connected to a pump and a quantity of charged acetone forced in, which. on the basis of pure acetone will fill the cylinder with respect to this constituent. If the charged acetone so admitted has a concentration of dissolved acetylene equal to that in a standard filled cylinder, the cylinder will be also filled with acetylene by the time the desired quantity of acetone has entered. The cylinder may be weighed at this point as a check. The system, however, is a very definite method of filling when it is known that there is no residual acetone in the cylinder after treatment. The quantity of charged acetone may be accurately metered by a meter or by a displacement pump which will automatically give a definite number of strokes. The charged liquid may further be held at a standard temperature to avoid errors due to the expansion of the liquid charged acetone due to temperature variation.

It may be said with equal applicability to all the modifications of this invention that the acetylene content of the charged acetone, the temperature of the solution and the pressure thereof may at all times be so regulated in order that definite and proper ratios exist between the components of the solution, so that when a definite quantity of liquid is added a definite quantity of acetylene will accompany it.

While I have in the foregoing matter described my processes with particular reference to acetylene, this gas is by no means the only one which may be so handled. The following list gives a number of gases which can be used instead of acetylene, and the appropriate solvent for each of the gases is also given in the list.

| Gas | Solvent |
|---|---|
| Carbon dioxide | Water, alcohol. |
| Carbon monoxide | Alcohol. |
| Nitrous oxide | Alcohol. |
| Phosphine | Alcohol. |
| Stibine | Alcohol. |
| Carbon oxysulfide | Alcohol. |
| Hydrogen chloride | Water. |
| Hydrogen bromide | Water. |
| Hydrogen iodide | Water. |
| Chlorine monoxide | Water. |
| Chlorine dioxide | Water. |
| Sulfur dioxide | Water. |
| Hydrogen sulfide | Water. |
| Cyanogen | Water. |
| Allylene, $CH_3CCH$ | Water, acetone. |

In the case of corrosive gas, acid-proof linings must be used in the cylinders. In general, my invention is applicable to any gas which has a solubility in a solvent at greater concentration per unit of volume than in the compressed gas alone at any one temperature and pressure.

I claim as my invention:

1. The process which comprises simultaneously curing the solvent and gas deficiency of a compressed gas cylinder which comprises injecting into a cylinder having such deficiency a very concentrated solution of such gas in such solvent, said solution being very cold and being under high pressure.

2. A process according to the preceding claim in which the gas is acetylene.

3. A process according to claim 1 in which the gas is acetylene and the solvent is acetone.

4. A process according to claim 1 in which a solvent deficiency is first created and in which the pressure on the solution is 150 lbs. per square inch or over and the temperature of said solution is −20° F., or below.

5. A process according to claim 1 in which a solvent deficiency is first created and in which a solution of acetylene in acetone is used, the temperature of which is −20° F. or below and the pressure on which is 150 lbs. per square inch or above.

6. A process according to claim 1 in which the temperature of the solution is at least as low as −80° F., and the pressure on which is at least as high as 300 lbs. per square inch.

7. A process according to claim 1 in which the gas is acetylene and the solvent is acetone and the temperature of the solution is at least as low as −80° F. and the pressure on which is at least as high as 300 lbs. per square inch.

8. The process of eliminating residual liquid gas solvent from compressed gas containers which comprises heating the container, allowing exit of the gas and solvent, separating the gas from the solvent, recirculating the gas through the cylinder while the latter is still hot, to eliminate the solvent approxi-

mately completely, then closing the cylinder and allowing it to cool.

9. A process of recharging a gas container which comprises heating the container, allowing exit of the gas and solvent, separating the gas from the solvent, recirculating the gas through the cylinder to eliminate the solvent approximately completely, then recharging the container with a solution of predetermined composition of the gas in the solvent, the quantity of said solution being sufficient to provide within the container the amounts of gas and solvent as are customarily found in fully charged commercial cylinders.

10. A process according to claim 9 in which the gas is acetylene.

11. A process according to claim 9 in which the gas is acetylene and the solvent is acetone.

12. The process of eliminating residual liquid gas solvent from compressed gas containers which comprises heating the container, allowing exit of the gas and solvent, separating the gas from the solvent, recirculating the gas through the cylinder while the latter is still hot, to eliminate the solvent approximately completely, charging a small quantity of gas into the cylinder before the latter cools, to avoid substantial vacuum formation, then closing the cylinder and allowing it to cool.

In testimony whereof, I have hereunto set my hand at Los Angeles, California, this 27th day of August, 1928.

ROBERT G. WULFF.