



Standard Operating Procedure

Purchase, Delivery, Control and Collection of Ethanol

1. OBJECTIVE

This Standard Operating Procedure defines the procedure used in the Purchase, Delivery, Control and Collection of Ethanol in LEO Pharma Cork.

Ethanol 93% w/v batches are generated as per SOP_002755 'Recovery of Ethanol 93% w/v Reg. from Mother Liquor'.

2. SCOPE

This SOP details the instructions for Production Operators, Team Leaders, Process Flow Specialists and Operations Specialist Scheduler when Purchasing, Obtaining a Delivery, and the Control & Collection of Ethanol.

3. REASON FOR UPDATE

- Updated to align with SOP_002755 'Recovery of Ethanol 93% w/v Reg. from Mother Liquor' to clarify the steps required when Ethanol 93% W/V batch is for backload.
- Numbering corrected

4. DEFINITIONS

- TL Team Leaders
- PFS Process Flow Specialists
- OSS Operations Specialist Scheduler

5. RESPONSIBILITY

Production Operators, Team Leaders, Process Flow Specialists and Operations Specialist Scheduler are responsible for purchasing, delivery, control and transfer of Ethanol Absolute 99.9% and the Collection, control and transfer of Ethanol 93% Regen. They are responsible for compliance with SOP_002734.

6. PROCEDURE

This procedure is divided into the following sections:

- 6.1 Ordering of Ethanol Absolute 99.9%.
- 6.2 Safety Precautions during Ethanol Transfer
- 6.3 Arrival of Road Tanker
- 6.4 Unloading of Road Tanker
- 6.5 Loading of Road Tanker (Backload)
- 6.6 Control of Ethanol Absolute 99.9%
- 6.7 Control of Ethanol 93% Regen.
- 6.8 Transfer of Ethanol 93% w/v Regen. between Storage Tanks HTTF02 (25000L) and HTTF07 (25000L)



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6.1. ORDERING OF ETHANOL ABSOLUTE 99.9%

Ethanol Absolute 99.9% is ordered approx. 1-2 weeks prior to its' required date. The order date is calculated based on the quantities of ethanol in both Ethanol Absolute Storage Tanks (HTTF09 and HTTF03) and the expected consumption in production. The Bulk Tank Filling Guide detailed in the Road Transport Regulations (ADR) indicates the maximum and minimum filling ratios required as follows: Contact MAINTENANCE if in doubt.

Note: The minimum draw-off level for HTTF03 is 2419L and for HTTF09 is 350L

Unbaffled Tanker	Baffled Tanker
91-100% Not Allowed 80-90% Allowed 21-79% Not Allowed <20% Allowed	Baffled tanker must be filled as per Unbaffled Tanker or so that the liquid level is at the baffle.

The supplier is responsible for advising the exact date and time of delivery. Both 92/12 EEC and the Finance Act 1992 must be complied with. The Registered Trader No. for Leo Pharma cork (Wexport Ltd.) **Warehouse IETW000012629 Warehouse Keeper IEWK000015762**.

- 6.1.1 Order the required quantity of Ethanol Absolute. Advise on suitable delivery date and time.
- 6.1.2 Notify Quality/QC of the planned ethanol Road Tanker arrival.
- 6.1.3 Notify Customs and Excise of the planned delivery date >24hrs prior to delivery.

