

An Overview of Lab Procedures

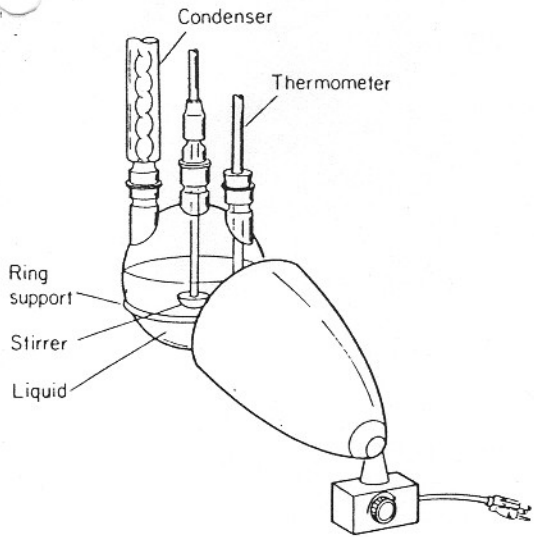


Fig. 1.3. Infrared lamp assembled from an infrared bulb (red), aluminum swivel reflector, dimmer switch, heavy-duty switch box, three-wire lamp cord and plug, and aluminum mounting rod.

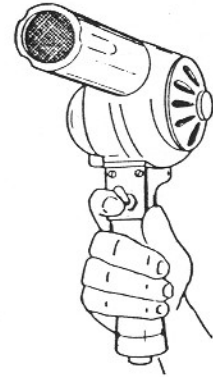
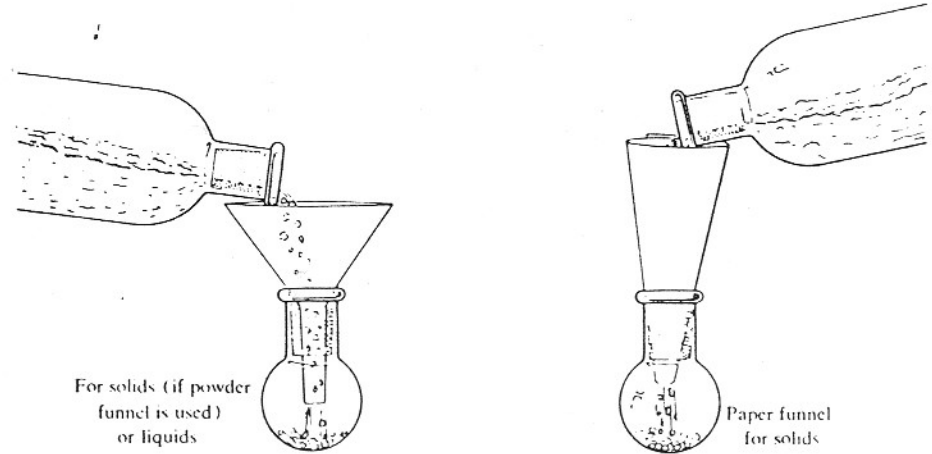
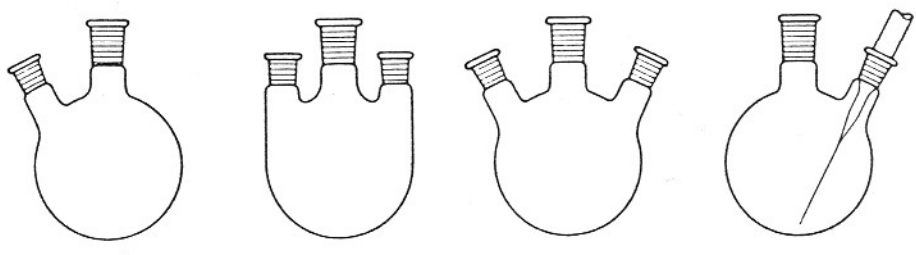
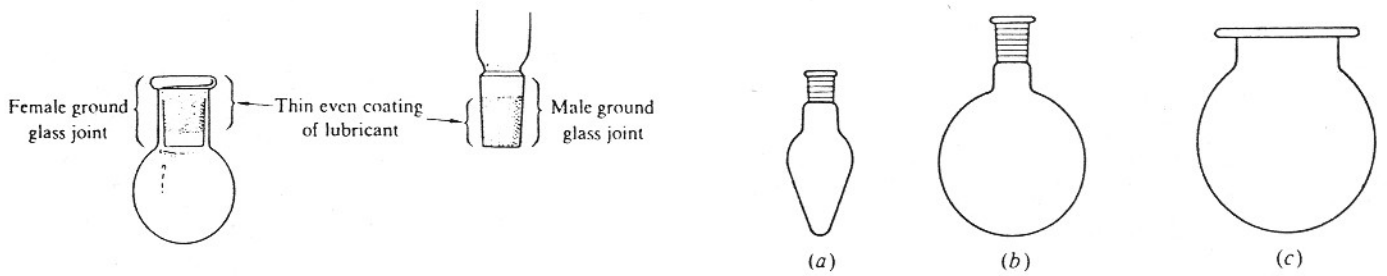
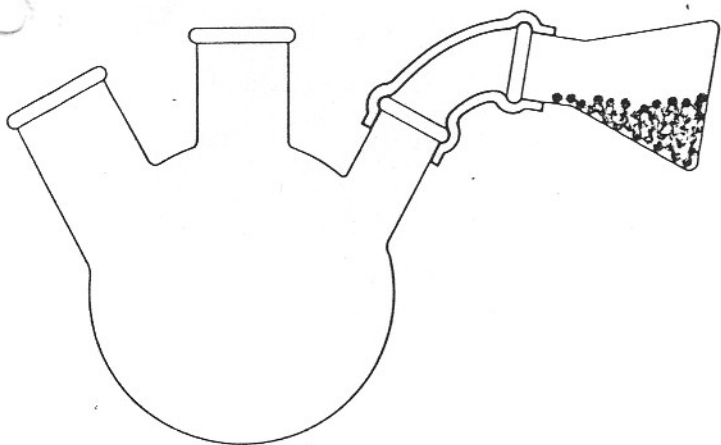


