

In a modification of this embodiment, the post-flash vapouriser may be integral with the fractional distillation column used to effect the fractional distillation of step (6) so that steps (6) to (9) are modified to:

- 5 (6') separating the volatile fraction from (4) by fractional distillation in a distillation column into an overhead fraction comprising unreacted carbonylation feedstock and iodine-containing promoter, a base vapour fraction comprising carboxylic acid and/or anhydride having reduced iodide contamination and a liquid base
10 fraction,
(7') recycling the overhead fraction from (6') to the carbonylation step,
(8') withdrawing the base vapour fraction from the base of the distillation column and the base liquid fraction from the base of
15 the distillation column,
(9') recycling the base liquid fraction from (8') as wash liquid to the scrubbing section of the preliminary flash vapouriser.

As previously discussed the base vapour fraction may be removed from the distillation column at about one or two trays from the base
20 to reduce entrainment.

Details of preferred reactants, reaction conditions and procedures for effecting this particularly preferred embodiment may be found in the previously referred to EP-A-0087870.

A particularly preferred embodiment of the process of the
25 invention is further illustrated with reference to the accompanying drawings in which Figure 1 is a simplified flow diagram of the relevant part of a process for the manufacture of acetic anhydride and acetic acid from methanol and carbon monoxide by the integrated esterification, carbonylation and separation steps of EP-A-0087870
30 and Figure 2 is a modification of the apparatus of Figure 1 with the vapouriser being integral with the distillation column.

Referring to Figure 1 in use the product of the carbonylation reaction consisting of predominantly acetic anhydride, acetic acid, unreacted methyl acetate, rhodium carbonylation catalyst, some
35 methyl iodide promoter and optional co-promoter such as N,N'dimethyl

iodide-contaminated carboxylic acid and/or anhydride which bottoms fraction is fed to the post-flash vapouriser wherein carboxylic acid and/or anhydride having a reduced iodide contamination is separated as a vapour fraction from a liquid fraction.

5. 3. A process as claimed in claim 1 or claim 2 in which the post-flash vapouriser is a flash vapouriser.
4. A process as claimed in claim 2 modified in that the post-flash vapouriser is integral with the distillation column and the vapour fraction from the preliminary flash vapouriser is passed to the distillation column wherein an overhead fraction comprising carbonylation feedstock and iodine-containing promoter is removed from the distillation column and a vapour fraction comprising carboxylic acid and/or anhydride is taken from the base of the distillation column separately from a liquid base fraction which is removed from the base of the distillation column.
- 15 5. A process as claimed in any one of claims 1 to 4 in which the post-flash vapouriser is operated at a pressure of up to 10 barg and/or a temperature in the range 100 to 200°C.
6. A process as claimed in any one of claims 2 to 5 in which the preliminary flash vapouriser is provided in the upper region thereof with a scrubbing section to which is provided a liquid as wash therefore.
- 20 7. A process as claimed in any one of the preceding claims in which there is purified a carboxylic acid having from 2 to 4 carbon atoms and/or an anhydride thereof.
- 25 8. A process as claimed in claim 7 in which there is purified a fraction comprising acetic acid and acetic anhydride.
9. A process as claimed in claim 8 in which the acetic acid and acetic anhydride are produced by the carbonylation of carbonylatable feedstock in the presence of a rhodium carbonylation catalyst, methyl iodide promoter and N,N'-dimethyl imidazolium iodide co-promoter.
- 30 10. A process for the production of acetic anhydride with or without net co-production of acetic acid from methanol and carbon monoxide in a series of esterification, carbonylation and separation
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①. and its purification according to claim 1