6.2. SAFETY PRECAUTIONS DURING ETHANOL TRANSFER.

Note: At all times during any Ethanol transfer the following must be adhered to:

- All flexible hoses must be metal braided and PTFE lined for solvent transfer
- Ethanol is a highly flammable liquid, safety precautions must be followed and appropriate PPE must be worn at all times.
- Operator present at all times during operations with Road Tanker e.g. Road Tanker arrival, unloading, loading and despatch
- No work, traffic other than emergencies/repairs should be carried out during the delivery of ethanol
- All non ex-rated equipment must be kept clear of area
- The 92/12 EEC and the Finance Act 1992 must be complied with
- Manage any spillage in accordance with SOP_002857 and SOP_002858

- 6.2.1 Ensure the area is free from obvious hazards e.g. sources of ignition, equipment not required for the Operation.
- 6.2.2 Place an appropriate fire extinguisher near the operation area.
- 6.2.3 Clear the immediate area of all other vehicles including fork-lifts.
- 6.2.4 Place portable hazard signs at both the front and rear of the docking area.
- 6.2.5 Ensure the Earth Connections are plugged in at all times to Road Tanker and Loading Bay.
- 6.2.6 Ensure one of the Road Tanker back wheels is 'chocked' on both sides at all times to stop movement of road Tanker either forwards or backwards.
- 6.2.7 Personnel must be present at all times during transfer to monitor hazards e.g.:
 - Leaks and spillages
 - Sources of heat ignition
 - Any other irregularities



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If any hazard is identified during transfer or if the person monitoring the transfer must leave for any reason, then he/she must perform the following:

- Stop the transfer pump using the pump Stop button.
- Close all appropriate valves on feed and receiving vessels e.g. On the line from Road Tanker to HTTF03 and HTTF09 and on the line between HTTF03 to HTTF09
- **Close** Valves SOLVTF 11, 18, 39 and 40
- **Close** Valves SOLVTF 01, 02, 03 and 04
- Inform a PFS immediately to initiate SOP_002857.
- If spillage occurs manage same in accordance with SOP_002857.

6.3. ARRIVAL OF ROAD TANKER

- 6.3.1 Carry out Safety Precautions in accordance with Section 6.2
- 6.3.2 Check the delivery note to ensure that the correct load has arrived.
- 6.3.3 Direct the driver to the Bulk Storage Loading Bay and instruct to remain there until further notification.
- 6.3.4 Position two 'Chocks' on a back wheel of the Road Tanker, one on either side to stop movement of road Tanker either forwards or backwards.
- 6.3.5 Ensure the Earth Connection from the Earth-Rite RTR earth monitoring system is attached to Road Tanker.

Note 1: This must remain attached to the tanker all the times if disconnected the pump will not function as it will be interlocked by the system.

- 6.3.6 Position the Road Tanker Ladder on the side of the road tanker to allow safe access to the Road Tanker Manhole

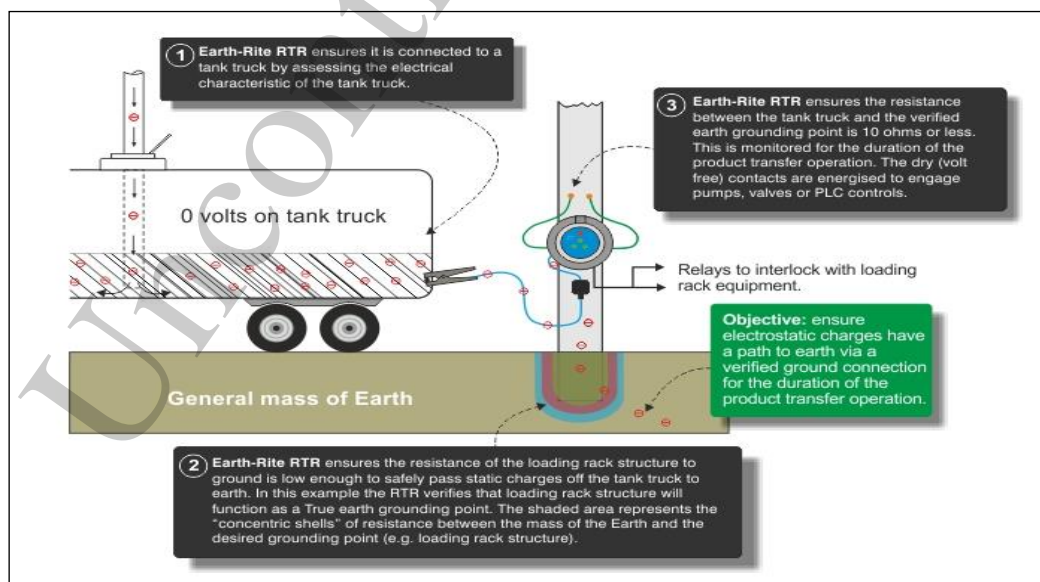


Diagram 1: How the Earth-Rite RTR system functions.

