The Ohio State University
Department of Chemistry

Standard Operation Procedures for the
Operation and Maintenance of Solvent Stills:

Hexanes
Tetrahydrofuran
Diethyl Ether
Dimethoxyethane
Benzene
Toluene
Dichloromethane

Prepared in accordance with the Department of Chemistry's
Chemical Hygiene Plan for:

Prof. T. V. RajanBabu

Prepared by:

Sandra Greau
Brian Bliss

Updated and Approved:

January 29, 1998

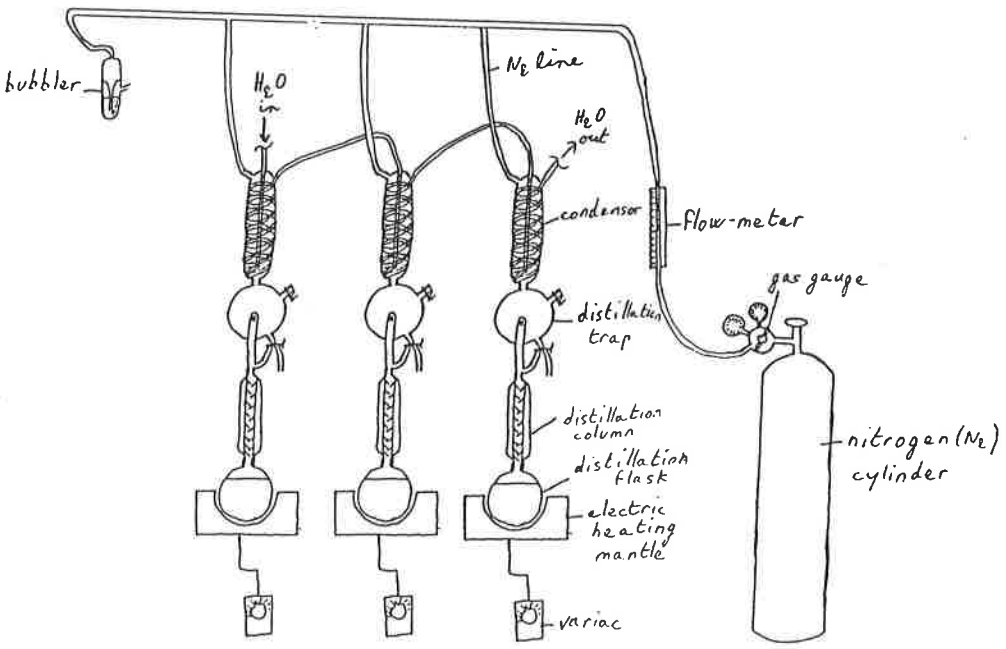
STANDARD OPERATION PROCEDURE (SOP) FOR THE HEXANES STILL

(last updated : Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1. Description of the operations of the still

a) Diagram



b) Description of operation. The hexane is distilled under a nitrogen atmosphere from the distillation flask containing metallic sodium (Na) and benzophenone. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the stopcock at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Furniss, B.S. et al., eds.; Longman: Singapore, 1989. p.397.
3. *Purification of Laboratory Chemicals* ; Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; 2nd Ed.; Pergomon: Oxford, 1980. p.283.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories* ,National Research Council, National Academy Press: Washington, 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2. Hazards Involved in the Operation of the Hexanes stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a toxic vapor hazard; water use in and around metallic sodium poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a toxic vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- Hexanes
- Drying agent : metallic sodium (Na)
- Benzophenone
- 2-propanol
- Absolute methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent to be distilled is then poured into the round-bottom flask to no more than 2/3 full.

3. In a fume-hood, a small amount of metallic sodium is cut under oil into small pieces, and the sodium quickly rinsed with a *minimum* amount of hexanes to remove the oil and the sodium added to the hexane distillation flask. (Note: Sodium metal and hexanes is a potential fire hazard. Ensure a dry-powder fire extinguisher is near by and accessible

before you begin working with sodium. Residual solutions of hexanes containing sodium are later *carefully* quenched with 2-propanol and several hours later *carefully* quenched with absolute methanol and finally water.)

4. Add 250 mg of benzophenone to the distillation flask and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, hexane containing the sodium/benzophenone will yield a deep blue-green color.

