

SECTION 1: INTRODUCTION

A quick lesson in chemistry

Part 1: Introduction to chemistry

The Preparatory Manual of Narcotics Vol 1. has been written to teach the art of pharmaceutical sciences to the reader. To do this, you should take a quick, yet vital lesson in chemistry. First of all, the world of chemistry is a fascinating world filled with a huge variety of chemicals, chemical reactions, formulas, laboratory apparatus, and an arsenal of equipment. All these elements are combined and used thoroughly to bring about chemical change of matter from one form to the next. In this book, the form of change that we will deal with mostly, is the formation of compounds that possess psychological and physical action upon the body. These compounds that possess characteristic psychological effects upon the body are called narcotics.

The world of narcotics is absolutely huge, and in essence, deals with virtually millions of chemical compounds. Amphetamines and derivatives actually range in the tens of thousands, but listing them all in this book would be impossible. Regardless how many possible drugs there might be, most see drugs or narcotics as something evil or something that is a cancer soar on our societies and our civilizations; however, in factuality drugs and narcotics are as old as life itself, and have been around for millions of years longer than we humans have. Narcotics are not evil, nor are they a pain in the side of our society—they are chemical compounds like everything else that exist, not for us, but because of atoms and molecules. These atoms and molecules, and their chemistry, will be around long after we are gone, and then some. The chemistry of these substances may be millions of years old, but their chemistry, as it pertains to our benefit, is only a hundred or so years old. Even though ancient civilizations, and numerous cultures have used narcotics for thousands of years, their exact chemical make-up has only been unraveled in the last 100 years—many psychedelic amphetamines only in the last 50 years.

For most of you, the procedures in this book will not make sense at first, or will appear to be complicated; as a result, many of the procedures in this book may seem foreign, or unfamiliar—if this is the case, then at this exact moment, you are in the right place. By the time you have read this book, these “foreign” procedures will no longer be foreign to you, but in the meantime, let's get started on the world of chemistry.

The world of chemistry involves every single aspect, corner, and micro drop of everything that is matter. Our solar system and the entire universe all function on a chemical level—In essence, chemistry is everything. The universe and everything in it is composed of atoms and molecules, and within this massive space, there exists tens of millions of chemical compounds—either known or unknown. The compounds that are known make up only 5% of the naturally occurring compounds, leaving a massive 95% of them being synthetic (prepared in the lab)—95% of all narcotics are synthetic. Note: synthetic does not denote anything that is less superior to natural. Synthetic means creating natural in an un-natural way.

Chemistry has been divided into three fields over the last 100 years to better organize and format the system. The three major branches of chemistry include: Inorganic chemistry, Organic chemistry, and Biochemistry. In short, inorganic chemistry deals with ionic compounds, which make up the chemical compounds that do not contain active carbon. Organic chemistry is the largest branch of chemistry and it deals with covalent compounds, which make-up our everyday items like plastics, drugs, dyes, pesticides, insecticides, resins, fibers, and explosives. Organic means “carbon bearing” which means any compound that bears carbon is classified as organic. Gasoline, turpentine, and candle wax are specific examples of organic compounds. Last but not least, biochemistry studies the field of enzymes, organisms, plants, and animals and their active chemical processes. Genetics research studies the DNA and RNA of living things and is a sublevel of biochemistry. DNA and RNA is composed of organic compounds all linked and actively working together. Biochemistry deals heavily with peptides, amino acids, carbohydrates, etc., etc., all of which play a major role in natural process such as cells, metabolism, and the like.

1. Chemical bonding: Oxidation states

First things first, you need to understand the nature of elements, and their oxidation states (number of bonds). Every single element is capable of forming chemical bonds with other elements (with the exception of a few “noble gases”). The oxidation states are what determines how many bonds a particular element can form, and to what other elements. When elements combine, they form chemical compounds. All of the atoms within a chemical compound show specific oxidation states.