- 6.3.7 Before the tanker truck that delivered the tanker can leave a good earth (<10 Ohms) must be confirmed from the Earth-Rite unit, this is indicated by 3 green flashing LEDs on the unit.



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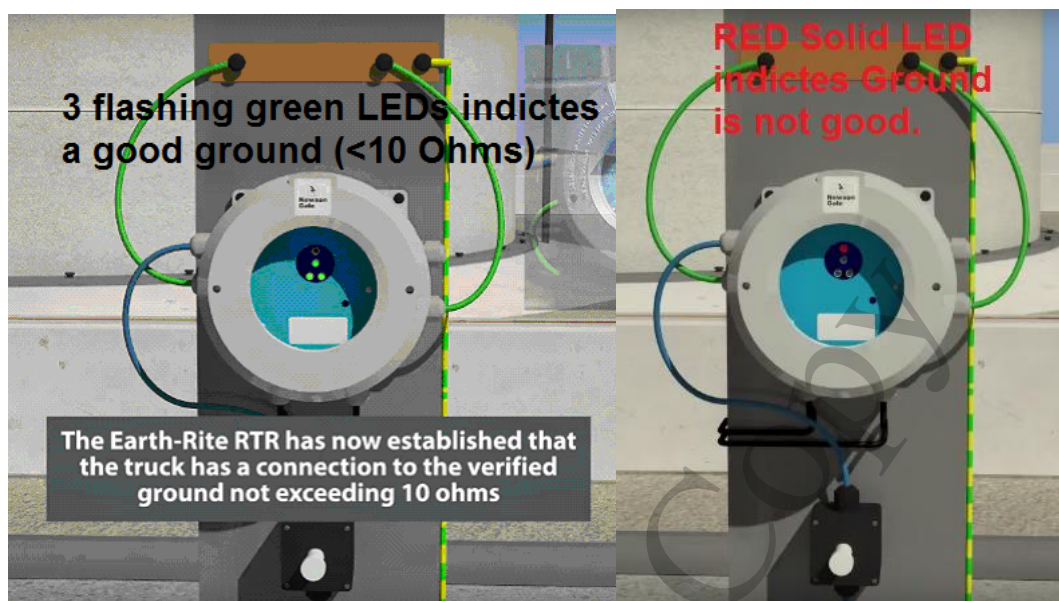


Diagram 2: Green (3 LEDs) illuminated flashing indicates good ground (<10 Ohms), Single Red LED illuminated solid indicated a poor ground (>10 Ohms)

- 6.3.8 Allow the Road Tanker to settle for 30 minutes before any operation takes place.
- 6.3.9 Check Road Tanker Security Seals are intact (proof of tampering) and confirm numbers against Delivery Docket. Record same on Delivery Docket. Report any discrepancies to PFS immediately.
- 6.3.10 Slowly open the air vent on the top of the Road Tanker to allow tanker to vent.
- 6.3.11 Inform Maintenance that Road Tanker is ready for connection of permanent transfer line to Tank Farm pipework.
- 6.3.12 Remove the Shore Covering Plate in the Loading Bay area.
- 6.3.13 Close the Surface Water Penstock Valve located near the effluent tank prior to loading and unloading of the tankers, to ensure that any accidental spillage is contained. Then open the valve when finished.
- 6.3.14 Generate a Product control as per SOP_002834.
The Product control card for the Ethanol Tanker must have the manufactures Batch number.
The Manufactures Batch number is printed on the Suppliers Note and INEOS paperwork .
Delivery Number (Example 80352261) Record this number as the Leo Pharma batch number on the product control card, and forward to the QC Laboratory.

