

We claim:

1. A process for separating at least one permanganate reducing compound (PRC) from a first mixture comprising the at least one PRC, one or more C₁-C₁₂ alkyl iodides and water, the process
5 comprising the steps of:
 - feeding the first mixture to a first location of a distillation column, wherein the first mixture has a methanol mass composition that is less than the difference based on mass between the one or more C₁-C₁₂ alkyl iodides and water;
 - distilling the first mixture into a second mixture and a lower stream, the second mixture
10 being at least one stream selected from the group consisting of an overhead stream and a sidedraw stream being withdrawn higher than that of the first location; and
 - withdrawing the lower stream from a second location lower than the first location, and wherein the lower stream satisfies at least one of the following conditions (i) to (iii):
 - (i) a total acetal mass composition in the lower stream is not more than 0.02 wt%;
 - 15 (ii) a total acetal mass composition in the lower stream is less than the combined mass composition of methanol and acetaldehyde in the lower stream; and
 - (iii) a total acetal mass composition in the lower stream is less than the methanol mass composition in the first mixture;
 - wherein the first mixture is separated into the second mixture and lower stream without
20 the supply of additional methanol; and
 - wherein the second mixture is enriched in the at least one PRC.
2. The process of claim 1, wherein the lower stream satisfies conditions (i) and (ii).
- 25 3. The process of claim 1, wherein the lower stream satisfies conditions (i) and (iii).
4. The process of claim 1, wherein the total acetal mass composition for condition (i) is from 0.0001 wt% to 0.02 wt%.

5. The process of claim 1, wherein the combined mass composition of methanol and acetaldehyde for condition (ii) is not more than 1.9 wt%.
6. The process of claim 1, wherein the methanol mass composition for condition (iii) is not more than 2 wt%.
7. The process of claim 1, wherein the first mixture comprises the one or more C₁-C₁₂ alkyl iodides in an amount from 2.5 wt% to 90 wt%, the at least one PRC in an amount from 0.05 to 50 wt%, and water in an amount from 0.5 wt% to 90 wt%.
8. The process of claim 1, wherein the methanol mass composition in the first mixture is less than or equal to 2 wt%.
9. The process of claim 1, wherein the first mixture is distilled using an extractant comprising not less than 80 wt% water.
10. The process of claim 9, wherein the flow rate ratio (on a weight basis) of the extractant to the first mixture is from 0.0001/100 to 100/100.
11. The process of claim 1, wherein the second mixture comprises the one or more C₁-C₁₂ alkyl iodides in an amount from 0.5 to 95 wt%, the at least one PRC in an amount from 0.1 to 90 wt%, and water in an amount from 0.1 to 20 wt%.
12. The process of claim 1, further comprising biphasically separating the second mixture into an aqueous phase and an organic phase.
13. An acetic acid production process, the process comprising the following steps for controlling the formation of acetals:

(a) allowing methanol to continuously react with carbon monoxide in the presence of a reactive mixture comprising a metal catalyst, an ionic metal iodide, and methyl iodide, acetic acid, methyl acetate, and water;

5 (b) evaporating the reaction mixture with or without heating to yield a vapor crude product and a catalyst recycle stream;

(c) distilling at least a portion of the vapor crude product to form an overhead and a side stream and condensing the overhead in one or more condensers and collecting the condensate(s) in a receiver into an upper phase and a lower phase; and

10 (d) separating at least a portion of the lower phase to form a second stream comprising acetaldehyde and a lower stream, wherein the lower stream satisfies at least one of the following conditions (i) to (iii):

- (i) a total acetal mass composition in the lower stream is not more than 0.02 wt%;
- (ii) a total acetal mass composition in the lower stream is less than the combined mass composition of methanol and acetaldehyde in the lower stream; or
- 15 (iii) a total acetal mass composition in the lower stream is less than the methanol mass composition in the lower phase;

wherein the lower phase is separated into the second stream and lower stream without the supply of additional methanol; and

20 wherein the second stream is enriched in at least one PRC.

14. The process of claim 13, wherein the lower phase comprises one or more C₁-C₁₂ alkyl iodides in an amount from 60 to 98 wt%, the at least one PRC in an amount of up to 5 wt%, water in an amount of up to 3 wt%, and methanol in an amount of up to 5 wt%.

25 15. The process of claim 13, wherein step (d) further comprises separating at least a portion of the upper phase, wherein step (d) separates a mixture having a weight ratio of the lower phase to the upper phase from 100:0 to 10:90.

16. The process of claim 13, wherein the lower stream is returned to step (a) or step (d).

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17. The process of claim 13, wherein the at least a portion of the lower phase is distilled using an extractant comprising not less than 80 wt% of water.
18. The process of claim 13, wherein the total acetal mass composition for condition (i) is from
5 0.0001 wt% to 0.02 wt%.
19. The process of claim 13, wherein the combined mass composition of methanol and acetaldehyde for condition (ii) is not more than 1.9 wt%.
- 10 20. The process of claim 13, wherein the methanol mass composition for condition (iii) is not more than 2 wt%.