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PREPARATION OF KETENE

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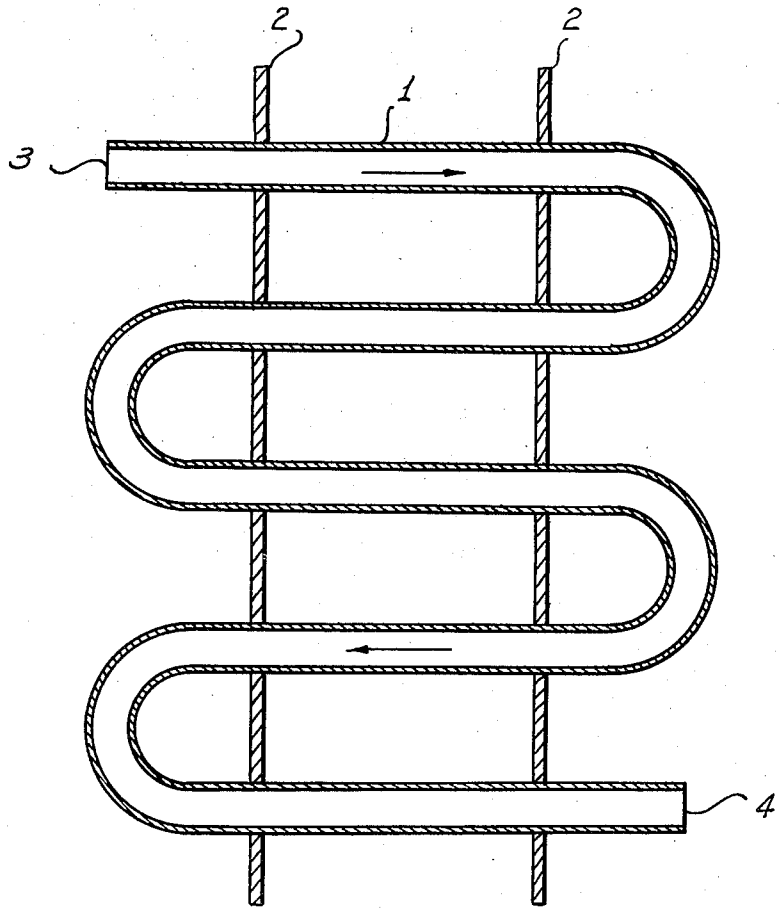

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PREPARATION OF KETENE

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This invention relates to a novel process of, and a novel apparatus for, conducting chemical reactions. More particularly, the invention relates to a novel apparatus for, and a novel process of, making ketene by the thermal decomposition of acetone.

Ketene has been made in the past by thermal decomposition of various organic substances. A known process for making ketene comprises thermally decomposing acetone by passing the vapor thereof through a heated tube. It has generally been thought advisable to construct the pyrolysis tube so that at least the inner surface thereof is made of a non-metallic material, such as quartz, or of a non-ferrous material, such as copper or copper-aluminum alloy or silver or platinum. Ferrous materials as a broad class have generally been avoided in the construction of pyrolysis tubes for cracking acetone to produce ketene. It has hitherto been considered that carbon steel catalyzes the undesired decomposition of ketene and other pyrolysis products of acetone, in the thermal decomposition process referred to, thereby forming coke or carbon. The side reaction of coking is objectionable in that it results ultimately in the plugging of the pyrolysis tube, and in the necessity for stoppage of manufacture in order to recondition the apparatus.

In one of its aspects, the invention provides an improved process for making ketene by thermal decomposition of acetone, the improvement comprising decomposing the acetone in a reaction vessel constructed essentially of a steel having the composition: from about 14% to about 18% chromium, a maximum of about 0.12% carbon, balance substantially entirely iron. This type of steel is conventionally referred to as Type 430 stainless steel; see "Chemical Engineer's Handbook," edited by John H. Perry, third edition, 1950, page 1532.

In another aspect, the invention provides an improved apparatus for the preparation of ketene by thermal decomposition of acetone, the improvement comprising the provision of a pyrolysis vessel in the form of an elongated tube constructed essentially of a steel having the composition: from about 14% to about 18% chromium, a maximum of about 0.12% carbon, balance substantially entirely iron.

The invention is further disclosed with reference to the accompanying drawing, which consists of a single figure. The figure illustrates diagrammatically one form of apparatus embodying the principles of the invention, in which the novel process of the invention may be practiced. In the drawing there is shown a cross-sectional view of a pyrolysis vessel in the form of an elongated tube 1 made of Type 430 stainless steel, and disposed in the form of a flat serpentine. Tube 1 is supported by bars 2 forming a supporting framework. The tube 1 and its supporting framework 2 are disposed within a brick heating furnace (not shown, being of conventional construction, as will be obvious to those skilled in the art). The tube is heated by suitable heating apparatus (not shown, also of conventional nature known to those skilled in the art, e. g. a gas heater or an electrical

apparatus for heating the furnace containing the pyrolysis vessel). The pyrolysis vessel is provided with an entrance port 3, into which acetone vapor is introduced, and also is provided with an exit port 4, through which unconverted acetone and the gaseous products of the reaction, including principally ketene, carbon monoxide, ethylene and methane, are withdrawn. It will be appreciated that the tube 1 can be constructed in shapes other than a flat serpentine, as will be obvious to those skilled in the art, e. g. in the form of a helical coil or in a straight line.

