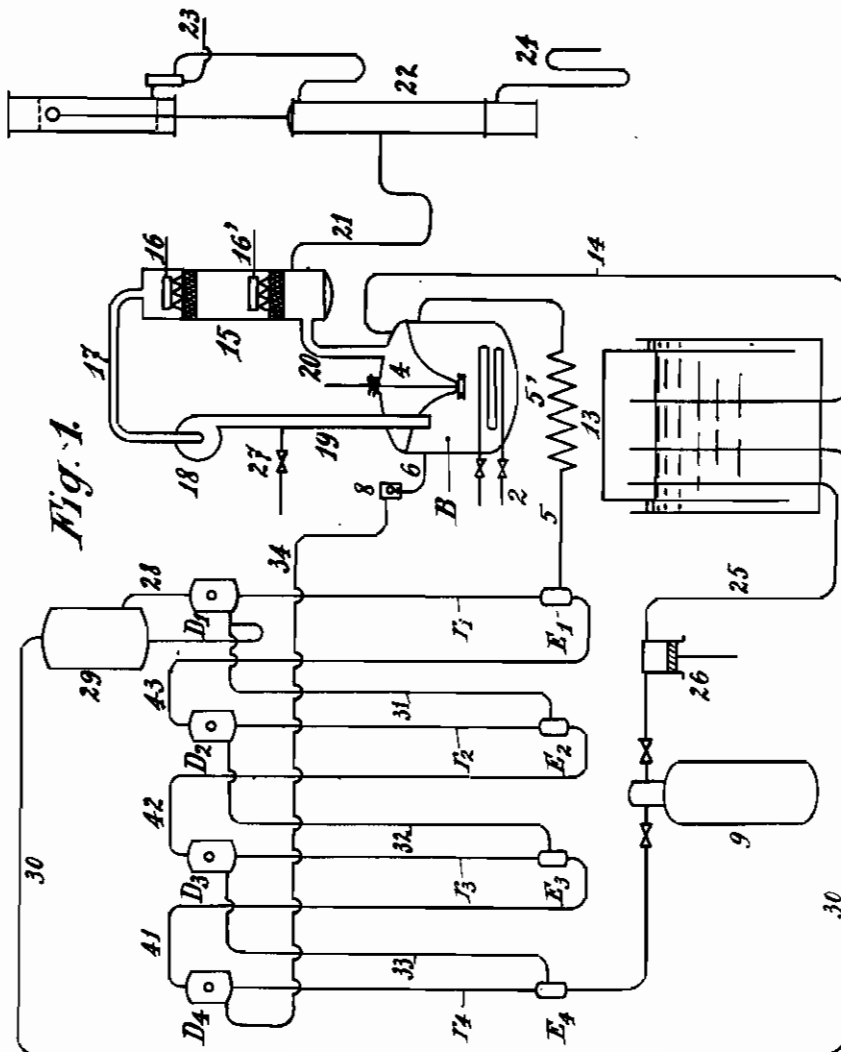


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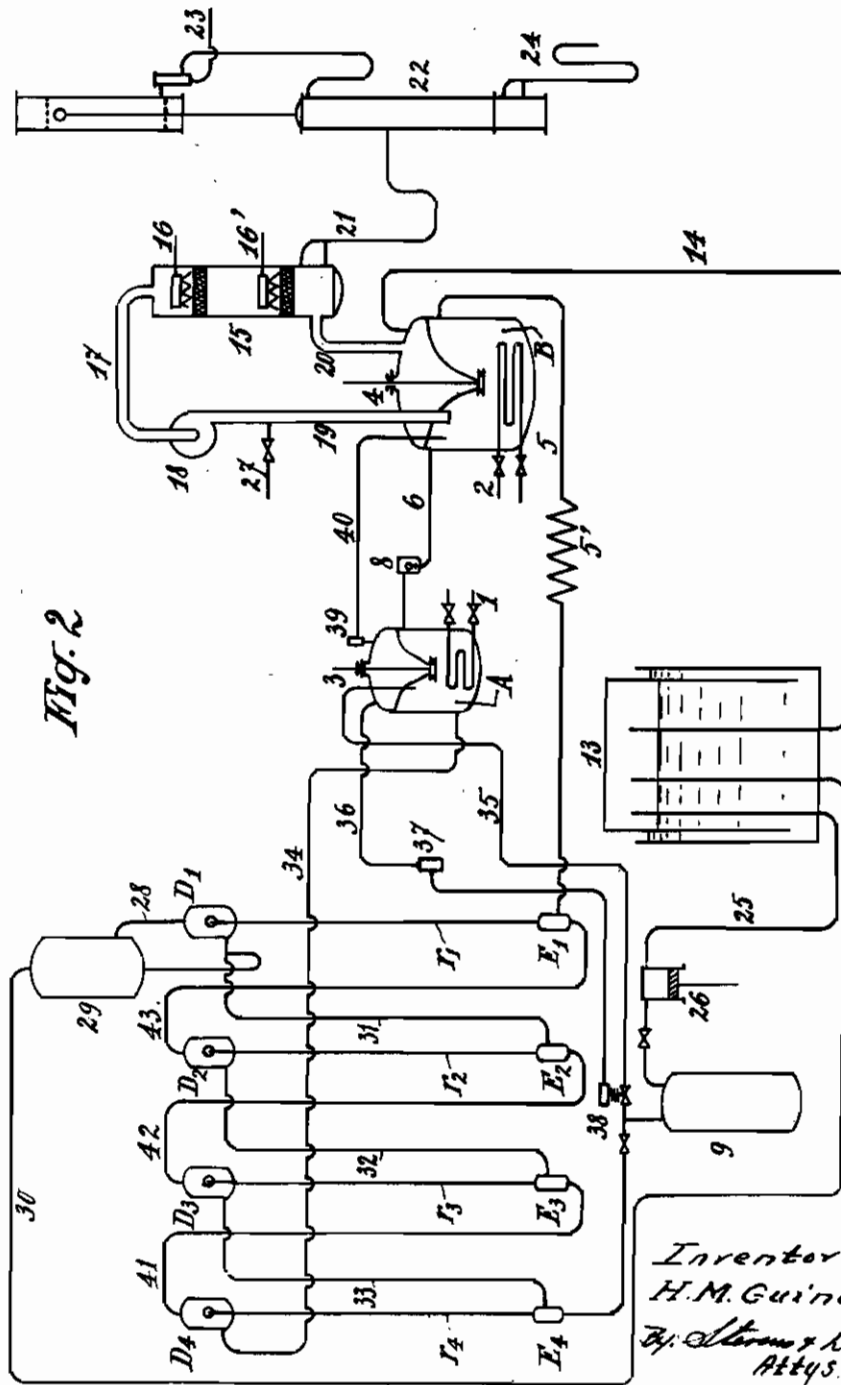


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ALIEN PROPERTY CUSTODIAN

HYDRATION OF OLEFINES

Henri Martin Guinot, Niort, France; vested in
the Allen Property Custodian

Application filed January 8, 1941

This invention relates to the hydration of olefines to form alcohols.

It is known that olefines may be hydrated to form alcohols by dissolving them in aqueous hydrating liquors from which the alcohol formed is removed by blowing through said liquors a stream of inert gas, or preferably of the actual olefine which is to be reacted.

It is also known that it is frequently desirable to operate under pressure with some olefines, the hydration of which is frequently sluggish at atmospheric pressure because it is necessary to use hydrating baths relatively rich in water in order to effect the rapid saponification of the alcohol-acid derivative transiently formed. However, the known processes are not wholly efficacious when they are made use of under pressure. As a matter of fact, such a pressure increases materially the solubility of the alcohol formed in the hydrating liquor and at the same time decreases its volatility so that at a given temperature, the difficulty of removing the alcohol from the hydrating bath increases with the pressure. Now, it is imperative that the alcohol should be removed from the hydrating bath as soon as possible since its prolonged contact, at an elevated temperature of reaction, with the hydrating liquor not only slows down the reaction, but gives rise to accessory reactions which quickly decrease the yield.