Oxidation states are not really states, but definitions of bonding, which are dictated by each individual element. Each element can form anywhere from either 0 to 7 bonds. These numbers represent the number of bonds the element can form (look at a modern periodic table, such that included in the “Merck Index”—the oxidation states are written in the upper left corner of each element). These numbers clearly indicate the number of bonds each element is capable of forming.

As most people are aware, periodic tables include rows and columns filled with elements. The elements within any given column have similar properties and characteristics along with similar oxidation states. For example, the elements of column 5A on the periodic table include nitrogen, phosphorus, arsenic, antimony, and bismuth. All these elements have similar oxidation

Red phosphorus

P₄

Red phosphorus forms red to reddish-violet powder or granules. The crystals sublime when heated to 416 Celsius, and are insoluble in water, and most organic solvents. Red phosphorus is soluble in phosphorus tribromide. Red phosphorus is capable of converting to white phosphorus when distilled at 290 Celsius, and will ignite in air when heated to 260 Celsius. Red phosphorus can be obtained from matches, by scraping-off the match chemicals, treating these scraped-off chemicals with hot water, filtering, then treating the filtered-off solids with 99% isopropyl alcohol, allowing this alcohol mixture to soak overnight, followed by filtering-off the insoluble materials, then treating these filtered-off materials with hexane, allowing this hexane mixture to soak overnight, and then finally filtering off the red phosphorus, and then allowing it to dry. Obviously it would take a large number of matches in order to achieve a significant amount of red phosphorus, and this phosphorus may be contaminated with small amounts of glass; however, this glass does not impede in the use of the red phosphorus in anyway. Red phosphorus can be purchased from a variety of locations, so check around.

Sodium (metallic)

Na

Sodium metal forms shiny to white to lustrous granules, sticks, or cubes with a melting point of 98 Celsius. Metallic sodium readily tarnishes on exposure to air, and may react violently with moisture, so it should be stored under kerosene. Metallic sodium reacts violently and explosively with water, so avoid contact with water at all cost. Sodium can be made by electrolyzing molten sodium hydroxide using a lead anode and stainless steel cathode.

Sodium azide

NaN₃

Sodium azide forms colorless hexagonal crystals, which decompose on heating into metallic sodium and nitrogen. This decomposition often occurs violently. Sodium azide is very soluble in water, and water solutions are rapidly converted to hydrazoic acid. Sodium azide solutions should not be stored for prolonged periods of time. Sodium azide is insoluble in ether, and liquid ammonia. It is a poisonous solid, and ingestion of 600 to 800 milligrams may be fatal. Keep sodium azide in tightly sealed bottles, and store in a cool dry place away from light. It can be prepared by reacting heated nitrous oxide (60 to 90 Celsius) with sodium amide heated at 60 to 100 Celsius, and then recrystallizing the sodium azide from water. Sodium azide is commercially available, but expensive.

Sodium bicarbonate

NaHCO₃

Sodium bicarbonate, also called baking soda, forms a white powder. The powder begins to lose carbon dioxide when heated to 50 Celsius, and it is converted into sodium carbonate when heated to above 100 Celsius. It is readily soluble in acid, with neutralization, but it is insoluble in alcohol and most organic solvents. A saturated sodium bicarbonate solution in water contains about 9% sodium bicarbonate by weight. The compound is obtained by bubbling carbon dioxide gas into a concentrated sodium hydroxide solution, followed by filtering-off the sodium bicarbonate. It is made on an industrial scale by bubbling carbon dioxide, or by adding dry ice to a solution of sodium chloride in ammonia.

5% Sodium bicarbonate solution: Prepare by adding and dissolving 50 grams of sodium bicarbonate into 950 milliliters of cold water.

Sodium bisulfite

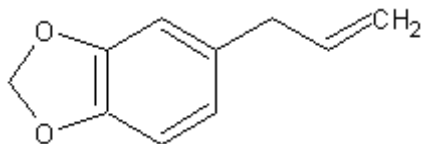
NaHSO₃

or

Na₂S₂O₅

Sodium bisulfite forms a white crystalline powder with a slight odor of sulfur dioxide. The crystalline material is slowly oxidized to sodium sulfate on standing, so bottles should be kept tightly closed and stored in cool places. It is soluble in water to the extent of 1 gram in 3.5 milliliters of water, and it is only very slightly soluble in alcohol. Aqueous solutions are acidic.

