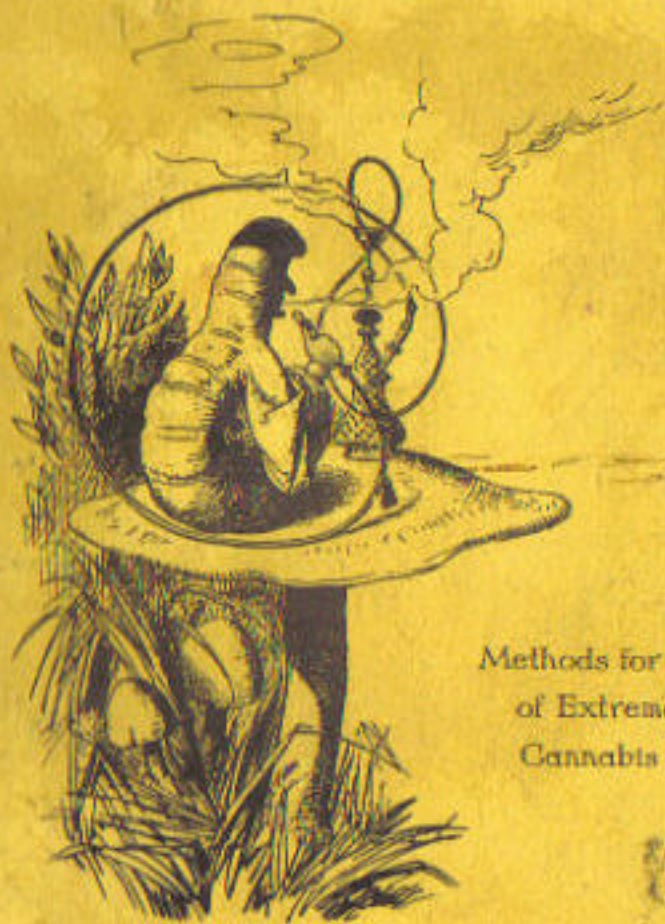


extracts of

# CANNABIS ALCHEMY:

## The Art of Modern Hashmaking



Methods for Preparation  
of Extremely Potent  
Cannabis Products

PRODUCED BY  
**The Twentieth Century Alchemist**

## TABLE OF CONTENTS

### **A SAFE METHOD FOR EXTRACTION AND PURIFICATION OF THE OILS OF MARIJUANA AND HASHISH** 9

*Step-by-step instructions for safely and efficiently removing the THC-containing oils of marijuana and hashish using components available at any hardware store.*

### **CONVERSION OF CANNABIDIOL, A NON-PSYCHOACTIVE COMPOUND IN THE OIL, TO TETRAHYDROCANNABINOL, RESULTING IN GREATLY INCREASED POTENCY. CONVERSION OF LOW-ROTATING THC TO THE HIGHER- ROTATING ISOMERIC FORM** 16

*Converting an inert element found in hashish and marijuana to pure THC, resulting in an increase in potency up to six times. Conversion of the low-rotating forms of THC as found in low-quality marijuana and hashish to the more psychedelic and spiritual high-rotating forms.*

### **CONVERSION OF THC TO THC ACETATE** 17

*Resulting in a total increase in the potency of the hashish, marijuana, or oil of up to twelve times.*

### **PREPARATION OF HASHISH FROM THE INTENSIFIED CANNABIS OIL** 24

*Manufacture of fine hashish from raw cannabis material.*

### **REMOVING THE OIL FROM INTACT MARIJUANA FLOWERS, INCREASING ITS POTENCY, AND REPLACING THE INTENSI- FIED OIL BACK ON THE ORIGINAL INTACT FLOWERS** 24

*Methods for increasing the strength of intact marijuana flowers up to twelve times at no increase in cost or change in taste or appearance.*

