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(54) Title: METHOD FOR REFINING ACETIC ANHYDRIDE

(57) Abstract

Disclosed is a continuous method for refining acetic anhydride produced by the reaction of ketene and acetic acid by means of a vacuum distillation system to provide a refined material comprised of at least 99.5 weight percent acetic anhydride, not more than 0.5 weight percent acetic acid and not more than 90 parts per million (ppm) diketene.
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METHOD FOR REFINING
ACETIC ANHYDRIDE

This invention pertains to a novel method for refining acetic anhydride produced by the reaction of ketene and acetic acid. More specifically, this invention pertains to a vacuum distillation method for purifying a stream comprised predominantly of acetic anhydride and acetic acid with minor amounts of diketene to obtain a refined material comprised of at least 99.5 weight percent acetic anhydride, not more than 0.5 weight percent acetic acid and not more than 90 parts per million (ppm) diketene.

The manufacture of acetic anhydride from ketene and acetic acid produces a crude product stream comprising 83 to 87 weight percent acetic anhydride, 13 to 17 weight percent acetic acid and 50 to 110 ppm diketene. The crude product stream has been refined by distillation using sub-atmospheric pressures to avoid or minimize degradation of the acetic anhydride. For example, the use of pressures of 200 torr (26.6 kPa) may be utilized to obtain acetic anhydride having a purity sufficient for general acetylation purposes. Such a grade of acetic anhydride typically comprises at least 99.5 weight percent acetic anhydride, not more than 0.5 weight percent acetic acid and not more than 90 parts per million (ppm) diketene and has a Hunter color of not more than 10.

We have discovered that acetic anhydride of the above-described grade can be produced continuously using two distillation columns and a particular combination of pressures and temperatures. The method of the present invention therefore provides a continuous means for obtaining purified acetic anhydride comprising at least 99.5 weight percent acetic anhydride, not more than 0.5
weight percent acetic acid and not more than 90 ppm diketene and having a Hunter color of not more than 10 by the steps comprising:

(1) feeding a mixture comprising 83 to 87 weight percent acetic anhydride, 13 to 17 weight percent acetic acid and 50 to 110 ppm diketene to the mid-section of an acid removal distillation column in which (i) a column base temperature of 114 to 127°C, (ii) a column head temperature of 87 to 105°C, and (iii) a column top pressure of 225 to 350 torr (29.93 to 46.55 kPa) are maintained;

(2) removing from the head of the acid removal distillation column a first stream comprised of 20 to 30 weight percent acetic anhydride, 70 to 80 weight percent acetic acid and 10 to 40 ppm diketene;

(3) removing a second stream from the base of the acid removal distillation column and feeding the second stream to the base of a color column in which (i) a column base temperature of 101 to 105°C, (ii) a column head temperature of 97 to 100°C, and (iii) a column top pressure of 225 to 350 torr (29.93 to 46.55 kPa) are maintained; and

(4) removing from the mid-section of the color column a stream comprising the purified acetic anhydride.

The acid removal distillation column preferably is operated at (i) a column base temperature of 125 to 127°C, (ii) a column head temperature of 103 to 105°C, and (iii) a column top pressure of 330 to 350 torr (43.89 to 46.55 kPa), and the color column preferably is operated at (i) a column base temperature of 101 to 105°C, (ii) a column head temperature of 97 to 100°C, and (iii) a column top pressure of 330 to 350 torr (43.89 to 46.55 kPa).
In the following description illustrating the operation of the process, the parts given are by volume. The acetic anhydride/acetic acid mixture having the composition described above is fed continuously to the mid-section of the acid removal column at a rate of 90 to 100 parts per minute. The exact feed point can vary depending, for example, on the particular design of the column and/or the column internals used. The mid-section feed point normally will be at a point which is from 50 to 80% of the distance from the bottom to the top of the acid removal column. In the specific apparatus used in the present invention the feed point is located 77% from the base of the column. The acid removal column is equipped with conventional trays and/or packing material to achieve the desired degree of anhydride/acid separation. We have found that beds of metal slotted ring packing material, both above and below the feed point, provide good separation.

The base of the acid removal column is maintained at a temperature of 125 to 127°C by means of a heat source. For example, a portion of the column underflow stream may be recycled through a heat exchanger (reboiler) and returned to the base of the column. The sub-atmospheric pressure specified above is measured at the top or head of the acid removal column. The pressure at the base of the column generally is 80 to 90 torr (10.64 to 11.97 kPa) more than that existing at the top. A mixture comprising 20 to 30 weight percent acetic anhydride and 70 to 80 weight percent acetic acid is removed continuously from the top of the column.