In my prior French application, filed September 14, 1939, for "Process of hydration of olefines," I have described a process which enables to avoid the disadvantages of working under pressure while retaining the advantages of such an operation. According to said process, the hydration of olefines is effected in two separate inter communicating tanks, in one of which solution and hydration of the olefine in the hydrating bath take place at elevated pressure, and in the other of which removal of the alcohol from said bath is effected at substantially atmospheric pressure. The circulation of the hot hydrating bath from the low pressure tank to the high pressure tank is obtained by means of a suitable apparatus, namely an injector operating with the olefine to be treated which has previously been raised to a convenient pressure; the hydrating bath is then returned to the low pressure tank simply by overflowing and passage through a suitable relief valve.

Now I have found that in the above described process the use of an injector is objectionable in some respect. I have particularly found that

with such a device the injection of a considerable amount of gas promotes only the entering of a low amount of hydrating bath in the high pressure tank, which results in an important consumption of power. It is also necessary to provide the high pressure tank with a stirring device of a special type allowing the gas forming the atmosphere in said tank to be finely dispersed in the hydrating bath.

An object of the present invention is to improve the above described process and apparatus by improving the circulation of the hydrating bath between the elimination tank and the hydrating tank.

Another object of this invention is to improve the manner in which olefines are brought into intimate contact with the hydrating bath, and consequently to simplify the previously known apparatus.

I have found that, to effect the circulation of the hydrating bath between the two operating tanks, it is particularly advantageous to make use of the emulsifying pump known under the commercial denomination of "Mammoth" pump provided that this is operated with the actual olefine to be treated. Said device ensures a progressive release of the driving olefine and consequently an excellent yield as compared with the injector.

Moreover, owing to the intimate contact obtained between the driving olefine and the hydrating bath, a considerable dissolution effect is obtained in the pump without any supplemental consumption of energy; this results in the hydrating bath leaving said pump being already charged with an important amount of olefine, and that proportionally decreases the work to be done by the tank under pressure, the size of which may be consequently reduced.

When an olefine is treated, such as propylene, the dissolution of which is very easy to perform, it happens sometimes that the hydrating bath is wholly saturated with the olefine when leaving the pump. In such a case, said bath may be directly forwarded, after being released, into the distillation tank, and the tank working under pressure, which is not employed, may be omitted.

Other objects and advantages of the present invention will result of the following description of some specific embodiments thereof.

The following examples, taken together with the accompanying diagrammatic drawings will make it clear how the invention may be carried into practice.

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In these drawings:

Fig. 1 shows the arrangement of the apparatus according to my invention.

Fig. 2 shows a variante of the arrangement according to Fig. 1.

The apparatus shown by Fig. 1 is employed in the most favourable case in which the hydrating bath is saturated with the olefine when leaving the pump. In case of the saturation being insufficient, use is made of the arrangement shown by Fig. 2 in which the saturation is achieved in a tank working under pressure.

Referring to Fig. 1, the tank B, which works under the atmospheric or a closely related pressure, is fitted with an heating coil 2, which may be eventually employed for cooling, and with a stirring device 4 of a special type allowing the gas constituting the atmosphere in the tank to be finely emulsified in the hydrating bath. The tank B is further over-topped by a washer-condenser 15 constituted by a tower containing filling materials sprayed with cold pulverized water coming from collars 16 and 16'. The top of this tower communicates through the pipe 17 with the intake of a fan 18 which forces back with a great speed the gas into the liquid of the tank B through the pipe 19.

Owing to this speedy circulation of olefine between the tank B and the washer-condenser 15, the alcohol dissolved in the hydrating bath is continuously carried away as vapours by the flow of olefine; said vapours which penetrate into the washer-condenser through the pipe 20 are dissolved in the washing water. The aqueous alcohol solution so obtained leaves the tower 15 through the pipe 21 and is introduced in the middle part of a small column 22 at the top of which the alcohol is collected at 23, whereas at the base, pure water flows at 24.

The injecting device is embodied by an emulsifying pump of the "Mammoth" type which is constituted by a series of emulsifiers E₁, E₂ . . . E₄ connected with gas removing chambers D₁, D₂ . . . D₄. Said pump is fed at one end by the compressed olefinic gas, and at the other end by the hydrating liquid issuing out of the distillation tank and the pressure of which must be raised. The operation of this device is as follows:

Gas cycle.—The compressed olefine issuing from the vessel 9 is introduced into the emulsifier E₄ in which it encounters the liquid coming from D₃, of which the olefine promotes carrying up to D₄ by the pipe r₄.

