

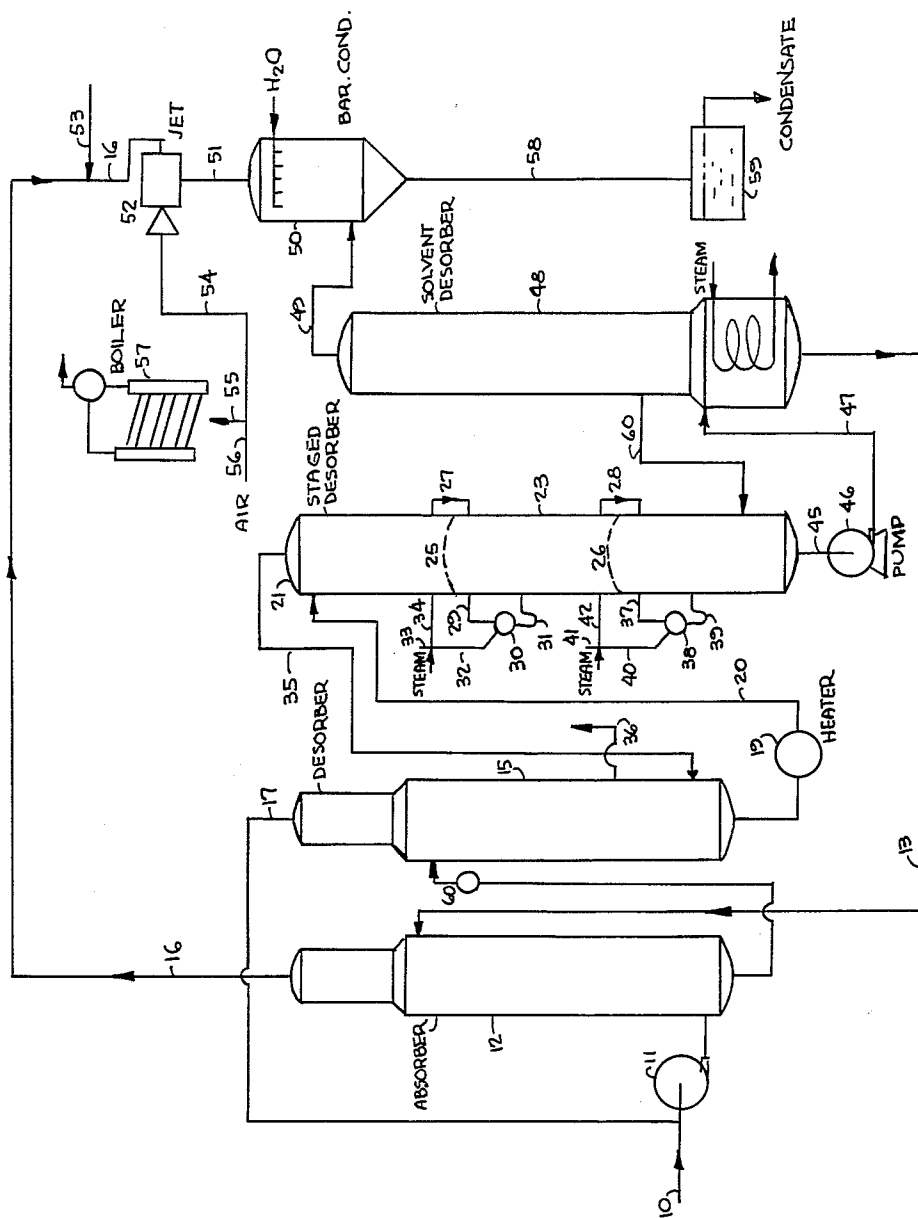
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CHEMICAL PURIFICATION PROCESS FOR ACETYLENES

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CHEMICAL PURIFICATION PROCESS FOR
ACETYLENES

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6 Claims. (Cl. 183—115)

This invention relates to a chemical purification process whereby acetylene polymers and other highly unsaturated compounds such as allylene, allene, etc. are removed from acetylene containing the same.

In the production of acetylene by high temperature processes involving the cracking or partial combustion of gaseous hydrocarbons it is known that small quantities of acetylene polymers such as diacetylene, and other highly unsaturated compounds such as allylene, allene, etc. are concurrently formed. Such polymers are spontaneously explosive and inflammable and constitute a distinct and major hazard in connection with the handling of gases and solutions containing the same. In the large scale manufacture of acetylene by cracking or partial combustion of gaseous hydrocarbons serious explosions and fires have resulted from the improper handling of these compounds. Such compounds as diacetylene, allylene and allene are collectively referred to herein as acetylene polymer impurities.