Fig. 1.6. Heat gun.



Transfer of material through a greased joint.

An Overview of Lab Procedures



Addition of a solid to a closed vessel.

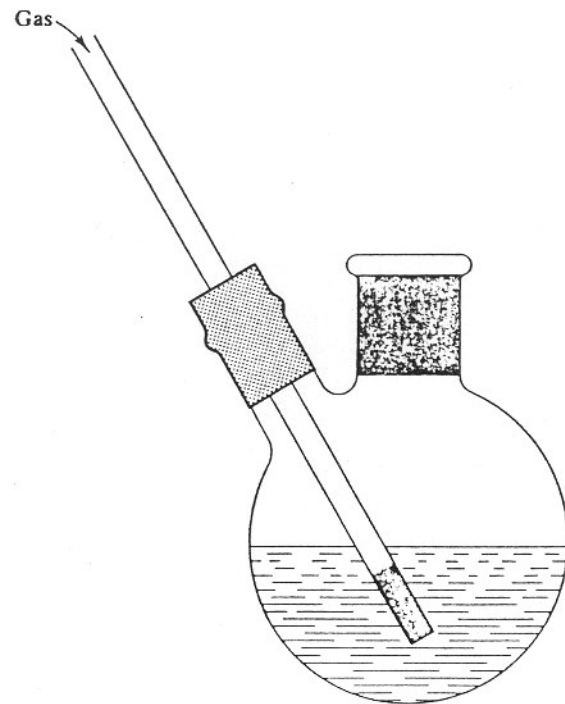
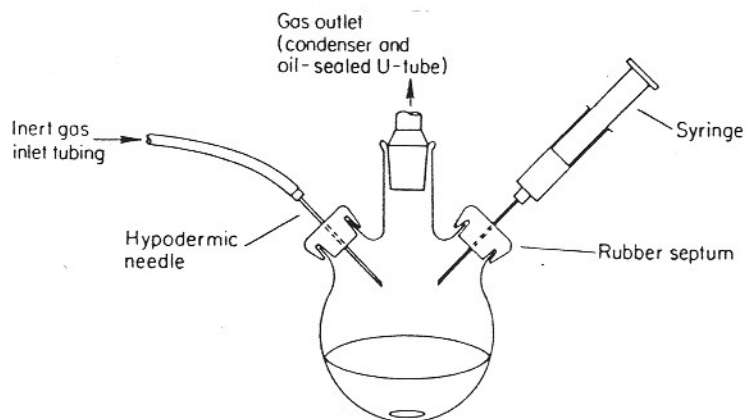
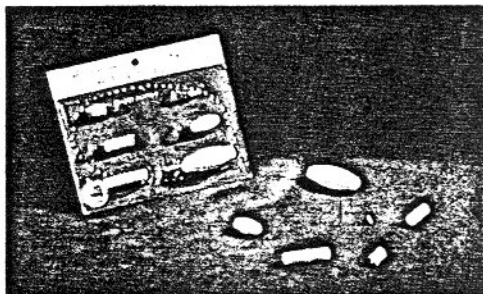