6.4. UNLOADING OF ROAD TANKER

- 6.4.1 Ensure the Road Tanker has been approved for transfer to the Tank Farm by Quality.
- 6.4.2 Receipt the new Ethanol Absolute 99.9%. batch on the SAP system in accordance with WI_005726. Generate a SAP Product ID label and label the designated storage tank to receive the new load of Ethanol Absolute 99.9%.
 - Material No.
 - Material Name
 - Batch No
- 6.4.3 Carry out Safety Precautions in accordance with Section 6.2
- 6.4.4 Collect the solvent keys from the PFS



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6.4.5 Prior to unloading ensure the following:

- Designated Tank to receive the load is clean and fit for use
- The controlling valves from the designated tank to receive the load to Production are closed and locked:
- Filling into HTTF03: **Close** SOLVTF01 and SOLVTF02
- Filling into HTTF09: **Close** SOLVTF03 and SOLVTF04
- Ensure the supply to R1.17 is provided by the second Ethanol Absolute 99.9% storage tank which must have a status of 'Unrestricted Use', reference WI_006154.

6.4.6 Ensure the Surface Water Penstock is closed and the Shore Covering Plate is open near the pump station.

6.4.7 Ensure that the Road Tanker brakes are secured and wheel 'Chocked' (Step 6.3.4).

6.4.8 Read the tank level of the Road Tanker and of both HTTF03 and HTTF09 in accordance with SOP_002766.

6.4.9 Confirm that the designated tank to receive the load has capacity to take the load, if not contact a PFS.

6.4.10 Inform Maintenance to connect the road tanker to the designated Ethanol Absolute 99.9% Storage Tank.

6.4.11 Set up the lines to the designated storage tank as appropriate:

Filling into HTTF03: **Open** SOLVTF40, **close** SOLVTF39 and SOLVTF05
 Filling into HTTF09: **Open** SOLVTF39 and SOLVTF05, **close** SOLVTF40

6.4.12 Open Valves SOLVTF11 and SOLVTF18 on the Ethanol Absolute transfer line.

6.4.13 Inform PFS to perform a check on the valve set-up performed in Steps 6.4.5, 6.4.11 and 6.5.12.

6.4.14 Once the valve set-up is checked by PFS, start the pump to transfer Ethanol from the Road Tanker into the designated storage tank.

6.4.15 Close and secure valves SOLVTF 11 and 18, and close the valve on the line to the designated storage tank as appropriate:

Filling into HTTF03: **Close** SOLVTF40, **check** SOLVTF39 and SOLVTF05 are closed
 Filling into HTTF09: **Close** SOLVTF39 and SOLVTF05, **check** SOLVTF40 is closed

6.4.16 Inform PFS to perform a check on the valve set-up.

6.4.17 Once the valve set-up is checked by PFS, read the tank levels of Tank HTTF03 and HTTF09 in accordance with SOP_002766. Inform OSS who will perform the relevant SAP transactions to reflect tank volumes in accordance with WI_005699.

6.4.18 Generate a Product control and forward to the QC Laboratory who will sample the batch.

6.4.19 If there is no backload, inform Maintenance to disconnect the Road Tanker from the permanent transfer hose to the Tank Farm pipework system. Once Road Tanker is disconnected, Maintenance must seal the permanent transfer hose on the Tank Farm pipework system. Production operator must store the ladder, chocks and any flexible hoses in designated storage areas. Return the solvent keys to a PFS.

6.4.20 At the start of every month details of the deliveries are forwarded to Customs & Excise by the OSS.



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6.5. LOADING OF ROAD TANKER (BACKLOAD.)