5. If, after several hours of reflux, the color of the solution is not dark blue-green, repeat steps 2 through 5.

4. Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full.
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5. Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual hexane is *carefully* quenched with small amounts of 2-propanol added under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual hexane is then *carefully* quenched with small amounts of absolute methanol over several hours until all sodium metal is destroyed. Now add excess water (carefully) under nitrogen and dispose of the contents.

6. Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

Dr. T.V. RajanBabu - 688-3543
John Herrington - Safety Coordinator (ph : 688-3957; pager : (9)-605-5075)
Don Tong - Hazardous Waste Specialist (ph.: 688-3957; pager : (9)-605-5076)
Dr. Malay Nandi - 3051 Evans Lab, ph: 688-3694

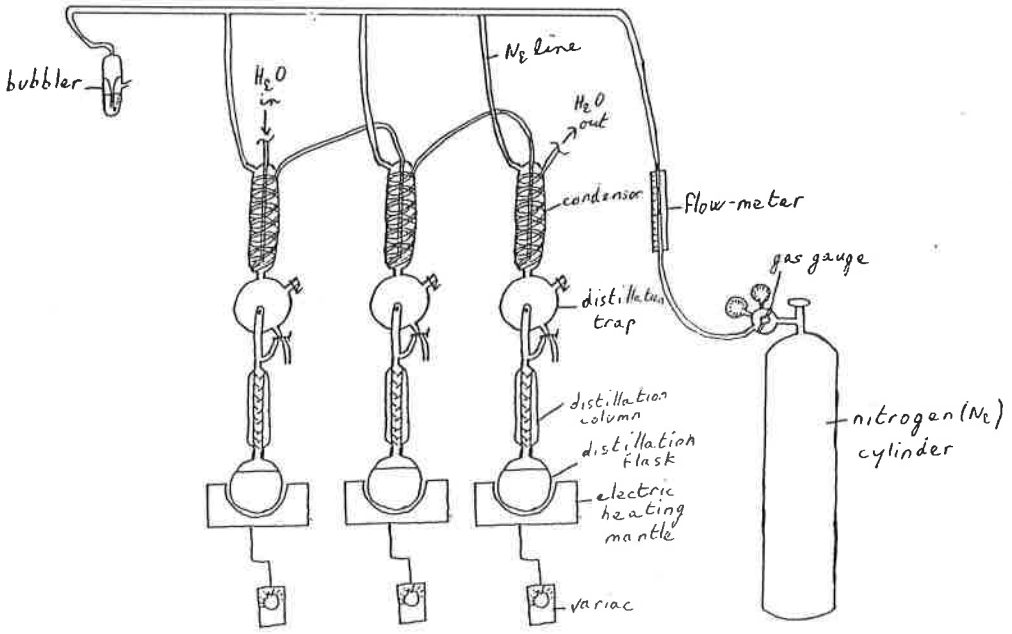
STANDARD OPERATION PROCEDURE (SOP) FOR THE TETRAHYDROFURAN (THF) STILL

(last updated : Jan 1997)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The tetrahydrofuran is first pre-dried for several days over potassium hydroxide (KOH) and then distilled under a nitrogen atmosphere from the distillation flask containing metallic sodium (Na) and benzophenone. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.406.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.426.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the THF Stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- Tetrahydrofuran
- Drying agents: 1. Potassium hydroxide (KOH)
  2. metallic sodium (Na)

- Benzophenone
- 2-propanol
- Absolute methanol
- Hexanes

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The solvent is pre-dried by storing over potassium hydroxide (KOH) several days before distillation.:

2. The still is cooled down to room temperature (ensure variac is off).

3. The pre-dried solvent is then poured into the round-bottom flask.

4. In a fume-hood, a small amount of metallic sodium is cut under oil into small pieces, and the sodium quickly rinsed with a *minimum* amount of hexanes to remove the oil and the sodium added to the THF distillation flask. (Note: Sodium metal and hexanes is a potential fire hazard. Ensure a dry-powder fire extinguisher is near by and accessible before you begin working with sodium. Residual solutions of hexanes containing sodium are later *carefully* quenched with 2-propanol and several hours later *carefully* quenched with absolute methanol and finally water.)

5. Add 250 mg of benzophenone to the distillation flask and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, THF containing the sodium/benzophenone will yield a deep blue color.