### **PREPARATION OF OIL CAPSULES FOR ORAL CONSUMPTION** 25

*Preparing cannabis for the most efficient means of internal consumption.*

### **PREPARATION OF TRANSLUCENT (HONEY) OIL** 25

*Removal of colored impurities to obtain this popular preparation.*

### **PREPARATION OF "REEFERS"** 26

*Methods of impregnating cigarettes or joints with cannabis oil.*

### **HIGH-VOLUME EXTRACTION METHOD** 26

*Construction and operation of apparatus with which one may process up to one hundred pounds of cannabis material daily.*

### **ADVANCED REFINEMENT TECHNIQUES** 31

*Preparation of various stages of red oil and a method for isolation of pure organic THC from cannabis.*

The cultivation of marijuana and the refinement of its preparations has concerned alchemists and hedonists on this planet for centuries. *Cannabis sativa* and *Cannabis indica* are both powerful allies. The body of the plant itself serves as a link between the physical plane and a host of Spirits of exceptional wisdom and subtlety. When the plant is ingested these qualities manifest in the mind of the worshipper, unlocking the storehouse of Wisdom within and revealing the hidden springs of pleasure. Smoking or eating the leaves or flowers is usually sufficient to bring about the desired state, although it seems inherent in the nature of Man to search for more concentrated forms of the drug which are stronger, more pleasant to ingest, or are more desirable in some other way. Thus it is that in every culture the technology of that period is applied to the work of the transmutation. As technology has evolved, so have the outward trappings of the operation, even though the principles underlying the operation remain constant throughout time and cultural differences. In primitive situations the refinement is carried on manually, the flowers being separated from the less psychoactive seeds, stems and leaves. Resins are extracted by simply rubbing the plant with the hands and then scraping the resin from the hands with the fingers. Water extractions are accomplished by boiling the plant parts in water, letting the water evaporate in the sun, and then collecting the residue. In cultures advanced to the state of mechanical technology, devices are used to this end. This might involve sifting the dried resin through mesh cloth, or mechanically pressing the resin into slabs. In cultures where the ingestion of the plant is accepted and desirable, these techniques become the formulae of power, and hashmakers are revered as Priests of the Holy Sacrament. In other situations their work is misunderstood, and they are branded as criminals to be persecuted. Yet their work continues.



## **A SAFE METHOD FOR EXTRACTION AND PURIFICATION OF THE OILS OF MARIJUANA AND HASHISH**

### **Step 1: Preparing the marijuana or hashish.**

If marijuana is to be used as a starting material the seeds must be removed prior to extraction. The remaining material is then crumbled or broken and the stems cut short with scissors. The marijuana should be thoroughly dried. Preheat an oven to 250°F. and then turn it off. Place the marijuana on a cookie sheet for fifteen-minute intervals until the loose leaf and flower parts may be easily crumbled to powder between the palms. Be careful not to scorch the plant.

Hashish may be heated for several minutes in an oven or in a frying pan at low heat until it begins to smoke slightly. It is then easily crumbled in the hands, or, if a mortar and pestle is available, it may be ground to a fine powder. Do not let the powdered hashish sit exposed to the air for long periods as this will decrease the potency.

### **Step 2: Pulverizing the cannabis material.**

There are several reasons for reducing the material to the finest powder possible: Ruptured cell walls allow the oil to be extracted more readily and the volume of the starting material is reduced, thus lessening the size of the extraction apparatus needed as well as the amount of solvent necessary.

Marijuana is put into a heavy-duty blender filled one-third full. A slower speed allows the ground material to fall into the blades while constantly flowing up the sides from the bottom. If necessary, the material can be agitated with a wooden stick while the blender is not running. It is dangerous to stir while the blender is operating as the stick can be shot from the blender with great force. It is easier to grind the chopped stems separately and then mix the powdered material before proceeding to the next step.

Hashish may also be ground in a blender, but small amounts should be run as larger amounts will put a strain on the motor. Pressed forms of hashish may be shredded with a cheese grater prior to blending.