FIG. A3.5. Gas delivery tube in place.

Addition with a syringe. Rubber septums that fit ∇ (standard-taper) joints are available from Aldrich Chemical Company, Inc.



"Utility Mix" Pack

A wild assortment for every stirring purpose. Consists of 1 each:

10×3 mm



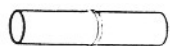
$3/8$ "



$1" \times 1/2$ "



$2" \times 3/4$ "



$1 1/2" \times 5/16$ "



$1" \times 5/16$ "

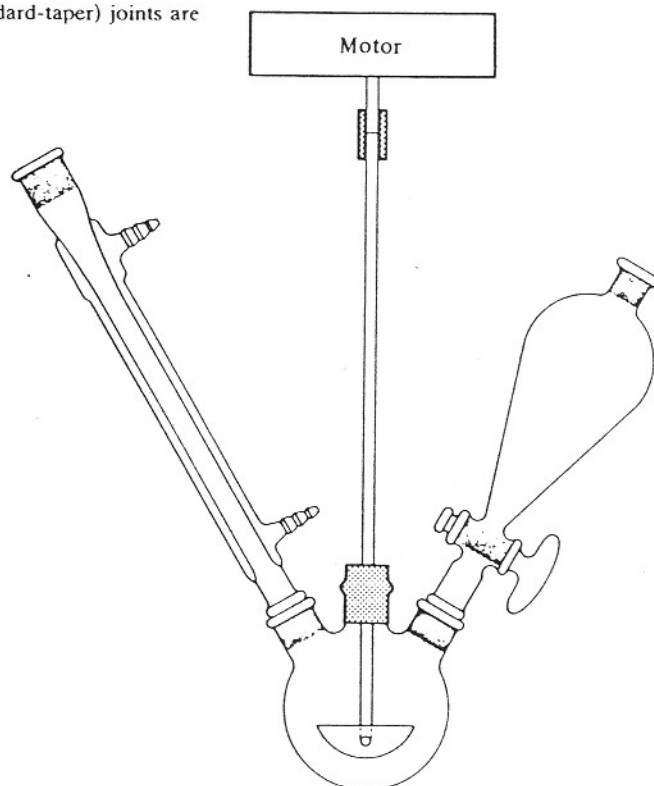


FIG. A3.1. Reaction setup employing mechanical stirring.

