

## UNITED STATES PATENT OFFICE

1,988,032

## PURIFICATION OF ACETYLENE

Paul Baumann, Hanns Bueckert, Ludwigshafen-on-the-Rhine, Wilhelm Sandhaas, Mannheim, and Heinrich Schilling, Ludwigshafen-on-the-Rhine, Germany, assignors to I. G. Farbenindustrie Aktiengesellschaft, Frankfurt-on-the-Main, Germany

No Drawing. Application July 23, 1932, Serial No. 624,358. In Germany July 25, 1931

8 Claims. (Cl. 260—170)

The present invention relates to the purification of acetylene.

In the production of acetylene by thermal dissociation, which expression hereinafter is to include the simple splitting at temperatures above 800° C., as well as electric treatments, in particular the treatment in high voltage electric arcs, of gaseous or vaporous or dispersed liquid or solid hydrocarbon products, hereinafter referred to as vaporized hydrocarbon products, gas mixtures are always obtained which contain not only acetylene but also small amounts of impurities, especially diacetylene, and also other strongly unsaturated compounds such as allylene, butadiene and other compounds in part not yet identified, which are injurious in the further working up of the acetylene, for example into aldehyde.

We have now found that the said impurities are readily removed from the gases containing acetylene obtained in the said thermal dissociation by washing the latter with organic liquids under such conditions of temperature and pressure that the solubility of acetylene is slight whereas that of diacetylene and other strongly unsaturated compounds is considerable; ordinarily it will be advantageous to work at ordinary temperature, but also temperatures ranging up to 150° C. or very low temperatures as for example 80° below zero C. may be suitable. The ratio of the solubilities of acetylene and diacetylene should be 1:10 or preferably still more. As suitable organic liquids may be mentioned liquid hydrocarbons, such as the higher liquid members of the paraffinic and olefinic series of hydrocarbons, as for example synthetic oils and mobile tars or mineral oils or fractions of these hydrocarbon products, as for example benzine or higher boiling oils, as middle oil or heavy oil, or also benzene, or animal or vegetable oils. Furthermore the liquid halogen substitution products of hydrocarbons, as for example hexachlorobenzene, tetrachloroethane, carbon tetrachloride, or chloroform or nitrobenzene are employed with very good success. It is of advantage to employ organic liquids having a relatively low vapor pressure, such as less than 30 or less than 10 millimetres mercury gauge, because in this case only small amounts of washing liquid are evaporated by the stream of gas to be purified. However, if a cooling expedient is arranged behind the washing plant and the evaporated portions of the liquids are condensed and returned to the bulk of the washing liquid, liquids having a higher vapor pressure may be employed with the same success.

With the said solvents the ratio of the solubilities of, for example, diacetylene and acetylene at a temperature of about 40° C. is from 20 to 80:1 so that the loss by reason of acetylene simultaneously absorbed may be kept very slight. The desired property of the solvents of withdrawing, as far as possible, only the impurities from the gases containing acetylene is enhanced by working at low temperatures, as for example between 10° and zero C. and, if desired, also temperatures below zero C., because the ratio of the amount of impurities dissolved in a given volume of solvent to the amount of acetylene simultaneously dissolved is considerably increased.

It is advantageous to carry out the washing under somewhat increased pressures because the amount of diacetylene dissolved in a given volume of solvent increases much more than proportionately with the pressure while the amount of acetylene dissolved increases approximately proportionately. However, the pressure is preferably not chosen so high that substantial amounts of acetylene are dissolved together with the other strongly unsaturated compounds. Apart from this it is a question of costs at what pressure the washing operation is carried out, as the compression of the gases requires energy. Thus the ratio of the amounts of diacetylene and acetylene dissolved increases with increasing pressure and consequently the loss of acetylene becomes less.

The dissolved impurities may be removed from the liquids by decreasing the pressure, blowing out with gases as for example nitrogen, methane or hydrogen, and/or increasing the temperature, and the liquids may then be employed again for absorption.

The following examples will further illustrate the nature of this invention but the invention is not restricted to these examples.

## Example 1

100 cubic metres of a gas containing 15 per cent of acetylene and 0.6 per cent of diacetylene and other highly unsaturated impurities are treated at 20° C. and 5 atmospheres with 0.2 cubic metre of nitrobenzene in a bell washer during the course of about an hour. The gas leaving the washer contains 0.02 per cent of diacetylene and 14.7 per cent of acetylene. This may be worked up into acetaldehyde without trouble. The nitrobenzene containing the impurities is released from pressure until it is at atmospheric pressure and is then blown with an inert gas, as for example the waste gas from the aldehyde plant. If the washing is carried out at a temperature of

40° C. somewhat more than double the amount of liquid, namely 0.45 cubic metre of nitrobenzene, is required in order to attain a similar effect. The gas leaving the washer in this case contains 14.5 per cent of acetylene.