### **Step 3: Refluxing**

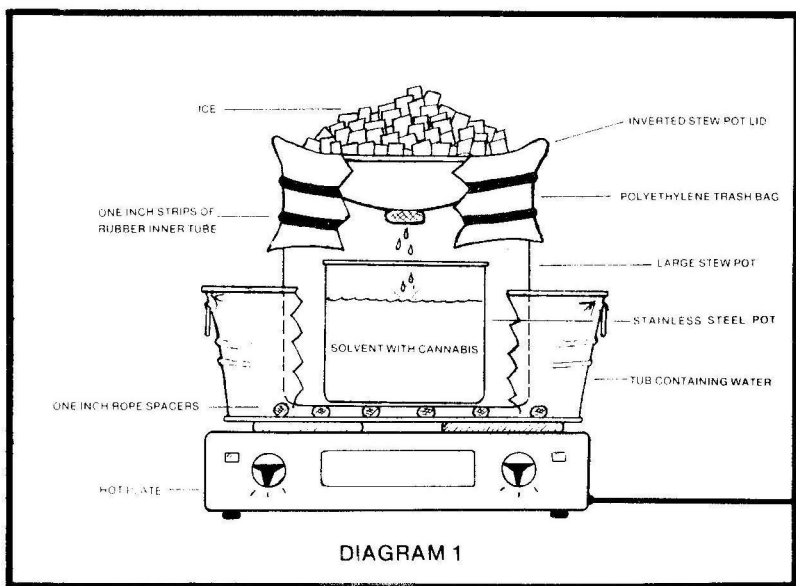
The essential oil is extracted from the cannabis material by refluxing (boiling) in a solvent. This essential oil (containing THC and related substances, chlorophyll, and the substances contributing the taste and smell) dissolves in the solvent (usually an alcohol) while the cellulose parts of the herb do not dissolve. The leached marijuana is removed by straining and the solvent containing the oil is evaporated, leaving as residue the essential oil of the herb.

As it is very dangerous to boil solvents (the fumes and liquid are quite flammable), it is necessary to use specialized methods in order to perform the operation safely.



Prepare refluxing apparatus by collecting the following items:

1. A small pot, preferably of stainless steel, to hold the powdered cannabis material and solvent. The pot should not be over two-thirds full when the marijuana is covered with half again its volume of solvent.
2. A large stew pot with lid at least fifty per cent wider and twice as deep as the smaller pot mentioned above. Both pots should have flat bottoms.
3. A large, deep tub for boiling water, at least twice as wide as the stew pot.
4. A heavy-duty electric hotplate with two burners.
5. Several yards of one-inch hemp or manila rope.
6. Large, thick polyethylene trash bags. Three-mil-thick trash bags are best.
7. Innertube cut into one-inch-wide rubber bands to fit tightly around the stew pot.



*Assembly:* Place the large tub securely on the hotplate. Lengths of one-inch rope are then placed in the bottom of the large tub to keep the stew pot (set in the tub on top of the rope) from resting directly on the bottom of the tub. The small stainless steel pot containing the powdered cannabis and solvent is placed in the stew pot, and the lid is placed on top of the stew pot in an inverted position (upside down). A piece of plastic trash bag is cut and placed over the lid of the large stew pot so that it extends halfway down the side. Seal the unit by securing the polyethylene sheet to the stew pot with two large innertube rubber bands. Position the innertube bands several inches down the side of the pot, allowing some slack in the plastic sheeting. Force out any air that is trapped under the plastic by loosening the rubber bands and flattening the bag with your hand. Pile as

much ice as possible on the plastic bag covering the inverted lid of the stew pot. Fill the tub half full of water and bring the water to a boil. This heats the apparatus to about 212°F., but not above.

As the stainless steel pot containing the cannabis and solvent is heated by boiling water in the tub, the solvent boils. As the fumes rise inside the apparatus, they make contact with the inverted lid of the stew pot, which is cooled by ice from above. These fumes of solvent then condense to liquid, relieving the pressure created by boiling, and drop off the inverted lid back into the stainless steel pot containing the cannabis and solvent. By refluxing in this manner, there is no danger of explosion or of toxic fumes escaping into the air.