6. If after several hours of reflux, the color of the solution is not dark blue, repeat steps 2 through 5.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual THF is *carefully* quenched with small amounts of 2-propanol added under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual THF is then *carefully* quenched with small amounts of absolute methanol over several hours until all sodium metal is destroyed. Now add excess water (*carefully*) under nitrogen and dispose of the contents.

6.Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

Dr. T.V. RajanBabu -
John Herrington - Safety Coordinator (ph : 8-3957; pager : (9)-605-5075)
Don Tong - Hazardous Waste Specialist (ph.: 8-3957; pager : (9)-605-5076)
Dr. Malay Nandi - 3051 Evans Lab, ph: 688-3694

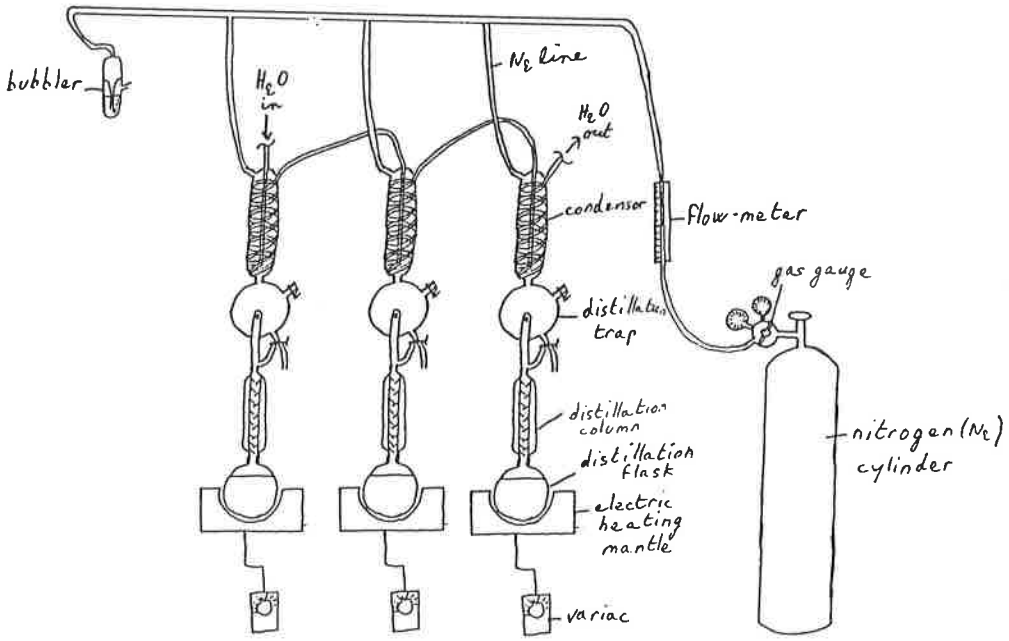
STANDARD OPERATION PROCEDURE (SOP) FOR THE DIETHYL ETHER STILL

(last updated: Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The diethyl ether is distilled under a nitrogen atmosphere from the distillation flask containing metallic sodium (Na) and benzophenone. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.404.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.259.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the diethyl ether stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- diethyl ether
- Drying agent : metallic sodium (Na)
- Benzophenone
- 2-propanol
- Absolute methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent is then poured into the round-bottom flask.

4. In a fume-hood, a small amount of metallic sodium is cut under oil into small pieces, and the sodium quickly rinsed with a *minimum* amount of hexanes to remove the oil and the sodium added to the diethyl ether distillation flask. (Note: Sodium metal and hexanes is a potential fire hazard. Ensure a dry-powder fire extinguisher is near by and accessible before you begin working with sodium. Residual solutions of hexanes containing sodium

are later *carefully* quenched with 2-propanol and several hours later *carefully* quenched with absolute methanol and finally water.)

5. Add 250 mg of benzophenone to the distillation flask and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, diethyl ether containing the sodium/benzophenone will yield a deep blue color.

6. If after several hours of reflux, the color of the solution is not dark blue, repeat steps 2 through 5.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full.
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual diethyl ether is *carefully* quenched with small amounts of 2-propanol under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual diethyl ether is then *carefully* quenched with small amounts of absolute methanol over several hours until all sodium metal is destroyed. Now add excess water (carefully) under nitrogen and dispose of the contents.

