PUBLISHED
APRIL 20, 1943.
BY A. P. C.

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PROCESS OF PREPARING KETENE
Filed July 30, 1941

Serial No.
404,666

Fig. 1

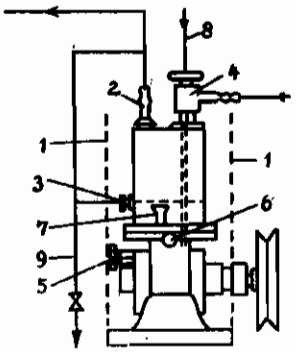
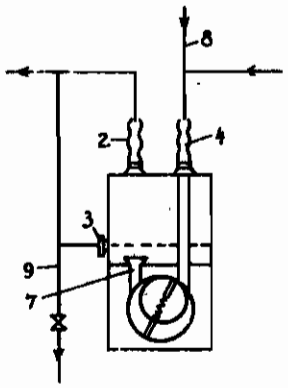


Fig. 2



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Published Apr. 20, 1943

Serial No. 404,666

ALIEN PROPERTY CUSTODIAN

PROCESS OF PREPARING KETENE

Bernhard Popp, Frankfurt am Main, and Walter Johannes Ostrowski, deceased, late of Frankfurt am Main, Germany, by Else Marie Emma Ostrowski, Frankfurt am Main, Germany, widow and guardian of Jorg Ulrich Ostrowski, sole heirs; vested in the Alien Property Custodian

Application filed July 30, 1941

It is known that ketene may be prepared by heating acetic acid or acetic anhydride to high temperatures under reduced pressure in the presence of catalysts splitting off water, cf., for instance, Patent No. 2,108,829. Directly after leaving the tube, the cleavage gases are preferably cooled or sucked through water of 0°C whereby the unchanged acetic acid is removed. In view of the great velocity of flow the ketene practically does not react with the water. In order to isolate the ketene which is rarefied by other gases such as CO, CO_2, ethylene or the like it was necessary, hitherto, to condense the ketene, which boils at −41°C under a pressure of 760 mm, on the vacuum-side in receivers cooled by means of liquefied air. This process is uneconomical and its carrying out on an industrial scale is complicated and difficult. On the other hand, it has not been possible hitherto to isolate on a mass scale the ketene on the pressure-side of the vacuum pump.

Now, we have found that the above problem may be solved by cooling the vacuum pump to temperatures as low as possible, using a lubricant which is indifferent against ketene and has a viscosity sufficient for the temperatures in question and, thereupon, isolating the ketene on the pressure-side or working it up without isolation.

Advantageously vacuum pumps are used which have very small lubricating surfaces and may be cooled to temperatures from about 0°C to about −30°C. For this purpose it is advisable to apply an external cooling from about −60°C to about −70°C. As lubricating agents there are used indifferent oils, for instance, higher saturated hydrocarbons which still show a suitable lubricating-consistency at temperatures ranging as much as possible below 0°C and do not react with, or change the ketene, for instance paraffin oil having a viscosity of ɣ−16=494.05, furthermore vaseline oil, indifferent mineral oils in general, as well as decahydronaphthalene, tetrahydronaphthalene.

It is advisable to provide for a continuous lubrication in order to remove the impurities which accumulate in the pump and which consist mainly of acetic anhydride. An accumulation of the impurities may very detrimentally affect the output of the vacuum pump already after a short time (about 24 hours). The impurities consisting mainly of acetic anhydride are due to the acetic acid vapors not split up and taken along by the carrier gases, which vapors easily react with ketene, especially above 0°C, with formation of acetic anhydride.

The splitting up of acetic acid or acetic anhydride may be performed in known manner, for instance, according to Patent No. 2,108,829. A pressure of about 2 to about 200 mm may be applied and temperatures of between about 400°C and about 900°C, advantageously between about 600°C and about 800°C may be used. As catalysts there are suitable, for instance: phosphorus compounds such as triethylphosphate; furthermore, volatile nitrogen compounds such as ammonia, pyridine, methylamine and phenylamine may be present.

The present process allows of isolating ketene in an economical manner or of further working it up, without previous isolation, into other products of industrial importance.

The following example illustrates the invention:

Gases containing ketene and obtained by dissociation of acetic anhydride are freed from water and acetic acid and introduced into a vacuum pump. The pump is a centrifugal piston pump with an output of 4 cbm per hour and is illustrated in the accompanying drawing, Figs. 1 and 2, as follows: the pump is provided with a cooling jacket 1, an exhaust pipe 2, a screw 3 for controlling the oil level, a suction pipe 4, a screw 5 for regulating the vacuum, a screw 6 for drawing off the oil, a valve 7, an oil supply 8 and an oil collector 9 for drawing off the oil. During the working, an inner temperature up to −20°C is maintained. The lubricant is introduced continuously through suction branch 4. As lubricant there is used paraffin oil having a viscosity of ɣ−16=494.05. Through the bore for screw 3 serving for the control of the oil surface the impurities formed which mainly consist of acetic anhydride besides traces of resinifications are collected. The continuous lubrication which at the same time serves as a cleaning means for the pump amounts to 200 cc. in 24 hours. The acetic anhydride in the collected paraffin oil separates easily and the paraffin oil may be used again without being purified. The acetic anhydride formed in the pump amounts to 0.2% (including resinification). In a forthnight's continuous working the pump transfers 99.5% of the ketene from the vacuum-side to the pressure-side at a pressure of 15–20 mm.

As vacuum pump there may be used in a corresponding manner a slide valve pump with an output of 240 cbm per hour. The oiler pumps the paraffin oil as lubricant into the gliding surfaces cooled by means of a cooling lye. The paraffin

oil contaminated with acetic anhydride is removed by separators arranged in the exhaust.

Instead of providing the pump with a cooling jacket it may also be cooled internally, for instance, by conducting in cycle an indifferent liquid which does not dissolve ketene, such as, paraffin oil, through the pump and a cooler mounted outside the pump.

It is possible by means of a two-stage turbine pump or an "Elmo"-pump to force deeply cooled decahydronaphthalene at a pressure of about 4 atmospheres above atmospheric pressure in cycle by means of water jet pumps into a vessel which may be cooled. The cooling is effected, for instance, by a mixture of carbonic acid and butanol. The water jet pumps produce a pressure of 15 mm. On the suction side of the water jet pumps the ketene is sucked and on the pressure side of the cooling vessel it is discharged.

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As administratrix of Walter Johannes Ostrowski, Deceased.