Note: Report any spillage immediately to a PFS. If required SOP_002857 will be initiated. Spillage is disposed of in accordance with SOP_002858.

- 6.5.1 Read the tank levels of the Ethanol 93% w/v Regen. Storage Tank (HTTF02, HTTF07) in accordance with SOP_002766.
- 6.5.2 Once the Ethanol 93% w/v Regen has reached a full tank in the tank farm a Product Control is completed and submitted to the QC lab so the batch can be sampled by QC. Once Sampled by QC the batch can be pumped to the designated Road tanker, as detailed below. When QC testing and Quality approval have been performed. The road tanker can leave site.
- 6.5.3 Confirm that the Road Tanker has the capacity to take the volume of Ethanol Regen.
- 6.5.4 Carry out Safety Precautions in accordance with Section 6.3
- 6.5.5 Open valves SOLVTF76 and SOLVTF15 on the Ethanol regen transfer line.
- 6.5.6 Connect the Road Tanker to the Ethanol 93% w/v Regen. Storage Tank by opening SOLVTF23 to connect HTTF02 and SOLVTF22 to connect HTTF07.
- 6.5.7 Inform PFS to perform a check on the valve set-up performed in Steps 6.5.5 and 6.5.6.
- 6.5.8 Once the valve set-up is checked by PFS, using ATF-SPUMPTF08 Pump, transfer the Ethanol 93% w/v Regen. from the Ethanol Regen Storage Tank (HTTF02, HTTF07) to the Road Tanker.
- 6.5.9 Once transfer is complete, close and secure all valves.
- 6.5.10 Seal valves on tanker using Leo numbered security tie-wraps, recording numbers on return shipment paperwork ('proof of tampering').
- 6.5.11 Inform PFS to perform a check on the valve set-up performed in Step 6.5.10.
- 6.5.12 Once the valve set-up is checked by PFS, read the tank level of the emptied Ethanol Regen. Storage Tank (HTTF02, HTTF07) as per SOP_002766. The OSS . will perform SAP transactions in accordance with WI_005699 or WI_005721 to reflect the change in tank levels.
- 6.5.13 Return solvent keys to a PFS.
- 6.5.14 Inform Maintenance to disconnect the Road Tanker from the permanent transfer hose to the Tank Farm pipework system.
- 6.5.15 Once Road Tanker is disconnected, Maintenance must seal the permanent transfer hose on the Tank Farm pipework System. Production operator must store the ladder, chockes and any flexible hosing in designated storage areas.
- 6.5.16 The following paperwork has to be completed for the backload: Dangerous Goods Note.
- 6.5.17 When QC testing and Quality approval have been performed. The road tanker can leave site.



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6.6 CONTROL OF ETHANOL ABSOLUTE 99.9%

A controlled batch system is in place to segregate each batch of Ethanol Absolute. The new batch is unloaded from the Road Tanker into the designated storage tank while production uses the Ethanol Absolute from the second storage tank. The Supply Valves from the newly filled tank remain locked until required for use. When changing from one batch to another of Ethanol Absolute, isolate the emptied storage tank. Ethanol may be drawn from a storage tank into production only when the following criteria are met:

- The batch has been assigned a status of 'Unrestricted Use'
- The storage tank and both supply points in R1.17 are labelled with a SAP Product ID label.
- The valve system to supply to production is correctly set up and checked by PFS as follows:

HTTF03 to Production: **Open** SOLVTF-01, SOLVTF-02 and SOLVTF-09
 Close SOLVTF-03 and SOLVTF-04

HTTF09 to Production: **Open** SOLVTF-03, SOLVTF-04 and SOLVTF-09
 Close SOLVTF-01 and SOLVTF-02

6.7 CONTROL OF ETHANOL 93% W/V REGEN.

A controlled batch system is in place to segregate each batch of Ethanol Regen. Each Ethanol Regen. Tank stores one batch of Ethanol Regen. at any one time.