In one embodiment, the invention provides a continuous process for making ketene which comprises continuously passing acetone vapor through an elongated tube constructed essentially of a steel having the composition: from about 14% to about 18% chromium, a maximum of about 0.12% carbon, balance substantially entirely iron; the tube being maintained at temperatures in the range between about 700° C. and about 800° C.; the acetone vapor being passed through the tube at mass velocities in the range between about 10,000 lbs./sq. ft./hr. and about 40,000 lbs./sq. ft./hr.; the time of contact of the acetone vapor with the heated tube being in the range between about 0.01 second and 1.0 second.

In a preferred embodiment of the process aspect of the invention, a pyrolysis temperature in the range between about 730° C. and 760° C. is employed, and the acetone vapor is passed through the pyrolysis tube at a mass velocity in the range between about 25,000 lbs./sq. ft./hr. and 35,000 lbs./sq. ft./hr., the contact time of the acetone vapor being in the range between about 0.1 second and 0.5 second.

In order to accommodate the pressure drop through the elongated tube 1, it is necessary that there be a higher pressure at the entrance port 3 than at the exit port 4. With the mass velocities and contact times taught by the present invention, a pressure differential of not more than about 15 p. s. i. is sufficient. In a preferred embodiment of the invention, the acetone vapor is introduced at the entrance port 3 at a pressure between about 2 and about 5 p. s. i. gauge, and the product vapors are withdrawn from the exit port 4 at approximately atmospheric pressure.

The invention is further disclosed in the following examples, which are illustrative but not limitative thereof.

Example 1

Acetone was vaporized and preheated to a temperature of 150° C. in a vaporization and preheating apparatus of conventional design (that of U. S. Patent 2,053,286). The acetone vapor, under a pressure of 5 p. s. i. gauge and at a temperature of 150° C., was continuously passed through a pyrolysis tube made entirely of Type 430 stainless steel, in the form of a flat serpentine as illustrated in the drawing; said tube having an inner diameter of ¾ inch and a total length of 40 feet. The tube was positioned in a heating furnace and was thereby maintained at a reaction temperature of 730° C. 40,000 g./hr. of acetone vapor was passed through at a mass velocity of about 29,000 lbs./sq. ft./hr.; contact time, 0.3 second. The product gases were removed at the end of the pyrolysis tube, at atmospheric pressure, and were passed through condensation apparatus (that shown in the above referenced Patent 2,053,286), thereby condensing out unreacted acetone. The uncondensed gases were passed to an apparatus for converting ketene to diketene, and the diketene was used as such.

Example 2

Acetone was vaporized and preheated to a temperature of 90° C. in the same apparatus employed in Example 1. The acetone vapor, under a pressure of 3 p. s. i. gauge, and at a temperature of 90° C., was continuously passed

through a pyrolysis tube made entirely of Type 430 stainless steel, in the form of a helical coil; said tube having an inner diameter of ¾ inch and a total length of 35 feet. The tube was positioned in a heating furnace and was thereby maintained at a reaction temperature of 750° C. 20,200 g./hr. of acetone vapor was passed through at a mass velocity of 14,500 lbs./sq. ft./hr.; contact time, 0.5 second. The product gases were removed at the end of the pyrolysis tube and were passed through the same condensation apparatus employed in Example 1, thereby condensing out unreacted acetone. The uncondensed gases were passed through an apparatus for converting ketene to acetic acid.

Example 3

Acetone was vaporized and preheated to a temperature of 90° C. in an apparatus of conventional design (that of U. S. Patent 2,053,286). The acetone vapor, under a pressure of 5 p. s. i. gauge and at a temperature of 90° C. was continuously passed through a pyrolysis tube made entirely of Type 430 stainless steel, in the form of a flat serpentine, as illustrated in the drawing; said tube having an inner diameter of ¾ inch and a total length of 40 feet. The tube was positioned in a furnace and maintained at a reaction temperature of 700° C. 33,700 g./hr. of acetone vapor was passed through at a mass velocity of 24,000 lbs./sq. ft./hr.; contact time, 0.4 second. The product gases were removed at the end of the pyrolysis tube and passed through condensation apparatus (that shown in the above referenced Patent 2,053,286), thereby condensing out unreacted acetone. The uncondensed gases were passed through an apparatus for converting ketene to diketene.

I claim:

1. In a process for making ketene by thermal decomposition of acetone, the improvement which comprises decomposing the acetone in a reaction vessel the inner surface of which is constructed of a steel having essentially the composition: from about 14% to about 18% chromium, a maximum of about 0.12% carbon, balance iron.

2. A continuous process for making ketene which comprises continuously passing acetone vapor through an elongated tube the inner surface of which is constructed of a steel having essentially the composition: from about 14% to about 18% chromium, a maximum of about 0.12% carbon, balance iron; the tube being maintained at temperatures in the range between about 700° C. and about 800° C.; the acetone vapor being passed through the tube at mass velocities in the range between about 20,000 lbs./sq. ft./hr. and about 40,000 lbs./sq. ft./hr.; the time of contact of the acetone vapor with the heated tube being in the range between about 0.01 second and 1.0 second.

3. A continuous process for making ketene which comprises continuously passing acetone vapor through an elongated tube constructed of Type 430 stainless steel; the tube being maintained at temperatures in the range between about 730° C. and about 760° C.; the contact time of the acetone vapor being in the range between about 0.1 second and 0.5 second.

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