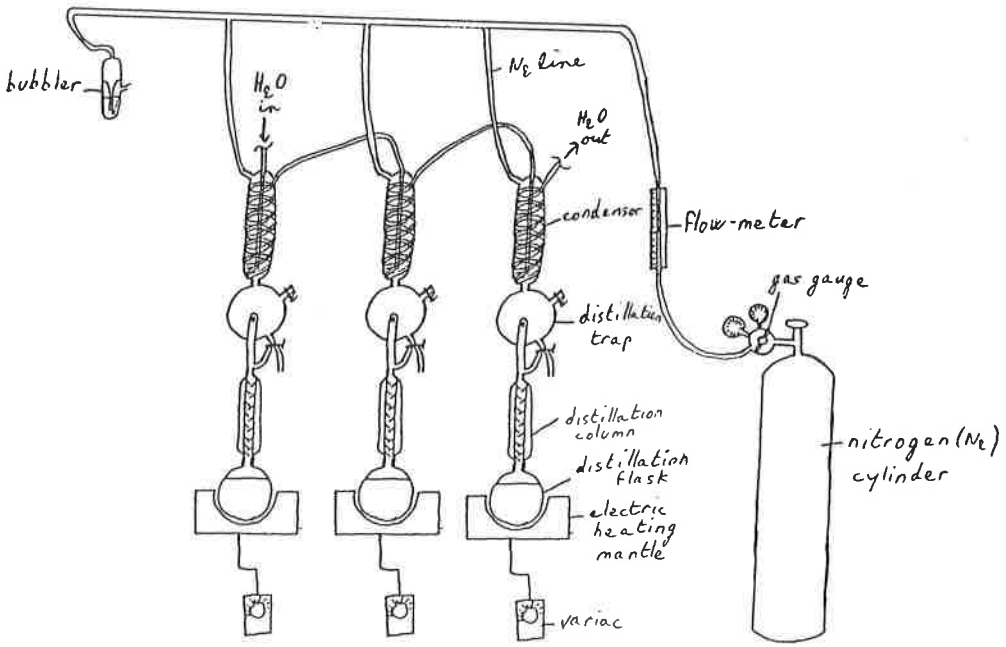
STANDARD OPERATION PROCEDURE (SOP) FOR THE DIMETHOXYETHANE (DME) STILL

(last updated : Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The DME is distilled under a nitrogen atmosphere from the distillation flask containing metallic sodium (Na) and benzophenone. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.406.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.218.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the DME stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- DME
- Drying agent : metallic sodium (Na)
- Benzophenone
- 2-propanol
- Absolute methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent is then poured into the round-bottom flask.

4. In a fume-hood, a small amount of metallic sodium is cut under oil into small pieces, and the sodium quickly rinsed with a *minimum* amount of hexanes to remove the oil and the sodium added to the DME distillation flask. (Note: Sodium metal and hexanes is a potential fire hazard. Ensure a dry-powder fire extinguisher is near by and accessible before you begin working with sodium. Residual solutions of hexanes containing sodium

are later *carefully* quenched with 2-propanol and several hours later *carefully* quenched with absolute methanol and finally water.)

5. Add 250 mg of benzophenone to the distillation flask and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, DME containing the sodium/benzophenone will yield a deep blue-green color.

6. If after several hours of reflux, the color of the solution is not dark blue-green, repeat steps 2 through 5.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full.
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual DME is *carefully* quenched with small amounts of 2-propanol under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual DME is then *carefully* quenched with small amounts of absolute methanol over several hours until all sodium metal is destroyed. Now add excess water (carefully) under nitrogen and dispose of the contents.