A new batch of Ethanol 93% w/v Regen. is created either by distillation of Mother Liquor in accordance with SOP_002755 or by transfer from another storage tank. Once Ethanol is added to either of the Ethanol 93% w/v Regen. Storage Tanks it is deemed a new batch.

Once the distillation is finished and the batch completed the ethanol 93% w/v Regen. is prepared for sampling in accordance with the procedure below.

- 6.7.1 Ensure that the batch of Ethanol 93% w/v Regen in the storage tank is complete and is labelled a SAP Product ID Label in accordance with SOP_002755.
- 6.7.2 Read the tank level in accordance with SOP_002766. The OSS will perform SAP transactions to reflect the change in tank levels in accordance with WI_005699 to reflect the change in tank levels.
- 6.7.3 Forward a Product Control to the QC Laboratory, detailing the tank number and new batch information and test requirements depending on the new batch designation e.g.
 - Full testing if new batch is designated for Production Processes.
 - Reduced testing if the batch is designated for backload.

Ethanol 93% W/V Regen. Required for Backload:

When a new batch of Ethanol 93% W/V Regen. is complete in a storage tank, a Product Control is submitted to the QC lab so the batch can be sampled by QC.

Once Sampled by QC the batch can be pumped to the designated Road tanker.

When QC testing and Quality approval have been performed. The road tanker can leave site.

Ethanol 93% W/V Regen. Required for Production Use:

When a new batch of Ethanol 93% W/V Regen. is complete in a storage tank the batch is re-circulated as detailed in SOP_002755, and sampled by QC. When the batch status is changed to 'Unrestricted Use' by the Quality Department it may be used for processing.

While one tank is in use the second storage tank may be filled from the Distillation Column in accordance with SOP_002755. The Supply Valves from the newly filled tank remain locked until required for use. When changing from one batch to another of Ethanol Regen 93%, the emptied storage tank valves are closed to isolate the tank.



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6.7.4 Ethanol Regen. 93% can only be drawn into production from a storage tank only when the following criteria are met:

- The batch has been assigned a status of 'Unrestricted Use'
- The storage tank and both supply points in R1.17 are labelled with a SAP Product ID label.
- The valve system to supply to production is correctly set up and checked by PFS as follows:

HTTF02 to Production: **Open** SOLVTF-21 and MV TF43, **close** SOLVTF-20 and MV TF44

HTTF07 to Production: **Open** SOLVTF-20 and MV TF44, **close** SOLVTF-21 and MV TF43

If both the Ethanol Regen. 93% storage tanks have a status of 'Quality Inspection' or during a shutdown period, ensure the following:

HTTF02 to Production: **Close** SOLVTF-21 and SOLVTF-20, MV TF43

HTTF07 to Production: **Close** SOLVTF-20 and SOLVTF-21, MV TF43

6.8 TRANSFER OF ETHANOL 93% W/V REGEN. BETWEEN STORAGE TANKS HTTF02 AND HTTF07

Carry out safety precautions in accordance with Section 6.3 and inform production that there is no supply of Ethanol 93% w/v Regen during the transfer.

6.8.1 Collect the solvent keys from PFS

6.8.2 Read the tank levels of both Ethanol 93% w/v Regen. storage tanks and record in accordance with SOP_002766. The OSS will perform SAP transactions to reflect the change in tank levels in accordance with WI_005699 or WI_005721 to reflect the change in tank levels.

Note: The minimum draw-off level in these tanks is 3,200L.

6.8.3 Close valve PTF3V03 to ensure no draw off from either storage tank to production.

6.8.4 Set-up the valve system as appropriate.

Into HTTF07 **Close** valve SOLVTF-26, SOLVTF-32 and SOLVTF-38.

Open valves SOLVTF-33, SOLVTF-34, SOLVTF-36 and SOLVTF-37.

