Run	1	2	3
Operating Conditions			
Reactor Inlet Temp, °C	140	140	140
Reactor Pressure, psig	880	880	880
Reactor Pressure, kPa	6067.39	6067.39	6067.39
LHSV, h^{-1}	2.6	2.6	3.1
Recycle Ratio, wt: wt	20	20	20
Product Analysis			
Total Unsats. (DMM + DMF), ppmwt	695	1087	1881
γ-butyrolactone, ppmwt	455	279	171
Overall Conversion, wt%	99.93	99.88	99.80

Table 2 – Test data at 140°C reactor inlet temperature

DMM = dimethyl maleate, DMF = dimethyl fumarate

Impact of higher temperature (215°C Inlet) on γ-butyrolactone formation

Experiment 1

To a trickle bed liquid recycle pilot plant unit (Fig. 1) was charged a Pd on C catalyst.

The catalyst was activated by heating under a continuous flow of hydrogen before the introduction of dimethyl succinate feed via a reciprocating pump. Once product was observed the liquid recycle was started (20: 1 wt: wt) and the feed was changed to a process stream as described in Table 1.

The unit was operated under conditions shown in Table 3.

Parameter	**Target**
Reactor Inlet Temperature, °C	215
Reactor Pressure, psig	880
Reactor Pressure, kPa	6067.39
LHSV, h^{-1}	3.0
Recycle Ratio, wt: wt	20: 1

Table 3 – High temperature operating conditions

The unit was operated under these conditions for 220 hours, during which time the overall conversion stabilised at approximately 99.37 wt%, with key components at the follow levels: dimethyl maleate 4390 ppmwt, dimethyl fumarate 1536 ppmwt and γ-butyrolactone 4161 ppmwt.

Experiment 2

Experiment 1 was repeated but with aged catalyst to lower conversion. Once stable this afforded an overall conversion of 97.93wt%, with key components at the follow levels: dimethyl maleate 13768 ppmw), dimethyl fumarate 5669 ppmwt and γ-butyrolactone 550 ppmwt.

Experiment 3

Experiment 1 was repeated with further aged catalyst, which had undergone a high temperature regeneration step. Once stable this afforded an overall conversion of 99.28wt%, with key components at the follow levels dimethyl maleate: 5117 ppmwt, dimethyl fumarate 1694 ppmwt and γ-butyrolactone 1674 ppmwt.

<u>Demonstrating low γ-butyrolactone formation / high conversion when operating at low temperature – Ni catalyst</u>

To a trickle bed reactor as described in Example 1 was charged a Ni on Al_20_3 / SiO_2 catalyst (62.5 mL).

The catalyst was activated by heating under a continuous flow of hydrogen before the introduction of dimethyl succinate feed via a reciprocating pump. Once product was observed the liquid recycle was started (20: 1 wt: wt) and the feed was changed to the feed shown in Table 1.

The Liquid Recycle unit was operated under the conditions shown in Table 4 using the same feed shown in Table 1.

Parameter	**Target**
Reactor Inlet Temperature, °C	75
Reactor Pressure, psig	880
Reactor Pressure, kPa	6067.39
LHSV, h^{-1}	3.5
Recycle Ratio, wt: wt	20: 1

Table 4 – Low temperature operating conditions

The unit was operated under these conditions for 49 hours, with dimethyl maleate stabilising at 41 ppmwt in the crude product. No dimethyl fumarate or γ-butyrolactone were observed.

Fig. 3 shows a comparison of γ-butyrolactone formation vs overall conversion of unsaturated compounds at 75°C (Ni catalyst), 140°C and 215°C (Pd catalyst).

Demonstrating second hydrogenation stage – polisher reactor

The unit was operated under the conditions shown in Table 5 using the Ni on Al_20_3 / SiO_2 catalyst. The feed for this run was crude product from previous runs, as shown in Table 6, such that the reactor is operating as a second hydrogenation stage in accordance with the invention.

Parameter	**Target**
Reactor Inlet Temperature, °C	85
Reactor Pressure, psig	880
Reactor Pressure, kPa	6067.39
LHSV, h^{-1}	3.5
Recycle Ratio, wt: wt	20: 1

Table 5 – Second reactor operating conditions