6.Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

Dr. T.V. RajanBabu -
John Herrington - Safety Coordinator (ph : 8-3957; pager : (9)-605-5075)
Don Tong - Hazardous Waste Specialist (ph.: 8-3957; pager : (9)-605-5076)
Dr. Malay Nandi - 3051 Evans Lab, ph: 688-3694

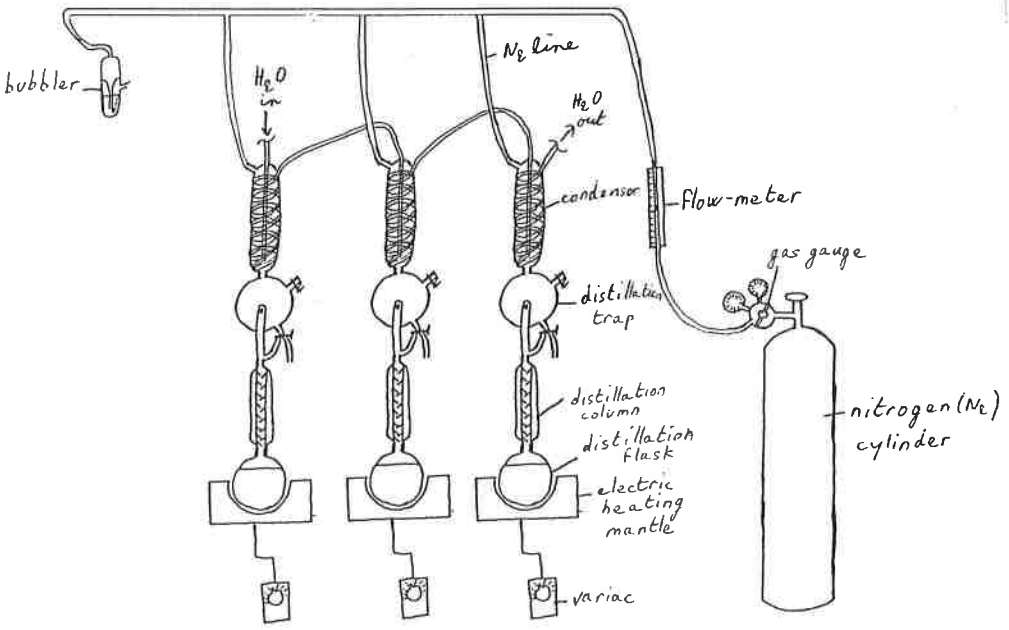
STANDARD OPERATION PROCEDURE (SOP) FOR THE BENZENE STILL

(last updated: Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The benzene is distilled under a nitrogen atmosphere from the distillation flask containing metallic sodium (Na) and benzophenone. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.398.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.118.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the benzene stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- benzene
- Drying agent : metallic sodium (Na)
- Benzophenone
- 2-propanol
- Absolute methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent is then poured into the round-bottom flask.

4. In a fume-hood, a small amount of metallic sodium is cut under oil into small pieces, and the sodium quickly rinsed with a *minimum* amount of hexanes to remove the oil and the sodium added to the diethyl ether distillation flask. (Note: Sodium metal and hexanes is a potential fire hazard. Ensure a dry-powder fire extinguisher is near by and accessible before you begin working with sodium. Residual solutions of hexanes containing sodium

are later *carefully* quenched with 2-propanol and several hours later *carefully* quenched with absolute methanol and finally water.)

5. Add 250 mg of benzophenone to the distillation flask and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, benzene containing the sodium/benzophenone will yield a deep blue color.

6. If after several hours of reflux, the color of the solution is not dark blue, repeat steps 2 through 5.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full.
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual benzene is *carefully* quenched with small amounts of 2-propanol under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual benzene is then *carefully* quenched with small amounts of absolute methanol over several hours until all sodium metal is destroyed. Now add excess water (carefully) under nitrogen and dispose of the contents.

6.Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

Dr. T.V. RajanBabu -
John Herrington - Safety Coordinator (ph : 8-3957; pager : (9)-605-5075)
Don Tong - Hazardous Waste Specialist (ph.: 8-3957; pager : (9)-605-5076)
Dr. Malay Nandi - 3051 Evans Lab, ph: 688-3694