#### Example 2

100 cubic metres of the gas specified in Example 1 are treated during the course of an hour at 20° C. and 2 atmospheres in a bell washer with a gas oil having a specific gravity of 0.88. In order to purify the gas so that it only contains 0.02 per cent of diacetylene, 2.0 cubic metres of gas oil are required. The gas oil, after the pressure thereon has been released to atmospheric pressure, is first blown with about 3 cubic metres of an inert gas as for example methane in order to recover the readily separable acetylene which has been dissolved together with the impurities. The inert gas is then returned to the crude gas in front of the gas oil washer. The loss of acetylene can thus be kept below 0.5 per cent of the total amount of acetylene. The absorption liquid, which still contains the dissolved impurities, is then completely freed from the dissolved diacetylene with about 65 cubic metres of another inert gas as for example nitrogen and the liquid is returned to the washing plant.

If the pressure in the washing tower be 10 atmospheres instead of 2 atmospheres, only from 0.2 to 0.3 cubic metres of gas oil are required in order to produce the same purification. By reason of the smaller amount of solvent employed, the loss of acetylene is still further reduced. The purified gas may be further worked up into aldehyde without trouble.

What we claim is:

1. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a thermal dissociation of a vaporized hydrocarbon product which comprises washing said gas with an organic liquid selected from the group consisting of hydrocarbon liquids, halogenated hydrocarbons and nitrobenzene at a temperature between 80° below zero C. and 150° C. and under a pressure at which at the said temperature the solubility of acetylene is by far smaller than that of the other strongly unsaturated compounds.

2. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a thermal dissociation of a vaporized hydrocarbon product which comprises washing said gas with an organic liquid selected from the group consisting of hydrocarbon liquids, halogenated hydrocarbons and nitrobenzene at a temperature between 80° below zero C. and 150° C. and under a pressure at which at the said temperature the solubility of acetylene is less than one tenth of that of the other strongly unsaturated compounds.

3. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a treatment in the

electric arc of a vaporized hydrocarbon product which comprises washing said gas with an organic liquid selected from the group consisting of hydrocarbon liquids, halogenated hydrocarbons and nitrobenzene at a temperature between 80° below zero C. and 150° C. and under a pressure at which at the said temperature the solubility of acetylene is by far smaller than that of the other strongly unsaturated compounds.

4. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a treatment in the electric arc of a vaporized hydrocarbon product which comprises washing said gas with a hydrocarbon liquid at a temperature between 80° below zero C. and 150° C. and under a pressure at which at the said temperature the solubility of acetylene is by far smaller than that of the other strongly unsaturated compounds.

5. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a treatment in the electric arc of a vaporized hydrocarbon product which comprises washing said gas with a hydrocarbon liquid at substantially ordinary temperature and under a pressure ranging from ordinary pressure to that at which the acetylene is not substantially dissolved.

6. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a treatment in the electric arc of a vaporized hydrocarbon product which comprises washing said gas with a halogenated hydrocarbon at a substantially ordinary temperature and under a pressure ranging from ordinary pressure to that at which the acetylene is not substantially dissolved.

7. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a thermal dissociation of a vaporized hydrocarbon product which comprises washing said gas with an organic liquid selected from the group consisting of hydrocarbon liquids, halogenated hydrocarbons and nitrobenzene, at a temperature between 80° below zero C. and 150° C. and under a pressure ranging from ordinary pressure to that at which the acetylene is not substantially dissolved.

8. A process for the separation from acetylene of other strongly unsaturated compounds contained in a gas issuing from a treatment in the electric arc of a vaporized hydrocarbon product which comprises washing said gas with an organic liquid selected from the group consisting of hydrocarbon liquids, halogenated hydrocarbons and nitrobenzene at a temperature between 80° below zero C. and 150° C. and under a pressure ranging from ordinary pressure to that at which the acetylene is not substantially dissolved.

PAUL BAUMANN,  
HANNES BUECKERT,  
WILHELM SANDHAAS,  
HEINRICH SCHILLING.

### DISCLAIMER

1,988,032.—*Paul Baumann* and *Hanns Bueckert*, Ludwigshafen-on-the-Rhine, *Wilhelm Sandhass*, Mannheim, and *Heinrich Schilling*, Ludwigshafen-on-the-Rhine, Germany. PURIFICATION OF ACETYLENE. Patent dated January 15, 1935. Disclaimer filed April 19, 1937, by the assignee, *I. G. Farbenindustrie Aktiengesellschaft*.

Hereby enter this disclaimer to claims 4 and 5 and to that part of claims 1 to 3, inclusive, and 7 and 8 in said specification, which is in the following words, to wit: "Hydrocarbon liquids," claim 1, line 5; claim 2, line 5; claim 3, line 5; claim 7, line 5; claim 8, line 5.

[*Official Gazette, May 25, 1937.*]

1,988,032.—*Paul Baumann and Hanns Bueckert*, of Ludwigshafen-on-the-Rhine; *Wilhelm Sandhaas*, of Mannheim; and *Heinrich Schilling*, of Ludwigshafen-on-the-Rhine, Germany. PURIFICATION OF ACETYLENE. Patent dated January 15, 1935. Disclaimer filed August 21, 1937, by the assignee, *I. G. Farbenindustrie Aktiengesellschaft*.

Hereby enter this disclaimer to claims 4 and 5 and to that part of claims 1 to 3 inclusive and 7 and 8 in said specification, which is in the following words, to wit: "hydrocarbon liquids", claim 1, lines 6 to 7; claim 2, lines 6 to 7; claim 3, lines 6 to 7; claim 7, lines 6 to 7; claim 8, lines 6 to 7.

[*Official Gazette September 28, 1937.*]