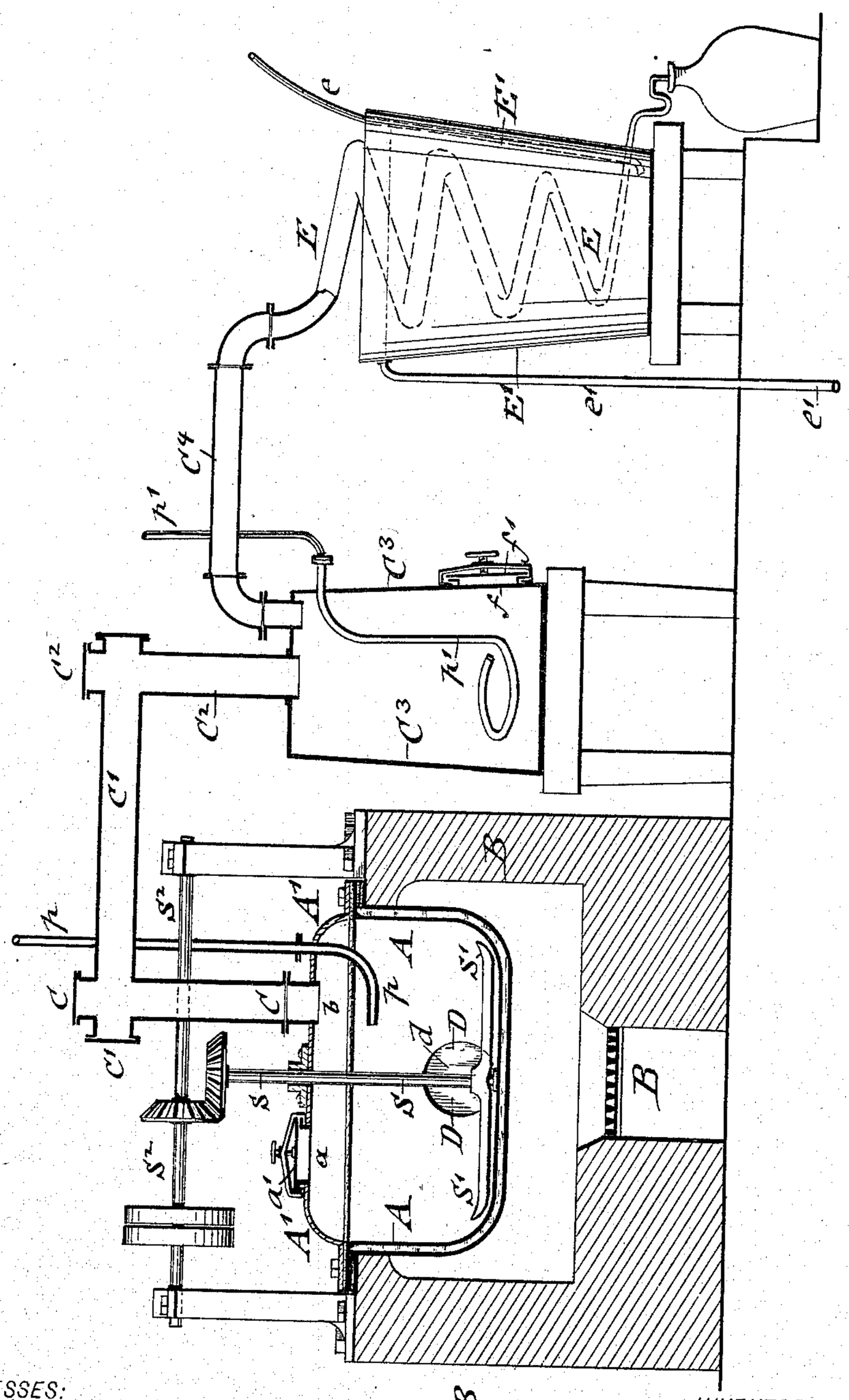
(No Model.)

O. PORSCH.

PROCESS OF MAKING ACETONE.

No. 535,552. Patented Mar. 12, 1895.



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PROCESS OF MAKING ACETONE.

SPECIFICATION forming part of Letters Patent No. 535,552, dated March 12, 1895.

Application filed February 10, 1894. Serial No. 499,759. (No specimens.)

To all whom it may concern:

Be it known that I, OTTOKAR PORSCH, a native of the Empire of Austria-Hungary, residing in the city, county, and State of New York, have invented certain new and useful Improvements in Processes of Making Pure Acetone, of which the following is a specification.