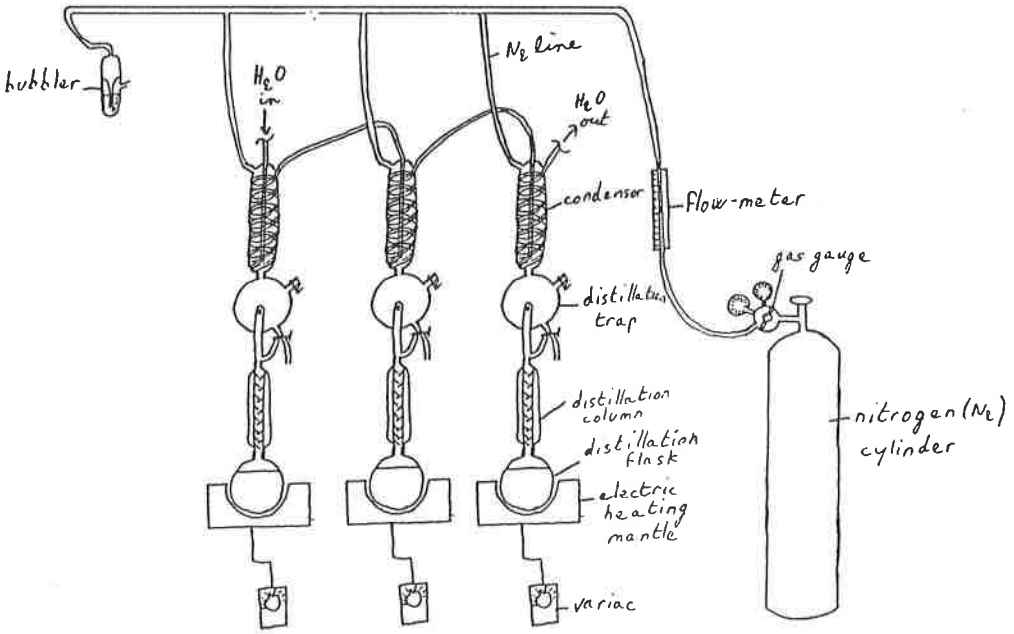
STANDARD OPERATION PROCEDURE (SOP) FOR THE TOLUENE STILL

(last updated: Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The toluene is distilled under a nitrogen atmosphere from the distillation flask containing calcium hydride (CaH_2). The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.398.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.436.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the Toluene Stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- Toluene
- Drying agent: calcium hydride (CaH_2)
- Methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required: - Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent is then poured into the round-bottom flask.

3. A small amount of calcium hydride is added and the solution is refluxed.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the pressure gauge is 100 psi or more. Change nitrogen tank when the gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual toluene is *carefully* quenched with small amounts of absolute methanol added under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual toluene is then *carefully* quenched with small amounts of water under nitrogen.

6.Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

Dr. T.V. RajanBabu -
John Herrington - Safety Coordinator (ph : 8-3957; pager : (9)-605-5075)
Don Tong - Hazardous Waste Specialist (ph.: 8-3957; pager : (9)-605-5076)
Dr. Malay Nandi - 3051 Evans Lab, ph: 688-3694