Intermediate-0010. Safrole. 5-allyl-1,3-benzodioxole



Safrole forms a colorless to slightly yellow liquid with the odor of sassafras. The oil is insoluble in water, but very soluble in alcohol, and miscible with chloroform and ether. The oil has a boiling point of 232 Celsius, but can be distilled under high vacuum at 100 Celsius under 11 millimeters of mercury. Safrole is the main component of sassafras oil, from which it makes up 70 to 75% of the oil by weight. Safrole also exists in *Ocotea cymbarum* oil (Brazilian oil of sassafras), which it exists up to 90% by weight. The oil of massoria bark and *Cinnamomum massoia* contains about 14% safrole. Safrole can be extracted from sassafras oil by the means described later, and it can be extracted from Massoria bark oil and *Cinnamomum massoia* by washing the corresponding oil with sodium hydroxide solution to remove the phenols, and then vacuum distilling to obtain the safrole boiling at about 100 Celsius under a vacuum of 11 millimeters of mercury, or by carefully fractionally distilling (two path distillation) the phenol free oil at 228 to 235 Celsius. Safrole can also be made synthetically from rather inexpensive reagents (see procedure B below). Sassafras oil can be obtained by steam distilling the ground up roots of the sassafras tree, which grows in the mid western United States. Other sassafras species of trees elsewhere in the world can also be used to obtain the safrole by steam distillation from the root. To identify a sassafras tree, consult a book that discusses the various types of trees and plants. The dried root bark of the sassafras tree contains about 10% safrole by weight, and the remainder of the root contains only about 1%. The dried root bark can be obtained from numerous sources, including herb stores, health food suppliers, and botanical suppliers. Sassafras oil can also be obtained from these aforementioned sources; if however your local suppliers do not offer the sale of sassafras oil, request them to order some for you, which they should have no problem doing. Safrole is also used in perfumes, so check out the types of perfumes, and their ingredients. Note: check out your local aromatherapy suppliers, as they are major consumers of oils, one of which may be sassafras oil. Sassafras oil may be used in adulterants in massage oils for use in aromatherapy. *Ocotea cymbarum* oil is obtained by steam distillation of the wood of the *Ocotea pretiosa* tree, which grows in South America. The wood contains about 1% oil by weight, which is easily collected by steam distillation of the wood chips, and the resulting steam distilled product contains about 90% safrole by weight. Distributors of perfume and flavoring compounds may contain this *Ocotea cymbarum* oil. Check the OPD directory for essential oils and botanical companies; also check out small herb shops nationwide.

Procedure A: Extraction of safrole from sassafras oil

Procedure:

Personnel notes for procedure A: Safrole

Note: as previously mentioned, sassafras oil can be obtained from the root bark of the sassafras tree. To do this, setup a standard steam distillation apparatus, and then steam distill the root bark (grind the root bark into pieces before use). The oil and water collect in the receiver flask, where upon the oil can be seen as droplets. The oil is denser than water so it will form droplets below the water. After the steam distillation process, the oil can be collected by placing the water/oil mixture into a separatory funnel, and then recovering the lower oil layer. The collected oil layer should then be dried by mixing with it, a small amount of anhydrous calcium chloride. After filtering-off the calcium chloride, place the oil into a vacuum distillation apparatus, and vacuum distill at 100 Celsius under a vacuum of 11 millimeters of mercury (see figure below). Note: other oils containing safrole can be vacuum distilled in a similar manner. Note: because of the expense involved in purchasing vacuum distillation apparatus, try freezing the sassafras oil, or other oils that contain the safrole. Safrole has a melting point of 11 Celsius, and it may be possible to crystallize the safrole out of any oil solution by using ice baths, cold-water baths, or even a freezer. Experiment with various techniques; solvent extractions may also work.