The gas, partially released, leaves the gas removing chamber through the pipe 41, and is introduced in the emulsifier E₃ promoting the carrying up of the liquid issuing from D₂; the gas consequently undergoes a further decompression, and so on until it reaches D₁ from which the olefine, which is substantially released, is sent through the pipe 30 in the gazometer 13 after having passed through the separator 29. Said gas is taken at 25 by the intake of the compressor 26 by which it is raised to the required pressure prior to be sent in the vessel 9.

Liquid cycle.—After being cooled at 5', the hydrating liquid freed from alcohol issuing from the tank B, enters through the pipe 6, under substantially atmospheric pressure, into the emulsifier E₁ from where it is carried up through r₁ under the action of the partially released olefine coming from D₂ through the pipe 43. In the course of this going up, the hydrating liquor dissolves a certain amount of olefine and its pressure

is increased. At D₁ the hydrating bath is separated from the emulsified gas and then flows away through 31 in the emulsifier E₂ where it encounters the relatively highly compressed olefine coming from D₃ through the pipe 42. The hydrating liquor is so carried up to D₂, and is simultaneously charged by a supplemental amount of olefine and submitted to a new compression. After a series of similar successive operations effected by means of a more and more compressed gas, the hydrating liquid separated in the last gas removing chamber has reached the required pressure and is saturated by the olefine. Said liquid leaves the pump through the pipe 34, passes through the pressure reducer 8 and enters through the pipe 6 into the tank B where it is freed from alcohol by means of a heavy stream of blown olefine issuing from the gazometer 13 through the pipe 14, as above described.

To sum up, whereas in the Mammoth pump the gas is progressively released in the different elements, on the contrary the hydrating liquor is more and more compressed and simultaneously charged with olefine.

The number of the elements which constitute the pump and the heights to which the liquid is carried up are determined in terms of the nature of the treated olefine.

If so desired, the pipes of the pump through which the liquid is carried up may be lined in order to control the temperature and to maintain it at the correct value.

Example 1.—Manufacture of isopropanol

The tank B is charged with 1000 kg of sulphuric acid at 60% heated to 98° C by the worm 2. The operative characteristics are as follows:

Number of elements of the pump: 8.

Amount and pressure of the propylene introduced in the pump: 400 kg/hour at a pressure of 7 kg and a temperature of 20° C.

Amount of hydrating liquid introduced in the pump: 550 kg/hour.

Amount of propylene absorbed by the hydrating liquid in the pump: 58.3 kg/hour.

Pressure of the hydraulic liquor at the exit of the pump: 5,6 kg.

With the conditions above defined, the production of the apparatus is 3000 kg of isopropanol each day. In this amount, 1000 kg are obtained in the tank B which, apart its primary function of eliminating the formed alcohol, allows for a supplementary production of alcohol by dissolution and hydration of the olefine blown through said tank.

When the dissolution of the olefine is slow, which for example is the case with ethylene, it is necessary to achieve saturating with olefine the hydrating solution which leaves the pump. In such a case, the device shown by Fig. 2 may be employed, the solution, in lieu of being directly sent to the distillation tank B, is first forwarded to the tank A by pipe 34. Said tank A provided with an heating coil 1 and with a stirring device 3, is fed, through the pipe 35, with olefine contained in the vessel 9. A regulator of any convenient type, comprising a pressure sensitive element 31, controlling the inlet valve 36, allows for the regulation of the pressure in the tank A at such a value that the liquid flows without difficulties from D₄ to A through the pipe 34. The olefine which is not absorbed in the tank A is directly sent through the pressure reducer 39 and the pipe 40 to the tank B where it promotes the elimination of alcohol. The hydrating solution,

charged to a maximum with olefine in the tank A, is released at 8 and sent through the pipe 6 into the distillation tank B.

Use may be made of pure olefines or of olefines in admixture with inert gases such as saturated hydrocarbons. In the later case, it is necessary, continuously or not, to effect a blow-off by the pipe 27 inserted in the discharge of the fan 18.

In all the cases, the yields remain highly satisfactory. However, if mixture of olefines and inert gases are treated, the reaction rate is reduced, being proportional to the partial pressure of the olefine in the atmosphere of each of the two tanks.

HENRI MARTIN GUINOT.