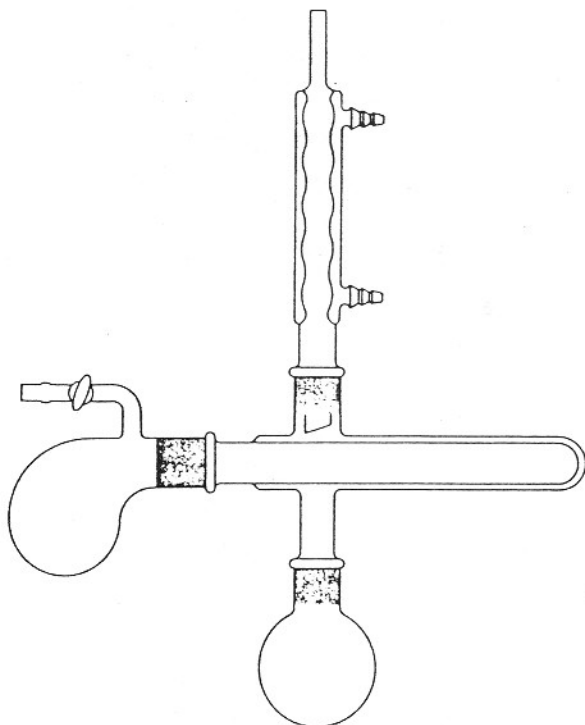


FIG. A3.18. Abderhalden (drying pistol).

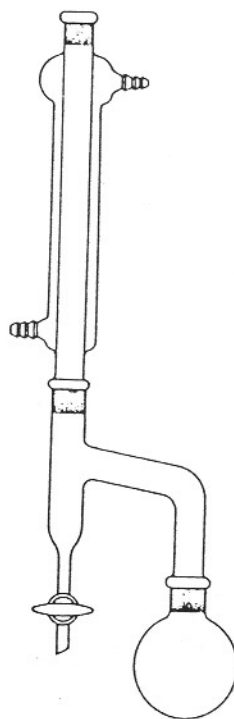
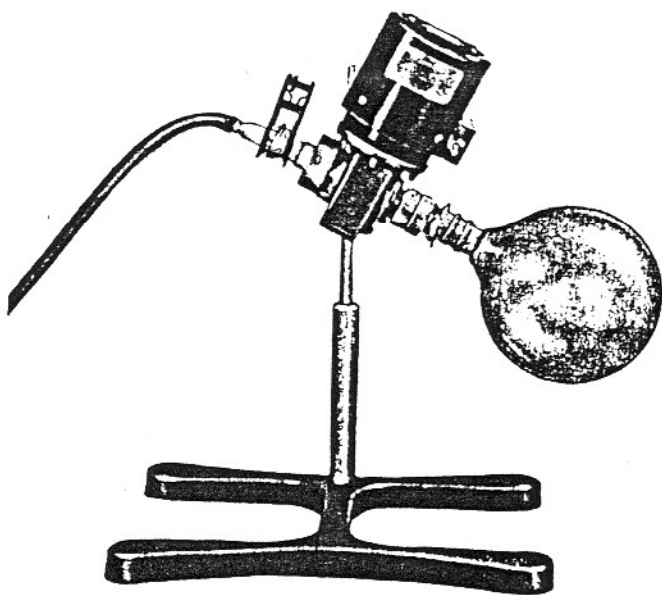
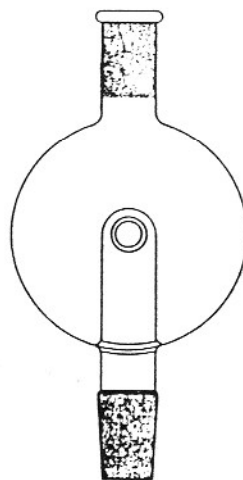


FIG. A3.6. Dean-Stark trap for continuous removal of water.



(a)



(b)

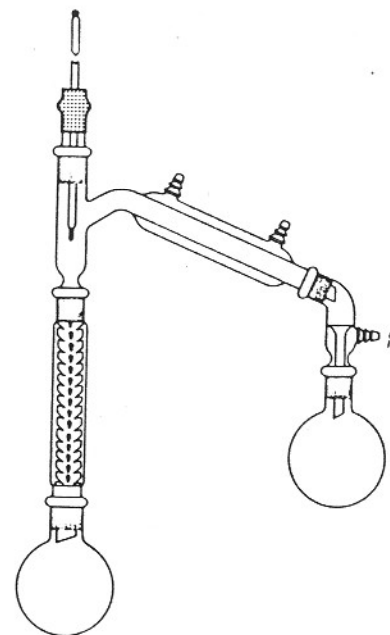


FIG. A3.12. (a) Rotary evaporator (Buchler Instruments) (b) Trap used with rotary evaporator.