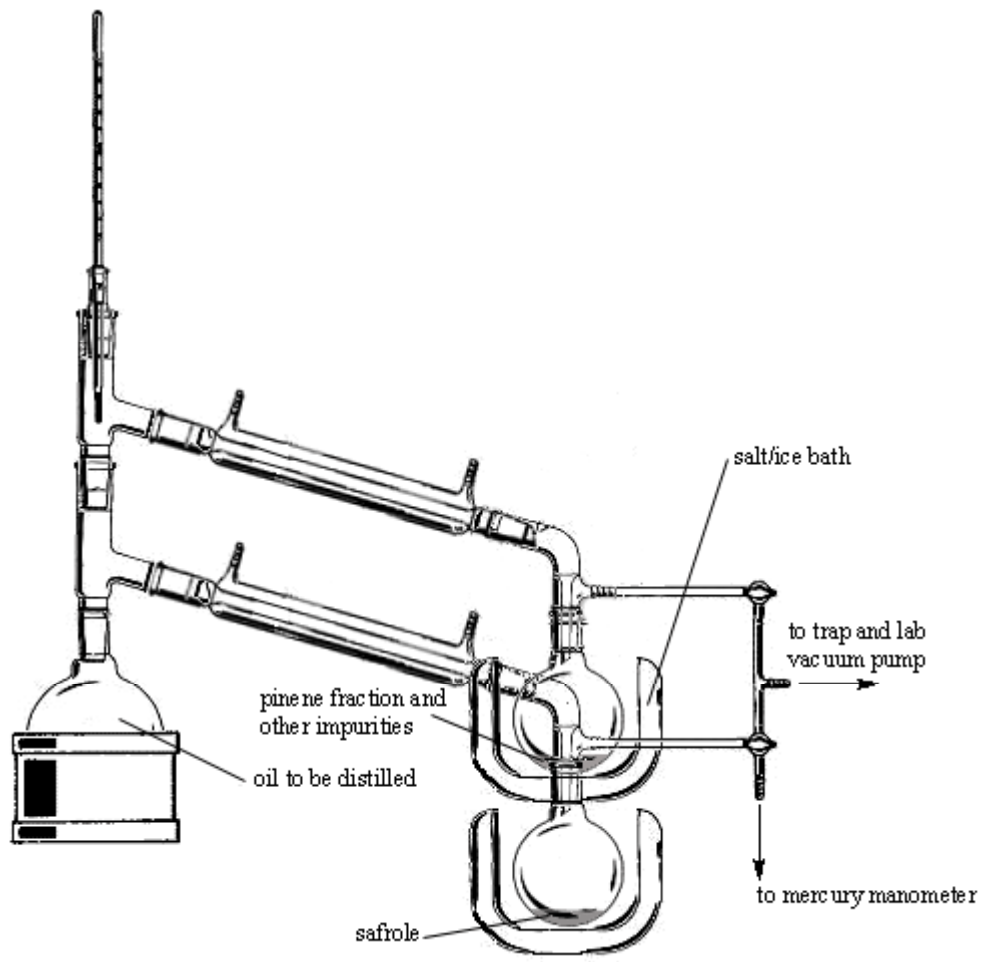
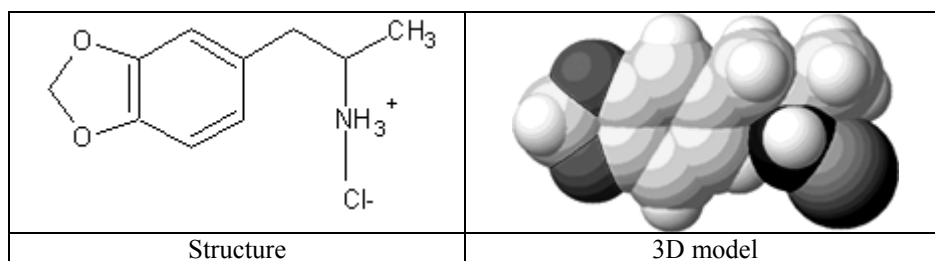


Figure 042. Two-path vacuum distillation apparatus for collecting safrole Note: in some cases, the safrole may be the upper fraction, depending on density, and impurities.