In the manufacture of smokeless powder chemically-pure acetone is required, which, for the purpose of meeting the requirements, must have the following characteristics: First, it must be pefectly clear and limpid; secondly, it must be miscible with distilled water in every proportion, and the mixture should not show, either directly after mixing or after considerable standing, a cloudiness or a precipitate; thirdly, it must be perfectly neutral; fourthly, it must indicate at a temperature of 15° Celsius on a thermo-alcoholometer of G. A. Schultze, of Berlin, at least 99.5 per cent.; fifthly, the acetone should not contain more than 0.1 per cent. of aldehyde, and the iodometric test should not indicate less than 98.5 per cent. of C_3H_6O; sixthly, in distilling the acetone at least 98.8 per cent. of the liquid should have passed over when the thermometer indicates 58° Celsius.

For the purpose of producing acetone having the foregoing characteristics, my invention consists in a process of making pure acetone, having the following steps: first mixing ordinary commercial acetate of lime with milk of lime, so as to obtain a ten per cent. surplus of lime, next drying the mixture, then subjecting the same under continuous stirring to a uniform heat while gradually admitting superheated steam to the same, next condensing the acetone vapors, mixing the crude liquid acetone with water and permitting the mixture to stand so as to separate the acetone from the water and tar-oils, and finally subjecting the clear water-containing acetone to fractional distillation and final rectification.

In carrying out my improved process, the mixing apparatus shown in vertical longitudinal section in the accompanying drawing, may be used to advantage.

The apparatus consists of a closed mixing-vessel A, which is formed of an interior and an exterior vessel, the space or jacket between which is filled with molten lead, that is retained in molten condition by the heat of a furnace B below the mixing-vessel.

The furnace B is built up of bricks and extended around the mixing-vessel A. The interior and exterior sections of the mixing-vessel A are provided with outwardly-bent flanges, to which the cover A′ is hermetically connected, so that no gases can escape from the interior of the mixing-vessel A. The cover A′ is provided at its center with a stuffing-box which forms a neck-bearing for a vertical shaft S, that extends in downward direction through the mixing-vessel and which is supported at its lower end in a step-bearing at the center of the interior sections of the mixing-vessel A.

A man-hole *a* with the usual man-hole cover *a′* is arranged in the cover A′ of the mixing-vessel, while a discharge opening *d* is arranged in the lower side-walls of the same near its bottom and connected through the inner and exterior sections and through the furnace-wall to the outside, said discharge-tube being closed by a tightly fitting cover (not shown) so as to permit thereby the ready discharge of the contents of the mixing-vessel, when the operation of distilling off the crude acetone from the charge is completed.

An opening *b* in the cover A′ is connected with a vertical tube C which is again connected by a horizontal tube C′ and downwardly-extending tube C^2 with an intermediate collecting-vessel C^3, from which the acetone vapors are conducted by a tube C^4 to a condensing coil E which latter is supported in a vat E′ to which cold water is supplied by an inlet-pipe *e* terminating near the bottom of the vat, while the heated water is drawn off through an outlet-pipe *e′* at the upper part of the vat.

The vertical shaft S that extends into the interior of the mixing-vessel A is provided at its lower end with diametrical stirrer-arms S′ which are arranged close to the bottom of the interior vessel and bent up at their ends so as to exert in rotating a mixing and scraping action on the contents of the vessel and prevent them from settling on the bottom of the same. Rotary motion is imparted to the ver-

tical shaft S by bevel-gears, which receive their motion from a horizontal shaft S^2 provided with a fast and loose pulley in the usual manner.

A supply-pipe *p* for super-heated steam extends through the cover of the mixing-vessel to the interior of the same, while a similar pipe *p'* extends through the wall of the collecting-vessel B to the lower part of the same, said collecting-vessel being provided at its lower part with a man-hole *f* and with a man-hole cover *f'* in the usual manner.

In producing pure acetone commercial grayish acetate of lime, containing eighty-two per cent. of acetic acid hydrate, is mixed with milk of lime, so as to produce an excess of lime of ten per cent. in the acetate of lime. The mixture is then dried and transferred while in a hot state into the mixing-vessel A. By adding the ten per cent. additional supply of lime, the loss of acetic acid hydrate, and the corresponding loss of acetone, are reduced to the smallest possible limit.

