CLAIMS

1. A process comprising:

carbonylating at least one member of selected from the group consisting of methanol, dimethyl ether, and methyl acetate in the presence of 0.1 to less than 14 wt.% water, a rhodium catalyst, methyl iodide and lithium iodide, to form a reaction medium comprising acetic acid in a reactor;

separating the reaction medium into a liquid recycle stream and a vapor product stream;

separating the vapor product stream in up to 2 distillation columns in a primary purification train to produce a crude acid product comprising acetic acid comprising up to 10 ppm lithium cations;

contacting the crude acetic acid product with a first purification resin comprising a cationic exchanger in the acid form within a first treatment device to produce an intermediate acid product having a lithium ion concentration of less than 50 ppb; and

contacting the intermediate acetic acid product with a second purification resin comprising a metal-exchanged ion exchange resin having acid cation exchange sites within a second treatment device to produce a purified acetic acid stream;

the first treatment device, the second treatment device, or both individually comprising at least one sampling port disposed through a side of the treatment device comprising a first open flow path between a sample inlet located within the treatment device and a first flow control element located external to the treatment device, which is operable between an open and a closed position to obtain a sample of the resin proximate to the sample inlet, and

a second flow path comprising a porous element disposed between the sample inlet and a second flow control element located external to the treatment device, which is operable between an open and a closed position to obtain a sample of only the liquid proximate to the sample inlet;

opening the first flow control element to obtain a sample of the purification resin at a point in the treatment device proximate to the sample inlet of the corresponding sample port;

determining a concentration of an impurity in the sample of the purification resin;

comparing the impurity concentration of the sample to a previously determined impurity concentration value; and

determining if the impurity concentration of the sample indicates exhaustion of the purification resin at the point in the treatment device proximate to the sample inlet.

2. The process of claim 1,
   further comprising opening the second flow control element to obtain a sample of only the liquid proximate to the sample inlet;
   determining a concentration of an impurity in the liquid sample; and
   comparing the impurity concentration of the liquid sample to a previously determined impurity concentration value, and
   determining if the impurity concentration of the liquid sample indicates exhaustion of the purification resin at a point in the treatment device proximate to the sample inlet.
3. The process of claim 2, further comprising stopping the flow of acetic acid through the treatment device prior to exhaustion of essentially all of the active purification resin present in the treatment device, regenerating at least a portion of the exhausted resin and resuming flow of the acetic acid stream through the treatment device to produce the purified acetic acid stream.
4. The process of claim 1, wherein separating the vapor product stream comprises:
   distilling the vapor product stream in a first distillation column and taking a sidedraw to yield a distilled acetic acid product; and
   distilling the distilled acetic acid product in a second distillation column to produce a crude acid product comprising acetic acid and lithium cations.
5. The process of claim 1, further comprising a step of adding a potassium salt selected from the group consisting of potassium acetate, potassium carbonate, and potassium hydroxide to the distilled acetic acid product prior to distilling the distilled acetic acid product in a second distillation column; wherein at least a portion of the potassium is removed by the cationic exchanger in the acid form.
6. The process of claim 1, wherein the crude acetic acid product is contacted with the cationic exchanger at a temperature from 50°C to 120°C.
7. The process of claim 1, wherein the intermediate acetic acid product is contacted with the metal-exchanged ion exchange resin at a temperature from 50°C to 85°C.

8. The process of claim 1, wherein the cationic exchanger in the acid form comprises a resin of acid-form strong acid cation exchange macroreticular, macroporous or mesoporous resins.
9. The process of claim 1, wherein the metal-exchanged ion exchange resin comprises at least 1% strong acid exchange sites occupied by silver.
10. The process of claim 1, further comprising treating the purified acetic acid product with a cationic exchange resin to recover any silver, mercury, palladium or rhodium.
11. The process of claim 1, wherein the water concentration in the reaction medium is controlled from 0.1 to 5 wt%, based on the total amount of reaction medium present.
12. The process of claim 1, further comprising introducing a lithium compound selected from the group consisted of lithium acetate, lithium carboxylates, lithium carbonates, lithium hydroxide, and mixtures thereof into the reactor to maintain the concentration of lithium acetate from 0.3 to 0.7 wt% in the reaction medium.
13. The process of claim 12, further comprising:
    maintaining the hydrogen iodide concentration from 0.1 to 1.3 wt% in the reaction medium;
    maintaining the rhodium catalyst concentration from 300 and 3000 wppm in the reaction medium;
    maintaining the water concentration from 0.1 to 4.1 wt% in the reaction medium;
    maintaining the methyl acetate concentration from 0.6 to 4.1 wt% in the reaction medium; or a combination thereof.
14. The process of claim 1, further comprising controlling a butyl acetate concentration in the acetic acid product at 10 wppm or less without directly removing butyl acetate from the acetic acid product.
15. The process of claim 14, wherein the butyl acetate concentration is controlled by
    maintaining an acetaldehyde concentration at 1500 ppm or less in the reaction medium;
    controlling a temperature in the reactor from 150 to 250°C;
    controlling a hydrogen partial pressure in the reactor from 0.3 to 2 atm;
    controlling a rhodium catalyst concentration from 100 to 3000 wppm in the reaction medium; or a combination thereof.

16. The process of claim 1, further comprising controlling an ethyl iodide concentration in the reaction medium at less than or equal to 750 wppm.
17. The process of claim 16, wherein the propionic acid concentration in the product acetic acid is less than 250 wppm, without directly removing propionic acid from the product acetic acid;

    wherein ethyl iodide in the reaction medium and propionic acid in the acetic acid product are present in a weight ratio from 3:1 to 1:2;

    wherein acetaldehyde and ethyl iodide are present in the reaction medium in a weight ratio from 2:1 to 20:1;

    wherein an ethanol concentration in the methanol feed into the reactor is less than 150 wppm; or a combination thereof.
18. The process of claim 17, wherein the ethyl iodide concentration in the reaction medium is controlled by adjusting at least one of a hydrogen partial pressure in the carbonylation reactor, a methyl acetate concentration in the reaction medium, and a methyl iodide concentration in the reaction medium.
19. The process of claim 1, wherein the first treatment device, the second treatment device, or both individually comprise a plurality of the sampling ports arranged longitudinally at essentially equal intervals along at least a portion of the side of the corresponding treatment device.