The reason that the cannabis and solvent is not put directly into the large stew pot is that the condensing surface area (the ice-cooled lid) must be larger than the surface area of the boiling solution in the stainless steel pot.

The plastic sheeting is used for several reasons: The reaction is completely sealed from the atmosphere, preventing any fumes from escaping or igniting. A rigid seal, such as the locking top of a pressure cooker, is not good, as it would prevent pressure build-up in the stew pot from causing the plastic bag to inflate. The inflation of the plastic notifies the chemist of the pressure increase and also causes ice to fall into the boiling water bath, cooling the rig to a safe temperature and reducing pressure within the system. Pressure will not build up too high unless one neglects to keep enough ice on top or allows the apparatus to heat up too fast before the ice has sufficiently cooled the inverted lid. Reflux for three or four hours. Most of the essential oils of the cannabis material are now dissolved in the solvent.

There are several solvents that work well. Their properties, and the advantages and disadvantages of each, are discussed:

1. Methyl alcohol, methanol, wood alcohol (boiling point 64°C.) This solvent is commonly employed and if used correctly does a fine job. Methanol is available at a lot of pharmacies and in larger quantities at industrial chemical supply companies. It is also available as paint thinner, but it is seldom very pure in this form. Methanol fumes are toxic and explosive. Inhalation of these fumes makes one sick, with pronounced body ache. Continued inhalation of even small amounts may cause permanent damage. Any traces of the solvent remaining in the oil product will be hazardous to the consumer. Methanol evaporates at a uniform temperature (approximately 190°F.) and does not extract a lot of the water-soluble tars, which are not psychoactive. A method for removing traces of the solvent will be discussed later.

2. Rubbing alcohol (most rubbing alcohol is 70% isopropyl alcohol and 30% water). There are several advantages to using isopropyl rubbing alcohol. It is available in many stores at a low price and is much less toxic and explosive than methanol. Unfortunately, because it contains water, many of the water soluble, non-psychoactive substances are also extracted. The oil yield with rubbing alcohol is twice that of methanol, and is proportionally less potent. Water-soluble tars may also give the oil undesirable taste and burning qualities. If the oil is to be re-extracted later with a more selective solvent, however, it matters little what it is like at this point. The water in the mixture also causes it to evaporate at a much higher temperature than methanol. Once the alcohol is completely evaporated the water

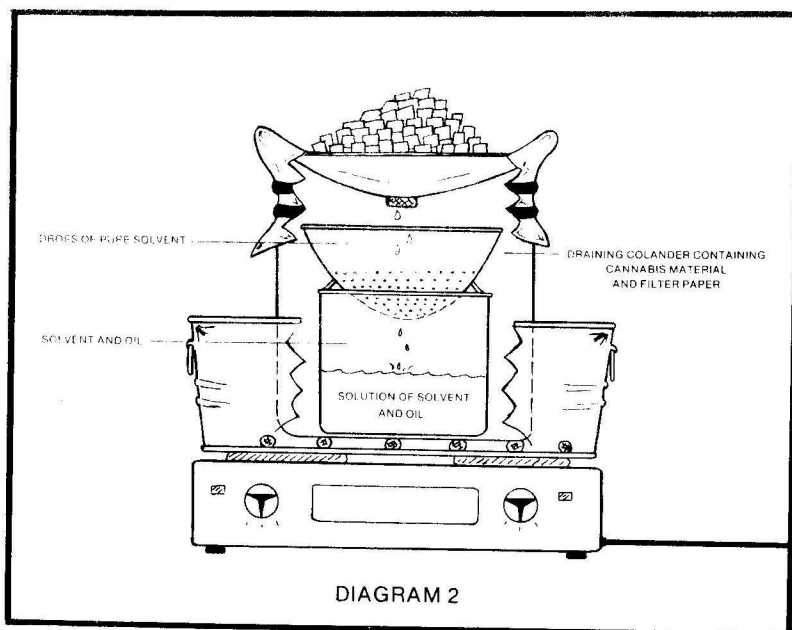
that was in the solvent remains with the oil. This takes a long time to evaporate in a boiling water bath. An oil bath may be utilized. Keep the temperature of the oil in the bath slightly higher than the boiling point of water. Be very careful that no water gets into the oil bath or it may spatter.