Each charge of acetate of lime for the mixing-vessel consists of about three hundred pounds, care being taken that the mass is charged in small grains of equal size, which are obtained by passing the acetate of lime through a screen so that all pieces of a larger size are separated, which are broken up and passed through the screen. The space between the interior and exterior sections of the mixing-vessel A is filled with molten lead, which lead-bath is retained in liquid condition by the heat of a furnace B. The lead-bath serves for the purpose of keeping the temperature in the mixing-vessel during the generation of the acetone-vapors perfectly constant, so that any chances of decomposing the acetone are prevented.

It is obvious that before the molten lead is introduced into the space between the interior and exterior sections of the mixing-vessel, the exterior section has to be heated to a light red heat, so that the lead remains in liquid condition and that the interior of the mixing-vessel has a temperature equal to the melting-point of the lead, and is ready for the introduction of the acetate of lime. The dry heated charge of acetate of lime and carbonate of lime is introduced through the man-hole in the cover as quickly as possible, for which purpose it stands ready in suitable vessels and is dropped through a large funnel that fits tightly into the man-hole of the cover of the mixing-vessel. During the filling of the mixing-vessel, the stirrers are set in motion. The man-hole cover is then closed quickly, as the generation of acetone commences instantly on the charging of the mixing-vessel. While the acetate of lime is subjected to the heat of the mixing-vessel under continuous stirring, the generation of acetone takes place, the vapors of the same passing through the connecting-tubes C, C', C^2 into the collecting-vessel C. When about twelve to fifteen per cent. of the acetone contained in the charge, have passed over, the charge becomes perfectly dry and assumes a dust-like shape, so that it is necessary to supply superheated steam into the mixing-vessel A and into the collecting-vessel B, which supply of steam is continued to the end of the operation. The entire operation of treating the acetate of lime and separating the acetone contained in the same should not take longer than four hours, inclusive of the charging and discharging of the mixing-vessel. The vapors of the acetone are condensed in the condensing-coil, which is provided at its lower end with a siphon-shaped discharge-pipe, the crude liquid acetone being dropped into a suitable receiver. As soon as condensed water is discharged through the siphon-shaped end of the coil, the operation is completed and the contents of the mixing-vessel have to be discharged which is accomplished by opening the discharge-tube D. The stirrers move then gradually the entire body of carbonate of lime in the mixing-vessel to the discharge-tube and through the same to the outside so that the mixing-vessel is in a condition to be immediately charged again with a dry mixture of acetate of lime and calcium-hydrate in excess, as before described. The thus-obtained crude acetone,—that is to say, the quantity of acetone which results from a working period of twenty-four hours,—is next mixed with ten times its weight of water and allowed to stand for twenty-four hours in a cool place. The vats are provided with a water-gage and with four discharge-faucets, located at different heights, so as to decant, on the one hand, any tar-oils that collect on the surface of the solution, and draw off on the other hand a clear mixture of acetone and water from the sediment collected at the bottom of the vats. The clear water-containing acetone is next transferred to a dephlegmator and subjected in the same to fractional distillation. The initial and final runs of about fifty pounds each, are set aside, while the quantity that runs off between the initial and terminal runs, and which is a distillate containing about from ninety to ninety-six per cent. of acetone, is separately treated, so as to permit the removal even of the last few per centum of water still contained in the same. This is accomplished by a final distillation of the acetone, which is produced in a column-rectificator, constructed in the following manner: On a copper still containing about twenty hectoliters is supported a rectifying column, composed of thirty-two trays, which column is connected with two condensation-cylinders that differ from those in the ordinary alcohol-rectifying column only in the fact that the cooling water supplied to the lower cylinder, should not be of a temperature below 15° centigrade, and that the cooling water, after it is heated to 25° centigrade, should be conducted to the lower end of the upper condensation-cylinder, and that the temperature of the cooling-water is so regulated that the water passing from

the upper condensation-cylinder should never rise above the boiling-point of pure acetone, namely, 58° centigrade, but should not pass off at a temperature below 50° centigrade. The rectification of the acetone is so regulated that not more than one hundred and fifty liters should pass over per hour; but even of this distillate, the first fifty liters and the last fifty liters should be set aside as containing perhaps some traces of aldehyde. If the iodometric test should show that the quantity of the acetone which has been set aside, being a certain quantity running over at the beginning and end of one distillation, mixed with the same quantity obtained by a second distillation, should contain more than 0.1 per cent. of aldehyde, then the acetone is passed in gas-form through tubes that are heated to red heat, and then condensed again, so that the ketones that boil at a higher temperature than the acetone, are split up. The so-treated initial and final runs of the distillation are mixed with the next quantity of acetone, and subjected to rectification, so that it is possible to obtain the greatest possible yield of chemically-pure acetone, which yield approaches almost the theoretical yield.