0012. MDA hydrochloride. *1-(1,3-benzodioxol-5-yl)propan-2-amine hydrochloride*



MDA hydrochloride is a psychedelic drug with stimulation properties, however its stimulation properties are rather muffled by its strong intoxicating effects. The drug produces mild hallucinations upon administration, with secondary effects resulting in bursts of energy, and/or feelings of well-being. MDA hydrochloride produces an interesting high when taken by users, and it has been explained by some to produce a “pleasant trip”.

Note: This substance is a controlled substance (hallucinogen/stimulant) as listed in the US code of Federal regulations.

Toxicity: Low	Rate of onset (average): Rapid
Stimulation dosage (ingestion): 85 to 160 milligrams	Duration of effects (average): 8 to 12 hours (depending on the person)
Stimulation dosage (inhalation): 50 to 100 milligrams	Habit forming potential: Moderate
Stimulation dosage (injection): 30 milligrams +	Estimated value U.S. (based on procedure): \$24 per gram

Procedure A: Preparation of MDA hydrochloride

Materials:

1. 90 grams of 47% hydrobromic acid	9. 20 milliliters of diethyl ether
2. 16 grams of safrole (see Intermediate-0010. Safrole)	10. 50 grams of calcium bicarbonate
3. 21 grams of dry hydrogen bromide gas	11. 20 grams of hexamine
4. 450 milliliters of diethyl ether	12. 31.5 grams of sodium iodide
5. 25 grams of anhydrous potassium carbonate	13. 40 grams of dry hydrogen chloride gas
6. or 40 milliliters of glacial acetic acid	14. 10 grams of sodium hydroxide
7. or 40 milliliters of 47% hydrobromic acid	15. 15 grams of anhydrous magnesium sulfate
8. or 20 grams of safrole (see Intermediate-0010. Safrole)	

Summary: MDA is prepared in a two-step process starting with the formation of bromosafrole. Bromosafrole can be prepared in several ways, but generally includes the bromination of the safrole with hydrobromic acid. The resulting bromosafrole is then obtained by solvent extraction, followed by removal of the solvent. The resulting bromosafrole is then converted into MDA by reaction with hexamethylenetetramine (hexamine) in the presence of sodium iodide and 95% ethyl alcohol. The reaction takes about 30 days, which is an astronomical amount, but little has to be done during this time period, as the reaction simply sits for the necessary amount of time. After 30 days, the reaction mixture is acidified with hydrogen chloride, and then treated with sodium hydroxide to liberate the freebase MDA. The freebase MDA is then extracted into ether, and the resulting ether mixture is then treated with hydrogen chloride gas to precipitate the MDA hydrochloride as the desired product.

Hazards: Use great care when handling concentrated hydrobromic acid, which is very irritating to the nose, and throat. Use proper ventilation when using hydrogen bromide, and hydrogen chloride gas. Extinguish all flames before using diethyl ether, which is highly flammable and can form explosive mixtures with air. Wear gloves when handling glacial acetic acid, and sodium hydroxide, as they both can cause skin irritation.

Procedure:

Personnel notes for procedure A: MDA hydrochloride

Step 1: Bromination of safrole (method 1)

Into a suitable flask equipped with motorized stirrer, thermometer, and addition funnel, place 90 grams of 47% hydrobromic acid, and then place 16 grams of safrole into the addition funnel. Thereafter, place the flask into an ice/salt bath, and chill to -5 Celsius. When the hydrobromic acid reaches a temperature of -5 Celsius, slowly add the safrole to the hydrobromic acid over a period sufficient to keep the reaction temperature below 0 Celsius at all times. During the addition, moderately stir the reaction mixture. Note: do not allow the reaction mixture to get above 0 Celsius, as unwanted side reactions will result decreasing the yield of desired product. After the addition of the safrole to the hydrobromic acid, replace the addition funnel with a gas inlet tube (make sure you leave a vent open to the atmosphere), and then bubble into the reaction mixture 21 grams of dry hydrogen bromide gas. During the addition of the hydrogen bromide gas, stir the reaction mixture, and maintain the temperature below 0 Celsius at all times. After the addition of the hydrogen bromide gas, continue to stir the reaction mixture at a temperature below 0 Celsius for 24 hours. Note: it may be possible to store the reaction mixture in a freezer at -5 Celsius instead of maintaining a pesky ice/salt bath, which has to be continuously replaced. If using a freezer, attach a sodium carbonate trap to the reaction flask, to capture any possible escaping acidic vapors. After allowing the reaction mixture to sit for 24 hours, remove the ice/salt bath, or take the reaction apparatus out of the freezer, and then pour the entire reaction mixture over 250