3. Ethanol, ethyl alcohol, pure grain alcohol (boiling point  $78.5^{\circ}\text{C}.$ ). This is a very desirable solvent. It has extraction properties very similar to methanol, but is not as toxic. It is very difficult to obtain, however, as it is a major active ingredient in liquor and is heavily taxed. Pure ethanol may be produced from either liquor or fermented material. Denatured ethanol, which is available in hardware stores and pharmacies, contains non-removable poisons which evaporate at the same temperature as pure ethanol. This makes the ethanol unfit for drinking.

4. Petroleum ether (boiling point  $30-60^{\circ}\text{C}.$ ) Pet ether is a light solvent much more selective than any of the alcohols. Extracting with pet ether produces an oil which is twice as potent by weight as oil extracted with alcohol. The cannabis material may be extracted directly with ether but, due to its highly explosive nature, it is advisable first to remove the oil from the plant material with alcohol and then to re-extract this crude oil with ether. This requires a much smaller amount of the dangerous solvent. Petroleum ether is usually available only through chemical supply companies.

#### Step 4: Soxhleting.

After refluxing, it is necessary to remove the oil-bearing solvent that remains still in the expended cannabis material. This is done by draining the dark oil-solvent liquid from the cannabis material and washing the material repeatedly with clean solvent. Obtain a vegetable-draining colan-



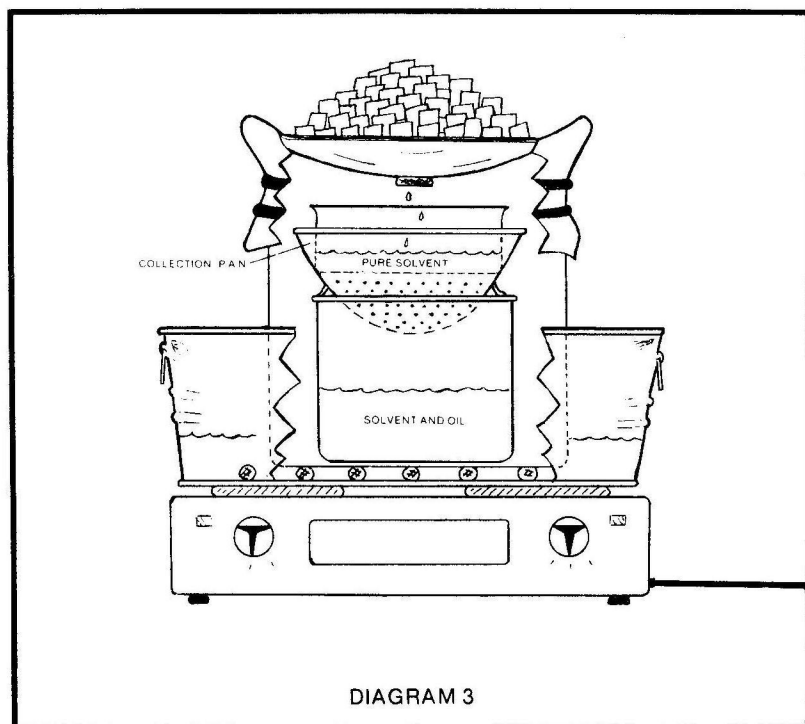


der to fit above the stainless steel pot mentioned before. The colander is fitted with a large coffee filter paper (twelve-inch David Douglas brand papers are quite adequate) and the cannabis-solvent-oil mixture is poured into the colander. The oil-bearing, dark-colored solvent-oil mixture drains from the bottom of the colander, free from particles of vegetable matter. The colander is then set on top of the stainless steel pot, now containing the alcohol-cannabis oil solution, and the apparatus is reassembled in the manner that it was for refluxing.