It is an object of the present invention to provide a safe method for the removal of such explosive and inflammable polymers from acetylenic gases or solutions containing the same and to render the same innocuous.

A number of oxygen-containing organic solvents suitable for the concentration of acetylene are known. Among these are esters of carbonic acid, e. g., diethyl carbonate, liquid aliphatic ketones, e. g., cyclohexanone, polyglycols and ethers or esters thereof, lactones, e. g., gamma butyrolactone and its homologues, dimethyl formamide, dimethyl acetamide, etc. These solvents are more or less selective in nature, that is they have the property of dissolving acetylene and acetylene polymers in relatively large volume, without dissolving the non-acetylene gases such as hydrogen, carbon monoxide, carbon dioxide and methane to any great extent.

Of particular interest in the present invention are those oxygen-containing organic solvents having a boiling point which is higher than that of water and which, furthermore, are miscible with water. It is not necessary that these solvents be miscible with water in all proportions, on the other hand if the solvent is miscible to the extent that it dissolves up to 10% or 15% by weight of water under the conditions employed in the process the material is satisfactory for the present purpose.

In the present process, as also in prior known processes, the dilute acetylene-containing gases are compressed and then passed into contact with an organic solvent for the acetylene. The solution thus formed containing dissolved acetylene is then subjected to desorption which may take place at progressively lower pressures and with or without being subjected to increasing temperatures. Since the desorption of the solvent is carried out stepwise, in a series of stages or zones wherein the solvent is subjected to progressively lower pressures (and if desired subjected to correspondingly higher temperatures), the gases evolved are compressed and returned to the next higher pressure zone. A process of this type is disclosed in U. S. Patent 2,250,925.

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In accordance with this invention gas mixtures comprising acetylene mixed with non-acetylenic gases and containing acetylene polymers are first compressed and then scrubbed with an oxygen-containing organic compound which has a boiling point above that of water and which, furthermore, is miscible with water to the extent of up to 10% to 15% by weight. Such scrubbing results in the production of a solution containing dissolved gases of which acetylene is present in greatest quantity. The solution also contains minor quantities of the associated non-acetylenic gases as well as acetylene polymers present in the original mixture. The pressure upon the solvent is reduced, the contained acetylene is evolved in concentrated form under said reduced pressure with the aid of heat, if necessary, after which the solvent still containing the acetylene polymers is subjected to distillation, the polymers vaporized and entrained with a high velocity jet of combustible gas, which gas is maintained out of contact with the said solvent. By this procedure a substantially complete removal of the volatile acetylene polymer is obtained without loss of solvent while avoiding the hazards occasioned by the handling of the polymers in concentrated form.

In a further modification of this invention the solvent which has been saturated with acetylene under pressure is treated for evolution of contained acetylene by injecting steam into said solvent under conditions such that at least a part of the steam is condensed to water, the latter being miscible with the solvent, the pure acetylene is withdrawn and the solvent containing the acetylene polymer then subjected to vaporization of both acetylene polymers and water vapor. The water vapor so released may be partly condensed, if desired, and the acetylene polymer still in the vapor state is drawn into a high velocity jet of combustible gas without coming into contact with said solvent, after which the gas mixture so formed is burned.

In a still further modification of this invention the pressurized non-acetylenic combustible gases, after separation from the scrubbing solution, are used to actuate a vacuum jet for withdrawing the acetylene polymer from the solution and to dilute the same so as to render the said polymers innocuous.

The invention will be understood from the following description when read in connection with the accompanying drawing showing in schematic form an assemblage of apparatus suitable for the practice of the process.

In the drawing, numeral 10 indicates a source of dilute acetylene-containing gas, 11 a pump and 12 an absorber in which gas and liquid are brought into contact under the pressure supplied by the pump. Item 15 is a desorber, 19 a heater, while 21 is a staged desorber, the function and operation of which are more particularly described and claimed in copending application Serial No. 254,387, filed November 1, 1951, which application is assigned to the same assignee as is the present case.

Staged desorber 21 essentially consists of three separate stages or zones numbered 22, 23 and 24, each stage containing gas-liquid contact means such, for example, as bubble plates or packing material. In the device shown in the drawing the separate stages or zones are formed by dividing a vertical, cylindrical vessel by means of internal diaphragm plates 25 and 26. Means for the flow of liquid from one stage to the next lower stage is provided by pipes 27 and 28. The separate stages of this absorber are usually operated under different pressures, the upper stage 22 being maintained at the higher pressure usually at or slightly above atmospheric pressure, intermediate stage 23 at about 500 mm., while the last stage 24 is operated at about 200 mm. pressure. It will be obvious that any number of stages

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may be employed for this process, the three shown herein constituting a practical number.

