

## SECTION 4: AMPHETAMINES AND DERIVATIVES

### Step 2: Preparation of DL-ephedrine

Into a suitable flask equipped with motorized stirrer, and gas inlet tube, place 15 grams of the oily organic product obtained in step 1, followed by a dilute alcohol solution prepared by mixing 25 milliliters of 95% ethyl alcohol into 120 milliliters of ice cold water, and then stir this entire mixture to dissolve the oily organic product from step 1. Thereafter, place this flask into an ice bath, and chill to 0 Celsius. When the contents of the flask reach a temperature of about 0 Celsius, add in 7 grams of a 37% formaldehyde solution. Thereafter, begin to stir these contents vigorously, and then carefully add in an acetic acid solution (prepared by adding and dissolving 6 grams of glacial acetic acid into 14 milliliters of ice cold water), and then carefully add in 6 grams of finely divide zinc (preferable zinc dust). During the addition of the acetic acid solution and zinc, vigorously stir the reaction mixture, and maintain its temperature below 5 celsius at all times. After the addition of the zinc, continue to stir the reaction mixture below 5 Celsius for about 90 minutes to complete the reaction. After 90 minutes, stop stirring, and then filter-off any insoluble zinc or other materials. Thereafter, place the filtered reaction mixture back into the ice bath, and then bubble into this filtered reaction mixture, 6 grams of hydrogen sulfide gas. Note: during the addition, vigorously stir the reaction mixture. Note: the addition of the hydrogen sulfide will precipitate any dissolved zinc. After the addition of the hydrogen sulfide, filter-off any insoluble materials, and then add to this filtered mixture, 250 milliliters of cold water, followed by 150 milliliters of diethyl ether, followed by 10 milliliters of a 35 to 38% hydrochloric acid solution. Then vigorously stir this entire mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a seperatory funnel, and remove the lower water layer (which will contain the desired DL-ephedrine product as the hydrochloride). Note: the upper ether layer can be discarded or recycled if desired. Now, to the recovered lower water layer, add in a sodium carbonate solution prepared by adding and dissolving 6 grams of anhydrous sodium carbonate into 15 milliliters of cold water, and then stir the whole mixture for about 15 minutes. Finally, extract this entire mixture with three 100-milliliter portions of diethyl ether, and after the extraction process (after each extraction, the ether will be the upper layer), combine all ether portions, if not already done so, and then dry this combined ether portion by adding in 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether is recovered, recover the left over remaining residue (after it has cooled to room temperature), and then store in an appropriate amber glass bottle until use.

Note: Other salts of the freebase DL-ephedrine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the freebase compound obtained at the end of step 2 (the freebase should be dissolved into ether in the ratio of 1 grams of freebase to 15 grams of diethyl ether). For the hydrochloride, 1 mole of acid gas should be bubbled into the ether mixture for every 1 mole of freebase DL-ephedrine (not for the total weight of the ether mixture). For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added for 2 moles of the freebase DL-ephedrine (not for the total weight of the ether mixture); and for citric acid or phosphoric acid, 1 mole of the citric acid or phosphoric acid should be added to 3 moles of the freebase DL-ephedrine (not for the total weight of the ether mixture). The ether mixture in each of these cases can be evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried. All the salts of DL-ephedrine are mild stimulants, but can be used in the preparation of stimulant compositions (by addition to amphetamines, or other CNS stimulants), or can be used in the preparation of other stimulants.

### Intermediate-0007-02. Extraction of L-ephedrine from Ma Huang herb

#### Materials:

1. 200 grams of ma huang herb	5. 225 milliliters of 10% hydrochloric acid solution
2. 450 milliliters of diethyl ether	6. 300 milliliters of 10% sodium hydroxide solution
3. 100 milliliters of 10% ammonia solution	7. 200 grams of sodium chloride
4. 110 grams of anhydrous sodium carbonate	8. 20 grams of dry hydrogen chloride gas

**Hazards:** Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves and use proper ventilation when handling ammonia water, which is very irritating—avoid prolonged inhalation of the vapors. Wear gloves when handling sodium hydroxide and hydrochloric acid, both of which are capable of causing skin irritation.

