## Appendix A. Summary of key process operating conditions for the Case Study

(LOD is the level of dampness in solids)

<table>
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<tr>
<th>Stage</th>
<th>Key process conditions</th>
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| 1. Reaction | 77 wt% ± 3 wt% actA purity API feed solids and catalyst.  
2 mol eq. (ratio to feed actA) reG solids.  
10.4 mol eq. (ratio to feed actA) 30% aq. reH solution, controlled addition rate to maintain a constant temperature, T₁.  
Maintain a constant temperature, T₁ ± 1 °C, throughout entire reaction.  
Termination at ~90-95% conversion of actA (typically 2.5-3 hours). |
| 2. Dilution | 1 volume eq. (ratio to Stage 1 reH) distilled water.  
15 min agitation period. |
| 3. Layer separation | 30 min settling period.  
Drain heavy organic phase to parallel vessel. |
| 4. reH destruction | 0.4 mol eq. (ratio to feed actA) 6% aq. baseI solution per shot, pending litmus paper test for residual reH presence.  
15 min agitation period. |
| 5. reG destruction | 0.7 mol eq. (ratio to feed actA) 50% aq. baseJ solution.  
Agitate mixture for 120 min at a constant temperature, T₅. |
| 6. Layer separation | 30 min settling period.  
Drain heavy organic phase to original vessel. |
| 7. pH neutralisation | 0.7 mol eq. (ratio to actA feed) baseK solids  
15 min agitation period. |
| 8. Layer separation | 30 min settling period.  
Drain heavy organic phase to parallel vessel. |
| 9. solF distillation | Distil solF until vessel minimum stir volume is reached.  
1 bar pressure and zero reflux. |
| 10. 1st solL distillation | Add a fraction of the total solL volume:product ratio between 14 and 15.  
Distil a fraction of the solL.  
1 bar pressure and zero reflux. |
| 11. 2nd solL distillation | Add remaining fraction of the total solL volume:product ratio.  
Distil solL to achieve a final solL volume:product ratio between 7 and 8.  
1 bar pressure and zero reflux. |
| 12. Crystallisation in solL | Cool boiling mixture to 25 °C and hold for 60 min.  
1 bar pressure. |
| 13. Filtration | Vacuum filter the slurry at a constant temperature, T₁₃, until ~W₁₃% LOD is achieved. |
| 14. Washing | Rinse the damp solids with a 2 volume:product ratio of pure solL at a constant temperature, T₁₄, and refilter to the prior LOD. |
| 15. Drying | Dry with pure N₂ at a high temperature to a low LOD value ~W₁₅%. |