The gases liberated in each stage of the desorber are removed from a lower pressure stage and injected into the next higher pressure stage by means of steam jets. Specifically the gases liberated in stage 23 are withdrawn through pipe 29 into separator 30, thence into pipe 32 and by means of steam jet 33 injected into stage 22 through pipe 34. By this means the gas pressure in stage 22 is maintained sufficiently high so that a flow of gas through line 35 into desorber 15 will take place. The pure concentrated acetylene is withdrawn at pipe 36. Any liquid drawn into pipe 29 with the gas enters separator 30 and is returned to the stage by pipe 31. Likewise, the gases liberated in stage 24 are withdrawn through pipe 37 into separator 38, thence into pipe 40 and by means of steam jet 41 are injected into stage 23 through pipe 42. Any liquid which is withdrawn with the gases into separator 38 is returned to stage 24 by pipe 39.

Line 45, pump 46 and line 47 convey the liquid solvent into solvent desorber 48. This device is provided with steam heating means in the base thereof and with gas-liquid contact means in the tower portion. A pipe line 49 connects the top of the tower portion with barometric condenser 50 which is provided with barometric leg 58 extending into condensate sump 59.

Pipe 51 connects the barometric condenser with vacuum jet 52 actuated by pressurized gas entering through pipe 16. Normally the pressurized gas is supplied by line 16 from the absorber 12 off gas, however vacuum jet 52 may also be supplied with pressurized natural gas supplied by pipe 53. The gas mixture supplied by the jet flows through line 54 to burner 55 where it meets air supplied by pipe 56. Combustion of the gas mixture then takes place under boiler 57.

Example

A gas mixture obtained by the partial combustion of methane with oxygen containing the following constituents in mole per cent:

	Per cent
C ₂ H ₂ -----	8.7
CH ₄ -----	4.4
CO -----	26.0
CO ₂ -----	4.0
H ₂ -----	54.5
Argon, acetylene polymers, etc. -----	Balance
	100.0

is compressed to 100 p. s. i. g. in compressor 11 and is then scrubbed in absorber 12 with a solution of dimethyl formamide containing about 1% of water. A solution containing approximately 1.2% by weight of acetylene together with minor amounts of methane, carbon monoxide, carbon dioxide and hydrogen, as well as diacetylene, and other acetylene polymers is obtained in the absorber and passed through pipe 14, valve 60 and into desorber 15. An overhead gas containing not more than 0.2% of acetylene leaves absorber 12 by pipe 16.

In desorber 15 the pressure of the gas in contact with the solution is reduced to approximately atmospheric pressure, the temperature being about 80° F. to 100° F. with the result that the major portion of the less soluble non-acetylenic gases is flashed off. These less soluble gases then flow through pipe 17 and are returned to the scrubber 12.

The dimethyl formamide solution leaving the desorber 15 by pipe 18 passes through heater 19, wherein the temperature is raised to approximately 200° F. and then conducted into zone 22 constituting the upper part of staged desorber 21. The pressure in this zone is about 7 p. s. i. g. The gases flashed from the solvent in this zone contain about 98.8% of acetylene and are returned to the base of desorber 15 by means of pipe 35.

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The solution treated in zone 22 flows through pipe 27 into zone 23, wherein the pressure is about 500 mm. The gases in this zone are withdrawn through pipe 29, passed into separator 30, thence into pipe 32 connected with steam injector 33. Injector 33 is supplied with high pressure steam and the mixed steam and acetylene passed through pipe 34 into zone 22. Any liquid collecting in separator 30 returns to zone 23 by means of pipe 31. The solution treated in zone 23 flows through pipe 28 into zone 24, wherein the pressure has been reduced to about 160 mm. and the temperature maintained at about 190° F. The gases liberated in this zone are withdrawn through pipe 37, passed into separator 38, thence into pipe 40 and are drawn into steam injector 41 by the action of steam applied to the injector. The mixture of steam and acetylene passes through pipe 42 and then into zone 23. Any condensate collecting in separator 38 is returned to zone 24 by means of pipe 39.

At this point in the process the solvent has been substantially completely freed of acetylene. It, however, still contains the more soluble acetylenic polymers and also contains the water which was originally present together with that water resulting from the condensation of steam employed for actuating injectors 33 and 41.