As the solvent-cannabis oil solution boils, the alcohol fumes rise until they meet the ice-cooled lid, and recondense into liquid. The oil does not evaporate and remains in the stainless steel pot. The drops of pure, recondensed solvent fall from the ice-cooled lid and drip through the colander containing the cannabis material. The oil remaining in the cannabis material is washed out and drains into the stainless steel pot. The oil is totally extracted when several drops of the liquid draining from the colander leave no colored residue when evaporated on a piece of glass. Before opening the apparatus after soxhleting, it is necessary to cool the rig sufficiently to condense any fumes. This can be done by setting the stew pot in a tub of ice and water for several minutes. A thick blanket can be kept soaking in the tub. This is an excellent safety measure, as a water-soaked blanket is an excellent fire extinguisher.

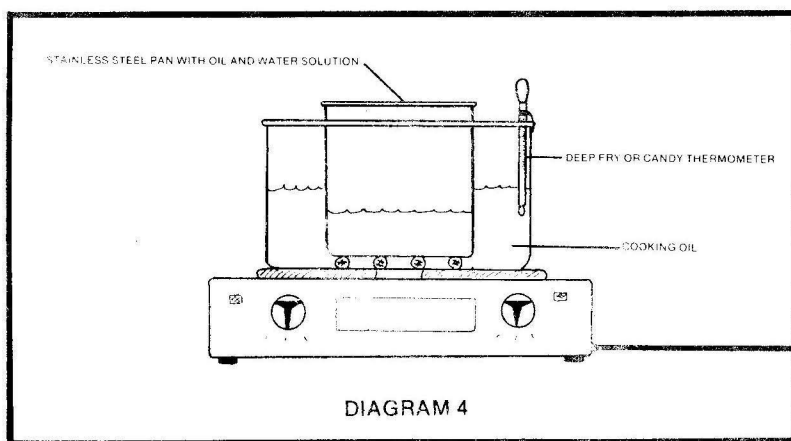
#### Step 5: Removal of the solvent from the oil.

To distill off the solvent, a small collection pan replaces the expended



cannabis material (which may now be discarded) in the colander. The apparatus is then reassembled and returned to the water bath. The solvent-oil solution in the small stainless steel pot boils; the fumes rise and are condensed on the ice-cooled lid as before. The pure solvent drips into the colander where it is collected in the small pan. The oil remains behind in the stainless steel pot. The collected solvent, which is essentially pure, may be saved for a future extraction.

After the solvent is removed and collected, the stainless steel pot containing the oil should be kept in boiling water to remove all traces of the solvent. If using a toxic solvent, or one containing water, it is advisable to add some water to the oil and evaporate it in an oil bath (cottonseed oil works fine) at approximately 220°F. When the water is gone, one can be sure that all traces of the solvent have been removed, as all solvents mentioned here evaporate at a temperature below that of boiling water. The oil may now be eaten or smoked.