Procedure B: Preparation of MDMA from piperonylacetone via amalgated aluminum reduction

Materials:

1. 40 grams of sodium hydroxide	8. 68 milliliters of a 25% sodium hydroxide solution
2. 20 grams of aluminum foil	9. 24.7 grams of piperonylacetone (see Intermediate-0011. Piperonylacetone)
3. 100 milliliters of 95% ethyl alcohol or 100 milliliters of denatured alcohol	10. 100 milliliters of methanol
4. 500 milligrams of mercury-II-chloride	11. 280 milliliters of 10% hydrochloric acid
5. 366 milliliters of diethyl ether	12. 495 milliliters of methylene chloride
6. 28 grams of methylamine hydrochloride	13. 15 grams of anhydrous magnesium sulfate
7. 247 milliliters of 99% isopropyl alcohol	14. 20 grams of hydrogen chloride gas

Summary: MDMA can be prepared by reacting piperonylacetone with amalgated aluminum in the presence of methylamine hydrochloride. The reaction is identical to that in procedure D for the preparation of MDA hydrochloride, and afterwards the reaction mixture is filtered, evaporated to remove solvents and water, and then extracted into hydrochloric acid, from where it forms the water-soluble hydrochloride. The hydrochloride is then purified by extraction of the freebase oil into methylene chloride by addition of sodium hydroxide, which liberates this freebase oil. The resulting methylene chloride mixture is then evaporated to remove the methylene chloride, and the left over oil is then dissolved into ether, whereby it is finally precipitated as the purified MDMA.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Wear gloves when handling mercury chloride, and any mercury containing solutions, or mixtures, as they can be absorbed into the skin. Ethyl alcohol, methanol, and isopropyl alcohol are flammable, and methanol burns with a colorless flame, so use caution. Sodium hydroxide, hydrochloric acid, and dry hydrogen chloride gas are corrosive, and capable of causing skin burns. Avoid inhalation of hydrogen chloride gas.

Procedure:

Personnel notes for procedure B: MDMA

Step 1: Amalgamation of aluminum

Into a suitable beaker, place 90 milliliters of distilled water, followed by 10 grams of sodium hydroxide. Thereafter stir the mixture to dissolve the sodium hydroxide. Note: much heat is generated when sodium hydroxide is dissolved in water, so allow the sodium hydroxide solution to cool to room temperature before using. Thereafter, add in 20 grams of aluminum foil pieces (cut into small squares), and allow the aluminum foil pieces to stand in the sodium hydroxide solution for about 20 minutes or until the evolution of hydrogen gas has drastically decreased. When the hydrogen gas evolution has almost ceased, filter-off the remaining pieces of aluminum, and then wash these collected pieces of aluminum with three 50-milliliter portions of distilled water, followed by one portion of 50 milliliters of 95% ethyl alcohol (denatured alcohol can be used if desired). After the washing portion, allow the aluminum pieces to air-dry. When the pieces have air-dried, prepare a solution by adding and dissolving 500 milligrams of mercury-II-chloride (mercuric chloride) into 25 milliliters of water. Thereafter, add to the mercury chloride solution, the air-dried aluminum pieces, and allow the mixture to stand for about 15 minutes. After 15 minutes, filter-off the insoluble amalgated aluminum pieces, and then wash these filtered-off pieces with two 50-milliliter portions of distilled water, followed by one 50-milliliter portion of 95% ethyl alcohol (denatured alcohol will work if desired), and then wash with one portion of 10 milliliters of diethyl ether. After the washings, store the amalgated aluminum pieces submerged in a small amount of diethyl ether until use.