The solution is next pumped to solvent desorber 48 by means of pump 46 and pipe 47, wherein it is subjected to a temperature of about 240° F. at a pressure of 200 mm. Under these conditions approximately one-half of the contained water and most of the diacetylene present in the solution are volatilized. A side stream consisting of water vapor flows by pipe 60 into stage 24 and serves to liberate dissolved gases present in the solution therein. The solvent containing about 1% of water is returned to the absorber for reuse in the process. The evolved vapors enter barometric condenser 50, are subjected to a cooling water spray whereby the water vapor present is condensed. The uncondensed diacetylene is drawn into jet 52 in which device it is mixed with non-acetylenic gases supplied by pipe 16. By reason of the jet action the acetylenic polymers are mixed with the non-acetylenic gases, passed into pipe 54 and conducted to burner 55. At this point they are mixed with the necessary proportion of air and burned under boiler 57. The output of the boiler is used to furnish steam for the steam jets 33 and 41 and for other purposes. If desired, additional combustible gas such, for example, as natural gas under pressure may be admitted to pipe 16 through pipe 53 and employed for actuating the jet 52.

There are certain features and sub-combinations disclosed herein which are of utility without reference to other features and sub-combinations. Such features are contemplated by and are within the scope of the appended claims. It will also be obvious that certain changes and modifications may be made in the details without departing from the spirit of the invention.

What is claimed is:

1. In the process for removing acetylene polymers from an organic oxygen-containing solvent containing said polymers and dissolved water, said solvent having a boiling point above that of water, the steps of vaporizing water simultaneously with said acetylene polymers from said solvent, condensing at least a part of said water and then mixing said acetylene polymers with a high velocity jet of combustible gas.

2. The process for purifying an acetylenic gas containing acetylene, acetylene polymer impurities and substantial quantities of non-acetylenic gas which comprises compressing said acetylenic gas, scrubbing said compressed gas with a solvent for acetylene comprising an organic oxygen-containing solvent and water, to form a solution of acetylene and acetylene polymers and a residual, substantially insoluble non-acetylenic gas, wherein said solvent has a boiling point above that of water and contains not in excess of 15% of water, reducing the pressure on said solution and removing contained acetylene there-

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from, separately vaporizing from said solution a mixture of said acetylene polymers and water vapor, condensing said water vapor and mixing said acetylene polymers with a high velocity jet of combustible gas.

3. The process for purifying an acetylenic gas containing acetylene, acetylene polymer impurities and substantial quantities of combustible non-acetylenic gas, which comprises compressing said acetylene gas, scrubbing said compressed gas with a solvent for acetylene and acetylene polymers comprising an organic oxygen-containing solvent and water, to form a solution of acetylene and acetylene polymers and a residual, non-acetylenic gas under super-atmospheric pressure and wherein said solvent has a boiling point above that of water and contains not in excess of 15% of water, reducing the pressure on said solution and removing contained acetylene therefrom, separately vaporizing from said solution a mixture of said acetylene polymers and water vapor, condensing at least a part of said water vapor and mixing said acetylenic polymers with a high velocity jet of said combustible non-acetylenic gas.

4. The process for purifying an acetylenic gas containing acetylene, acetylene polymer impurities and substantial quantities of combustible non-acetylenic gas, which comprises compressing said acetylenic gas, providing an organic oxygen-containing solvent for said acetylene, said solvent containing water miscible therewith, scrubbing said compressed gas with said solvent for acetylene and acetylene polymer impurities to form a solution thereof and a pressurized residual gas, reducing the pressure on said solution and removing dissolved acetylene therefrom, separately vaporizing said acetylene polymer impurities and water, condensing at least a part of said water, reducing the pressure on said pressurized residual gas and thereby producing a high velocity jet of said residual gas and drawing said acetylene polymer impurities into said high velocity residual gas jet.

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5. In the process for removing acetylene polymers from an organic oxygen-containing solvent containing dissolved water, said solvent having a boiling point above that of water, the steps of vaporizing water and said acetylene polymer by the application of heat thereto, condensing at least a part of said water and then mixing said vaporized acetylene polymers with a high velocity jet of combustible gas.

6. In the process for removing acetylene polymers from an organic oxygen-containing solvent containing said polymers and dissolved water, said solvent having a boiling point above that of water, the steps of vaporizing water simultaneously with said acetylene polymers by the application of heat and diminished pressure thereto, condensing at least a part of said water and mixing said vaporized acetylene polymers with a high velocity jet of combustible gas.

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