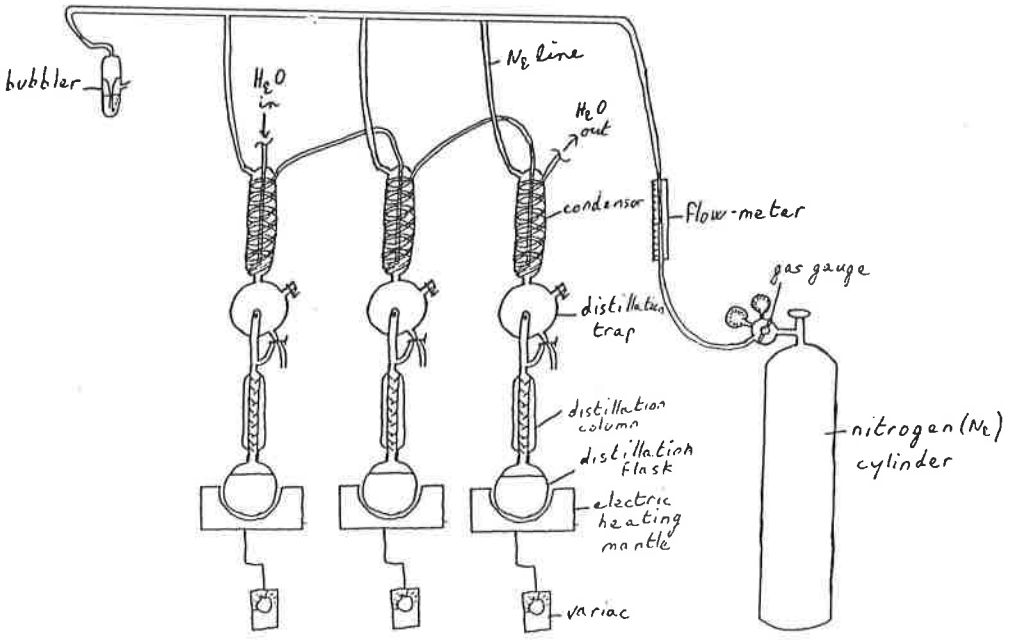
STANDARD OPERATION PROCEDURE (SOP) FOR THE DICHLOROMETHANE STILL

(last updated: Jan 1998)

Purpose. The purpose of this SOP is to provide new students and post-doctoral researchers with a detailed description and step by step instructions for the safe operation and maintenance of the solvent stills.

1.Description of the operations of the still

a) Diagram



b) Description of operation. The dichloromethane is distilled under a nitrogen atmosphere from the distillation flask containing calcium hydride (CaH_2). The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser and the distillate collects at the top of the column in the distillation trap. The purified solvent is then removed via syringe by way of the stopcock at the top of the distillation trap or the solvent tap at the bottom of the distillation trap. Each of the stills are connected in series by water lines for the condensers and nitrogen lines for inert atmosphere.

c) References. Information contained in this SOP is based upon information contained in one or more of the following references:

1. The Ohio State University Safety Handbook.
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; Longman; Singapore: 1989. p.399.
3. Perrin, D.D; Armarego, W.L.F.; Perrin, D.R.; *Purification of Laboratory Chemicals*, 2nd Ed.; Pergomon Press; Oxford: 1980. p.205.
4. *Prudent Practices for Handling Hazardous Chemicals in the Laboratories,* National Research Council, National Academy Press; Washington: 1981
5. Manufacturer's Safety Data Sheet (MSDS) provided with this SOP and can also be found via http://www.chemistry.ohio-state.edu/safety.

2.Hazards Involved in the Operation of the Dichloromethane Stills:

-Nitrogen (N_2) - High pressure hazard.

-Water - Water use in and around electrical equipment poses a shock hazard; failure to maintain cooling water to condensers poses a vapor hazard; water use in and around metallic sodium metal poses a fire/explosion hazard.

-Variable Temperature Variac and heating mantle. Electrical equipment in use around water poses a shock hazard; high temperature of heating mantle poses a burn hazard. Extreme temperatures during distillation pose a vapor hazard.

A detailed description of the safety hazards involved with the following reagents is provided in the corresponding MSDS attached as an appendix to this SOP.

- Dichloromethane
- Drying agent: calcium hydride (CaH_2)
- Methanol

3.Setting-up Solvent Stills:

Personal Safety Equipment Required:
- Laboratory glasses/goggles
- Laboratory coat
- butyl rubber protective gloves

1. The still is cooled down to room temperature (ensure variac is off).

2. The solvent is then poured into the round-bottom flask.

3. A small amount of calcium hydride is added and the solution is refluxed.

4.Periodic inspection/maintenance.

Daily: - Check gas pressure level in the nitrogen tank: ensure that the nitrogen tank pressure at the gas gauge is 100 psi or more. Change nitrogen tank when gas pressure falls below 100 psi.
- Check the nitrogen flow rate at the flow meter and the exit bubbler. Nitrogen flow rate should be about 2 on the flow meter scale.
- Check the water flow rate.
- Check the solvent level is at least 1/3 full.
- Check the solvent color.
- When done using the still and before leaving for the night, turn down still variac to 10, and empty the distillation trap.

Monthly: - Check nitrogen lines.
- Check water lines.
- Change rubber septa on distillation trap.

Quarterly: - Check electric lines and variacs.
- Check glassware for cracks.

Cleaning: as needed.

5.Regenerating/cleaning:

1. After the solvent still has cooled to room temperature, the distillation flask is moved to a fume-hood.

2. The residual dichloromethane is *carefully* quenched with small amounts of absolute methanol under a blanket of nitrogen over several hours and allowed to stand overnight.

3. The residual dichloromethane is then *carefully* quenched with small amounts of water under nitrogen.

6.Points of contact.

If you have any questions about this procedure, contact one of the following for assistance before continuing.

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