Step 2: Preparation of MDMA

Into a suitable flask or beaker, add in the amalgated aluminum prepared in step 1, followed by 28 grams of methylamine hydrochloride dissolved in water (prepared by adding and dissolving the methylamine hydrochloride into 30 milliliters of water), followed by 84 milliliters of 99% isopropyl alcohol, followed by 68 milliliters of a 25% sodium hydroxide solution, followed by 24.7 grams of piperonylacetone, and then followed by 163 milliliters of 99% isopropyl alcohol. Thereafter, moderately stir the reaction mixture for about 60 minutes. Note: during the reaction, keep the reaction mixtures temperature below 58 Celsius—a ice bath or cold water bath may or may not be needed, but most likely will be needed, so place the flask

or beaker into a ice bath or ice water bath prior to adding the ingredients. After stirring the reaction mixture for 60 minutes, filter the reaction mixture to remove insoluble materials. Note: instead of filtering using the normal methods, pour a layer of celite (diatomaceous silicate powder) over the filter paper before filtering the reaction mixture after the initial 60-minute period. After filtering, pass two 50-milliliter portions of methanol through the filter (containing the celite), and then combine these two methanol portions to the filtered reaction mixture, and then place the entire reaction mixture into a distillation apparatus, and distill at 100 Celsius to remove the methanol, isopropyl alcohol, and water. When no more methanol, isopropyl alcohol, or water passes over or is collected, stop the distillation process, and allow the left over remaining oily residue to cool to room temperature before collecting it. Thereafter, dissolve the recovered oily residue into 56 milliliters of diethyl ether, and then extract this ether mixture with two 140-milliliter portions of 10% hydrochloric acid. Note: after the extraction process, the hydrochloric acid mixture will be the lower layer each time. After the extraction process, briefly extract this hydrochloric acid mixture with three 25-milliliter portions of methylene chloride (to remove impurities), and then discard or recycle the methylene chloride portions. Note: after each extraction, the methylene chloride portion will be the lower layer each time. After the extraction process, basify the hydrochloric acid mixture by adding to it, a sodium hydroxide solution prepared by adding and dissolving 30 grams of sodium hydroxide into 150 milliliters of water. Note: sodium hydroxide generates much heat when dissolved in water, so allow the solution to cool to room temperature before using. After adding the sodium hydroxide solution, moderately stir the alkaline mixture for about 30 minutes at room temperature. Finally, extract this alkaline mixture with three 140-milliliter portions of methylene chloride, and after the extraction process, combine all methylene chloride portions (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this methylene chloride mixture into a distillation apparatus, and remove it at 40 Celsius. When no more methylene chloride passes over, recover the left over remaining oily residue (after it has cooled), and then dissolve it into 300 milliliters of diethyl ether. Thereafter, place this ether mixture into an ice bath, and chill to 0 Celsius. Then bubble into this chilled ether mixture, 20 grams (excess) of hydrogen chloride gas, and after the addition of the hydrogen chloride, stir the entire mixture for about 30 minutes at 0 Celsius. Then filter-off the precipitated MDMA product, and then vacuum dry or air-dry the crystals.

Note: Other salts of ecstasy (besides the hydrochloride) such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the ether mixture of the extracted freebase compound obtained at the end of step 2. For sulfuric acid or tartaric acid, 1 mole of 98% sulfuric acid or d-tartaric acid should be added to 2 moles of the ether mixture of the extracted freebase. For citric acid or phosphoric acid, 1 mole of the acid should be added to 3 moles of the ether mixture of the extracted freebase. The ether mixture after treatment with the corresponding acid in each of these cases can then be filtered to recover the precipitated crystals of the desired product. All the salts of ecstasy (other than the hydrochloride) are hallucinogens/stimulants and are psychedelic in nature. The tartrate and citrate salts may be twice as potent as the original hydrochloride (ecstasy).