The advantages of my improved process of obtaining pure acetone consist in the following facts:

First. By adding an excess of calcium-hydrate to the acetate of lime employed, every atom of acetic acid is liberated and an alkaline residue of carbonate of lime obtained that is perfectly free from acetic acid, so that the yield of acetone approaches almost the limit of the theoretical yield and thereby twenty-five per cent. of pure acetone, boiling at 58° centigrade, be regularly obtained from ordinary commercial acetate of lime.

Second. By the use of a lead-bath having a constant melting point the superheating of the interior of the mixing-vessel is obviated and any variations of temperature during the generation of the acetone from the charge prevented, whereby an acetone is obtained which is free from decomposition-products, such as aldehyde and ketones.

Third. By the introduction of superheated steam into the mixing-vessel, after about twelve per cent. of the acetone has gone over, the generation of the remaining acetone is accelerated, while the formation of mechanical impurities and the passing over of any portion of the charge in powder-form from which the acetone has not been separated, is prevented.

Fourth. The tubes by which the connection of the mixing-vessel with the collecting-vessel are made, can be readily taken apart and cleaned after each operation, so that the choking of the same by the settling of dust-particles and the occurrence of explosions arising from the same, can be entirely prevented.

Fifth. By supplying the superheated steam to the collecting-vessel, the free and uniform passage of the acetone vapors into the condenser is accomplished, and thereby the formation of mechanical impurities and the choking of the condensing-coil with dust-particles prevented.

Sixth. By mixing the crude acetone with water and subjecting the same afterward to fractional distillation, it is possible to remove every trace of tar-oil from the acetone.

Seventh. By the use of a column-rectifying apparatus, anhydrous acetone of a high degree of purity is obtained as is required in the manufacture of smokeless powder for dissolving the pyroxilin and which acetone has all the characteristics and properties hereinbefore stated.

Eighth. That any ketones of higher boiling points contained in the acetone, are split up by distillation over tubes heated to red heat, so that they are thereby removed from the acetone.

Having thus described my invention, I consider as new and desire to secure by Letters Patent—

1. The process herein described of producing chemically-pure acetone, which consists in subjecting an acetate in the presence of calcium-hydrate in excess under continuous agitation to distillation and to the action of superheated steam, so as to separate the acetone-vapors from the remaining carbonated lime, substantially as set forth.

2. The process herein-described of producing chemically pure acetone, which consists of the following steps: first, mixing ordinary commercial acetate of lime with calcium-hydrate in excess, second, subjecting the mixture under continuous stirring to a constant heat under addition of superheated steam so as to separate the acetone-vapors from the remaining carbonate of lime, third, condensing said vapors into crude liquid acetone, and lastly, purifying the crude liquid acetone by subjecting it to fractional distillation and rectification, substantially as set forth.

3. The process herein described of producing chemically-pure acetone, which consists of the following successive steps: first, mixing the ordinary commercial acetate of lime with calcium-hydrate in excess, second, subjecting the mixture under continuous stirring to a constant heat under addition of superheated steam so as to separate the vapors of acetone from the remaining carbonate of lime, third, condensing the acetone-vapors into crude liquid acetone, fourth, mixing the crude acetone with water and permitting it to stand so as to separate it from the tar-oils and sediments, fifth, subjecting the purified acetone to fractional distillation, and finally rectifying the same, substantially as set forth.

4. The process herein described of producing chemically-pure acetone, which consists in subjecting a mixture of commercial acetate of lime and calcium-hydrate to heat under continuous stirring, supplying superheated steam to the charge so as to facilitate

the generation of the acetone-vapors, condensing the vapors, mixing the crude liquid acetone with water in excess, so as to produce the separation of the tar-oils and sediments, subjecting the washed acetone to fractional distillation and rectification and separating the higher-boiling ketones contained in the rectified acetone by subjecting the same in gas form to surfaces heated to red heat so as to break up the ketones, substantially as set forth.

In testimony that I claim the foregoing as my invention I have signed my name in presence of two subscribing witnesses.

OTTOKAR PORSCH.

Witnesses:
PAUL GOEPEL,
K. R. BRENNAN.