#### Process:

Personnel notes for intermediae-0007-02: L-ephedrine

Into a standard lab blender (just like a standard kitchen blender with stainless steel blade), place 200 grams of ma huang herb (obtained from a number of sources such as herbal stores, on-line sites, ect., ect.), and then add in 450 milliliters of diethyl ether. Thereafter, blend the entire mixture at slow speed (note: a quick rapid blend may be needed in order to mutilate or chop up the ma huang herb for proper blending) for about 1 hour. After 1 hour, add in 100 milliliters of a 10% ammonia solution, followed by 10 grams of anhydrous sodium carbonate, and then blend the entire mixture for about 6 hours at a slow speed setting. After blending the mixture for 6 hours, stop the blending, and then allow the entire mixture to stand overnight. The next day, slowly begin the stirring process again, and then add in three 75-milliliter portions of 10% hydrochloric solution, followed thereby with three 100-milliliter portions of 10% sodium hydroxide solution. After all additions, slowly blend the entire mixture for 1 hour. After 1 hour, add in 100 grams of anhydrous sodium carbonate, followed by 200 grams of sodium chloride to saturate the entire mixture. After the addition of the salt, slowly blend the entire mixture for about 30 minutes. After 30 minutes, stop the blending procedure, and then filter the entire mixture to remove insoluble matter, and then remove the upper ether layer from the filtered mixture by using a separatory funnel or by decantation. Once the ether layer has been collected, place it into a distillation apparatus, and gently remove the ether until only 70% of the total volume remains. Once this point is reached, stop the distillation process, and allow the remaining ether concentrate to cool to room temperature before removing it from the apparatus. Thereafter, quickly filter this ether concentrate to remove any insoluble materials, and then place this filtered ether concentrate into a clean beaker, and then place this beaker into an ice bath, and chill to 0 Celsius. Thereafter, bubble into this chilled ether mixture, 20 grams of dry hydrogen chloride gas (to the point of saturation). Afterwards, filter-off any precipitated products, and then vacuum dry or air-dry the collected precipitates. The dry solids obtained will consist of predominately L-ephedrine, with small amounts of D-pseudoephedrine, and small amounts of impurities.

Note: obviously there are numerous modifications to this process, and the person carrying out this process is encouraged to experiment and try different solvents and techniques.

### **Intermediate-0007-03. Extraction of pseudoephedrine from store bought pseudoephedrine tablets (“Sudafed” “Galpseud”, “Novafed”, “Rhinalair”, “Otrinol”, “Sinufed”, Symptom 2”, “Afrinol”, and other nasal decongestants and/or bronchodilators)**

#### **Materials:**

1. 1.5 grams worth of store bought pseudoephedrine pills	7. 5 to 10 milliliters of 99% isopropyl alcohol
2. 50 milliliters of turpentine	8. 30 milliliters of toluene or xylene
3. 50 milliliters of toluene	9. 5 grams of anhydrous sodium sulfate
4. 75 milliliters of acetone	10. 70 milliliters of dry diethyl ether
5. 2 grams of anhydrous sodium carbonate	11. 5 grams of dry hydrogen chloride gas
6. 2 grams of sodium chloride	

**Hazards:** Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Follow similar guidelines when handling acetone, which is also highly flammable. Inhalation of toluene or xylene vapors should be avoided, as toluene and xylene are listed as suspected carcinogens. Wear gloves when handling sodium hydroxide and hydrogen chloride gas, both of which are capable of causing skin irritation.

#### **Procedure:**

Personnel notes for intermediate-0007-03: Pseudoephedrine

Note: the below process (process 1A) works for all generic brands of pseudoephedrine, and the so called “time” release tablets that may contain up to 120 to 240 milligrams worth of pseudoephedrine per tablet. Many brands of pseudoephedrine, other than just the generic brands can be used with success. The following lists of ingredients are commonly found in both the generic and name brand products, and can be used with satisfactory results.