SOLVENT SYSTEMS FOR THIN LAYER CHROMATOGRAPHY

The following lists contain solvent systems which have been used successfully for thin layer chromatography of the classes of compounds indicated. The numbers in parentheses after the solvents indicate the proportion (by volume) of solvent used.

Silica Gel Coating

Class of Compound	Solvent System
hydrocarbons	<ol style="list-style-type: none"> 1. pentane 2. cyclohexane 3. benzene (2) : ether (1) 4. cyclohexane (3) : ethyl acetate (1)
ketones	<ol style="list-style-type: none"> 1. cyclohexane (1) : ethyl acetate (1 to 2) 2. ether (1) : petroleum ether (2) 3. ether (1) : hexane (9)
alcohols	<ol style="list-style-type: none"> 1. cyclohexane (1) : ethyl acetate (1 to 2) 2. pentane (10) : ether (1)
esters	<ol style="list-style-type: none"> 1. cyclohexane (1) : ethyl acetate (1 to 2)
lactones	<ol style="list-style-type: none"> 1. cyclohexane (1) : ethyl acetate (1 to 2) 2. ether (1) : petroleum ether (2) 3. ethyl acetate
enolic diketones	<ol style="list-style-type: none"> 1. ethyl acetate (4) : methanol (1) 2. ethyl acetate
carboxylic acids	<ol style="list-style-type: none"> 1. chloroform saturated with 90 per cent formic acid
amines and amino alcohols	<ol style="list-style-type: none"> 1. methanol (2) : chloroform (3) containing 1 per cent by volume of 33 per cent aqueous ammonia 2. ethyl acetate (1) : methanol (1)

quaternary ammonium salts

1. methanol (6) : chloroform (6) :
conc. hydrochloric acid (1)

Phosphorus ylids	1. methanol (1) : ethyl acetate (1)
triphenylphosphine oxide	1. methanol (1) : ethyl acetate (1)
phosphonium salts	1. methanol (1) : ethyl acetate (1)
amides	1. ethyl acetate (5) : methanol (1)
Alumina Coating	
Class of Compound	Solvent System
hydrocarbons	1. pentane
ketones	1. cyclohexane (1) : ethyl acetate (1 to 2)
alcohols	1. cyclohexane (1) : ethyl acetate (1 to 2)
lactones	1. cyclohexane (1) : ethyl acetate (1 to 2)

SUMMARY OF COMMON RECRYSTALLIZATION SOLVENTS

Solvent	Formula	Boiling Point (°C)	Freezing Point (°C)	Miscibility with Water	Fire Hazard
Ether	$(C_2H_5)_2O$	34.6	-116	-	++++
Pentane	C_5H_{12}	36.2	-131.5	-	++++
Acetone	$(CH_3)_2CO$	56.1	- 95	+	+++
Chloroform	$CHCl_3$	61.3	- 63.5	-	0
Methyl alcohol	CH_3OH	64.7	- 98	+	++
Hexane	C_6H_{14}	69	- 94.3	-	++++
Carbon tetrachloride	CCl_4	76.7	- 23	-	0
Ethyl acetate	$CH_3-\overset{O}{\parallel}C-OC_2H_5$	77.2	- 84	-	++
95% ethyl alcohol	C_2H_5OH	78.1	-116	+	++
Benzene	C_6H_6	80.2	5.5	-	++++
Acetic acid	$CH_3-\overset{O}{\parallel}C-OH$	118.1	16.6	+	+
Dimethylformamide	$H-\overset{O}{\parallel}C-N(CH_3)_2$	153.0	- 61	+	+
Nitrobenzene	$C_6H_5NO_2$	210.9	5.7	-	+