The stream removed from the base of the acid removal column is unacceptably dark in color due to the presence therein of colored materials such as carbonaceous compounds and other decomposition products
resulting from the acetic anhydride manufacturing process and/or the reboiler employed in conjunction with the first distillation. The highly colored material removed from the base of the acid removal column is pumped to the base of a color column in which (i) a column base temperature of 101 to 105°C, (ii) a column head temperature of 97 to 100°C, and (iii) a column top pressure of 330 to 350 torr (43.89 to 46.55 kPa) are maintained. The color column is equipped with trays and/or packing material and a heat source in a manner analogous to the acid removal column.

The purified acetic anhydride having the composition set forth above is removed from the mid-section of the color column at a rate of 80 to 90 parts per minute. The removal of the colored material is accomplished by continuously removing a stream comprised mainly of acetic anhydride in addition to the color-causing material at a rate of 1 to 5 parts per minute. A third stream comprising 89 to 98 weight percent acetic anhydride, 2 to 11 weight percent acetic acid and 100 to 300 ppm diketene is removed from the top of the color column at a rate of 1 to 3 parts per minute.

The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention.
We claim:

1. Continuous process for obtaining purified acetic anhydride comprising at least 99.5 weight percent acetic anhydride, not more than 0.5 weight percent acetic acid and not more than 90 ppm diketene and having a Hunter color of not more than 10 by the steps comprising:

   (1) continuously feeding a mixture comprising 83 to 87 weight percent acetic anhydride, 13 to 17 weight percent acetic acid and 50 to 110 ppm diketene to the mid-section of an acid removal distillation column in which (i) a column base temperature of 114 to 127°C, (ii) a column head temperature of 87 to 105°C, and (iii) a column top pressure of 225 to 350 torr (29.93 to 46.55 kPa) are maintained;

   (2) continuously removing from the head of the acid removal distillation column a first stream comprised of 20 to 30 weight percent acetic anhydride, 70 to 80 weight percent acetic acid and 10 to 40 ppm diketene;

   (3) continuously removing a second stream from the base of the acid removal distillation column and feeding the second stream to the base of a color column in which (i) a column base temperature of 101 to 105°C, (ii) a column head temperature of 97 to 100°C, and (iii) a column top pressure of 225 to 350 torr (29.93 to 46.55 kPa) are maintained; and

   (4) continuously removing from the mid-section of the color column a stream comprising the purified acetic anhydride.

2. Process according to Claim 1 further comprising the steps of:
(5) continuously removing from the base of the color
column a stream comprised of acetic anhydride and
color-causing materials; and
(6) continuously removing from the top of the color
column a stream comprised of 89 to 98 weight
percent acetic anhydride, 2 to 11 weight percent
acetic acid and 100 to 300 ppm diketene.

3. Continuous process for obtaining purified acetic
anhydride comprising at least 99.5 weight percent acetic
anhydride, not more than 0.5 weight percent acetic acid
and not more than 90 ppm diketene and having a Hunter
color of not more than 10 by the steps comprising:
(1) continuously feeding a mixture comprising 83 to 87
weight percent acetic anhydride, 13 to 17 weight
percent acetic acid and 50 to 110 ppm diketene to
the mid-section of an acid removal distillation
column in which (i) a column base temperature of
125 to 127°C, (ii) a column head temperature of 103
to 105°C, and (iii) a column top pressure of 330 to
350 torr (43.89 to 46.55 kPa) are maintained;
(2) continuously removing from the head of the acid
removal distillation column a first stream
comprised of 20 to 30 weight percent acetic
anhydride, 70 to 80 weight percent acetic acid and
10 to 40 ppm diketene;
(3) continuously removing a second stream from the base
of the acid removal distillation column and feeding
the second stream to the base of a color column in
which (i) a column base temperature of 101 to
105°C, (ii) a column head temperature of 97 to
100°C, and (iii) a column top pressure of 330 to
350 torr (43.89 to 46.55 kPa) are maintained; and
(4) continuously removing from the mid-section of the color column a stream comprising the purified acetic anhydride.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 5 C07C53/12 C07C51/573

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 C07C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>A</td>
<td>US,A,2 232 705 (HULL) 25 February 1941 see claim 1</td>
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Date of actual completion of the international search 16 December 1993

Date of mailing of the international search report 28. 12. 93

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