

School of Chemistry SOP For Operation Of Grubbs-Type Dry Solvent Stills

School SOP for Operation of Grubbs-Type Dry Solvent Stills:

This SOP describes the operation of the PureSolv 400-3-MD Solvent Purification System. It includes the general procedures for:

- Dispensing dry solvent
- Refilling solvent kegs

Safety Information:

- When dispensing solvent, never fill the flask more than 2/3 full
- Gauges should be set as follows at all times:
 - Nitrogen supply gauge (building supply): ~ 50 psi
 - Gas manifold gauges: ~ 3 psi
 - Keg regulator gauges: ~ 15 psi
- The gas manifold gauges are equipped with pressure relief valves. These safety valves are set to release any pressure above 7 psi. If pressure exceeds 7 psi a “hissing” sound may be observed. If this happens check that all the gauges are set as described above.
- Pump exhaust should be vented.
- Over time or through frequent use, the so-called “5-way valve” (evacuate/dispense/refill valve) may become loose and require tightening. If any of the following are observed seek further assistance:
 - If valve feels loose when turning between evacuate/dispense/refill
 - If solvent is observed in vacuum line
- To protect the 5-way valve, the green solvent tap should be kept closed when the system is not in use.
- To ensure optimum quality, solvent should be dispensed into a Straus flask or a drop-funnel type collection vessel which is kept in place at all times (to protect dispensing line from atmospheric moisture).
- Solvent used in the system must not contain any inhibitors/stabilisers.
- The quantity of solvent added to each keg during the refill processes must be recorded in order to allow estimation of when cylinder reactivation is due.

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- Each cylinder should be reactivated once approximately 400 L of solvent has passed through that particular system. Seek further assistance when this is due.
- Never leave “evacuate/dispense/refill” valve on “refill” if there is solvent in the dispensing vessel (this creates route for solvent to ingress and damage gas manifold gauges).

Dispensing:

Before commencing, refer to section above.

- Turn on house nitrogen supply for dry solvent system.
- Switch on pump.
- Open the green solvent tap by turning half a turn anticlockwise.
- Check that collection vessel is empty and tap is closed.
- Turn to “Evacuate” until level on vacuum gauge reaches approx. 25 psi.
- When vessel is evacuated turn valve to “Refill” (*via* “Closed” rather than “Dispense”) until level on nitrogen gauge reaches approx. 3 psi.
- Repeat the two steps above a total of three times.
- Evacuate once more and then turn valve to “Dispense”.
- When dispensing complete, turn valve to “Refill” momentarily in order to back fill collection flask with inert gas, also clearing dispensing line of solvent preventing dripping of solvent. (After dispensing, do not turn to closed *via* “Evacuate”, this will cause solvent to enter system causing solvent degradation of parts, if this accidentally occurs leave the pump on for *ca.* 1 hour to clear solvent in system).
- Turn valve to “Closed” position.
- Switch off pump.
- Close green solvent tap by turning half a turn clockwise.
- Turn off house nitrogen supply at wall.

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Refilling Solvent Kegs (Sparging/Purging):



Before commencing refer to the first section.

- Close solvent line (at Port #4) and disconnect it.
- Disconnect the solvent keg from solvent purification system at nitrogen regulator (Port #1).
- Connect a 1 inch line (supplied in safety cupboard) to the two-way valve at Port #2 and run line to fume hood or venting point.
- Carefully pull keg out of safety cupboard and depressurize it slowly by turning the two-way valve at Port #2 to open position.
- Once depressurized, close two-way valve at Port #2 and disconnect nitrogen line.
- Move keg to fumehood.
- Carefully open the two-way valve at Port #2 to ensure depressurization is complete.
- Remove lid and fill " " full with solvent (NB-note quantity of solvent added).
- Replace lid (to "hand tightness").
- Close two-way valve at Port #2. Return keg to safety cupboard and reconnect line to two-way valve at Port #2 and open it.
- Connect nitrogen regulator to the three-way valve at Port #4.
- Turn handle on three-way valve very slowly toward the regulator until a gentle nitrogen flow is achieved (feel side of keg for indication).
- Purge solvent for 45 mins (this is the time required for a 15 L/4 gallon keg).
- When purging is complete, turn inlet (Port #4) and outlet (Port #2) valves on keg to closed positions.
- Disconnect nitrogen regulator from Port #4 and reconnect to Port #1 (head pressure inlet).
- Re-secure keg in the solvent purification safety cupboard and open solvent line (Port #4) to system.

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Quick Guide



Grubbs-Type Dry Solvent Stills

PureSolv 400-3-MD

1. Turn on house nitrogen supply slowly and ensure the pressure is *ca* 50 psi.
2. Switch on pump.
3. Open the green solvent tap by turning half a turn anticlockwise.
4. Ensure solvent collection vessels are empty and taps are closed.
5. Turn valve to "Evacuate" until level on vacuum gauge reaches approx. 25 psi.
6. Turn valve to "Refill" (*via* "Closed" rather than "Dispense") until level on nitrogen gauge reaches approx. 3 psi.
7. Repeat steps 4 & 5 a total of three times.
8. Evacuate once more and then turn valve to "Dispense".
9. When dispensing complete, turn valve to "Refill" momentarily and then turn valve to closed position.
10. Switch off pump.
11. Close green solvent tap by turning half a turn clockwise.
12. Turn off house nitrogen supply.
13. Dispense collected dry solvent with the aid of a nitrogen balloon.

Note:

- After dispensing do not turn to closed *via* "Evacuate".
- Never leave on "refill" if solvent in is the dispensing flask.