Into HTTF02 **Close** valve SOLVTF-33, SOLVTF-34 and SOLVTF-37.

Open valves SOLVTF-26, SOLVTF-32, SOLVTF-38 and SOLVTF-36.

6.8.5 Inform PFS to perform a check on the valve set-up performed in Steps 6.8.3 and 6.8.4.

6.8.6 Once the valve set-up is checked by PFS, start Pump PUMPTF05 and transfer the required volume of Ethanol 93% w/v Regen. to the designated storage tank.

6.8.7 Close all opened valves from Step 6.8.4.

6.8.8 Inform to perform a check on the valve set-up performed in Step 6.8.7.

6.8.9 Once the valve set-up is checked by PFS, read the level of both tanks to reconcile the transfer and record in accordance with SOP_002766.

6.8.10 Prepare the new batch of Ethanol 93% Regen in accordance with Section 6.7.

6.8.11 Return the solvent keys to a PFS



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7.0 ENCLOSURES

N/A

8.0 REFERENCES

SOP_002755 'Recovery of Ethanol 93% from Mother Liquor'

SOP_002857 'Procedure to deal with an environmental emergency'

SOP_002858 'Collection, Storage, Disposal and of Waste'

SOP_002766 'Measurement of Tank Farm Tank Volumes'

SOP_002834 'Material Status Control and Labelling of Raw Materials, Packaging Materials, Intermediates and APIs'

WI_006154 'Check Inventory Status - MMBE'

WI_005699 'Globe - DTS - batch to batch'

WI_005721 'Globe - DTS - Stock Count IM'

WI_005726 'Globe - DTS - Vendor Purchase Order GR'

9.0 CHANGE LOG

11.0	- Section numbering has been corrected - Section 9.0 has been removed and included in section 8.0 (correctly renamed 6.7) - Section 6.5.2 - Instructions regarding the control of Ethanol Regen have been corrected to align with SOP_002755. - Step 6.3.5 - Remove note 2 as both points are now monitored. - Section 6.7 - Remove requirement to re circulate the backload as this is no longer the practice.
10.0	- Updated the number format for all sections and updated section references in the document. - Updated section 1 to include SOP_002755 Recovery of Ethanol 93% w/v from Mother Liquor. - Updated section 2 removing SOP number. - Updated section 3 to include CC 188152 - Updated section 4 references. - Updated section 5 to include PFS,TL, Production & scheduler. - Updated section 6.2.7 to remove SOLVTF12 & SOLVTF14. - Updated section 6.3.13 to include the penstock valve will be closed prior to loading and unloading of the tankers and open the valve when finished. - Updated section 6.4.12 to remove SOLVTF12 & SOLVTF14 and to include Ethanol Absolute transfer line. - Updated section 6.4.15 to remove SOLVTF12 & SOLVTF14. - Deleted section 6.5.5 and updated the remaining section numbers accordingly. - Updated section 6.5.5 to remove SOLVTF16,17,14,13 & include SOLVTF76,15 and to include reference to Ethanol Regen transfer line. - Update section 6.5.8 to include the pump plant item number. - Deleted section 16.0 Detailing cleaning of Pump and hose because the lines are segregated and updated section 6.0 to remove reference to section 16.0. - Updated section 12.0 to remove WI_005766.

Document Approvals

Approved Date: 16 Jul 2019

Technical Approval Task Verdict: Approve	Clodagh Crowley (CCWIE), Senior Professional (ccwie@leopharma.com) Technical Approval 01-Jul-2019 07:53:27 GMT+0000
Management Approval Task Verdict: Approve	Malcolm Garde (MGGIE), Operations Manager (mggie@leopharma.com) Management Approval 01-Jul-2019 12:18:36 GMT+0000
Quality Approval Task Verdict: Approve	Denise Sheehan (DDLIE), Professional (ddlie@leopharma.com) Quality Approval 16-Jul-2019 11:35:04 GMT+0000