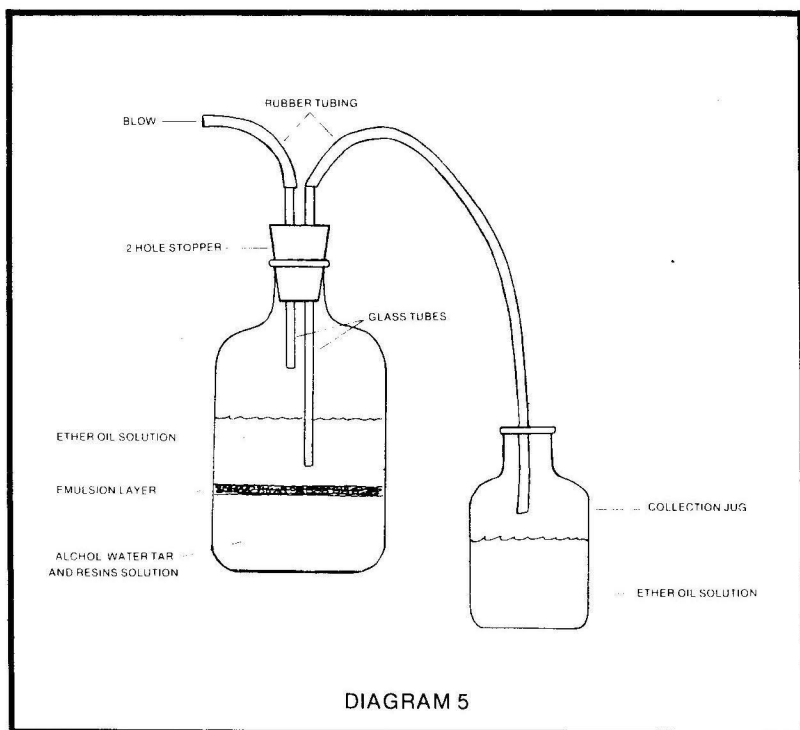
### Step 6: Purification.

Oil produced using this method is quite potent, but still contains substances which give the oil its taste, smell, and color. These are sometimes very pleasant to smoke, and one may decide to leave them in the oil. Removing them, however, greatly increases the potency, but decreases the yield proportionally.

Dissolve the oil from the extraction in five times its weight of alcohol and pour the solution into an equal volume of water in a large glass jug with a screw-on cap. Make certain that the oil, water, and alcohol mixture is not warm. Add a volume of petroleum ether equal to half the volume of the water used. Tighten the screw cap and invert the jug. Turn upright immediately and after waiting a few seconds for the mixture to run down the sides of the jug, slowly open the screw cap to relieve the pressure. Repeat the inversion of the jug about twenty-five times, releasing the pressure each time, and then let the jug sit for about half an hour. The mixture of liquids will separate into three distinct layers. The bottom layer will contain water, alcohol, and the substances in the oil (tars and resins) which are

not soluble in petroleum ether. The thin middle layer is an emulsion of waxes, ether, and air bubbles. The top layer is the purified oil dissolved in petroleum ether.

Fit the jug with a two-holed rubber stopper, glass tubing, and rubber hose. Two pieces of glass tubing are fitted into the two-holed rubber stopper. Be sure to remove the sharp edges by heating the tubing in a gas flame. When fitting the tubing, hold it in a thick towel, as the glass is likely to break and cut your hands. One piece of tubing need only protrude from the stopper an inch on each side. Position the other tube so that when the stopper is tightly fitted to the jug, the tube extends into the mixture to a half-inch from the bottom of the ether-oil layer. Attach to the other end of the tube a length of rubber tubing to transfer the ether-oil solution to a collection jug. The end of the tubing must be lower than the end of the tube in the ether-oil solution to obtain a siphon action. Attach a short piece of rubber tubing to the short glass tube and blow into it gently to start the siphon.



Do not get the glass tube too close to the emulsion layer; any ether-oil solution not removed will be recovered later. Save the ether-oil solution in the collection jug. Add another volume of fresh petroleum ether to the extraction by inverting, separating, and collecting the ether solution, which is added to the first ether-oil solution. Repeat this process until the ether layer remains clear after inverting. This indicates that the ether-soluble oil



is totally extracted from the alcohol-and-water layer. Put no more than several ounces of the combined ether-oil solutions in the stainless steel pot and place the collection pan in the colander. Reassemble the apparatus as was done for the removal of the solvent from the oil after soxhletting. Place the rig in the water bath and slowly heat to 140°F. After evaporating and collecting the ether (save for future use), put the stainless steel pot with the oil into a boiling water bath for several minutes with occasional stirring to remove any residual traces of solvent. The refined oil thus obtained is much superior to the oil obtained from the original alcohol extraction.

**CONVERSION OF CANNABIDIOL, A NON-PSYCHOACTIVE  
COMPOUND IN THE OIL, TO TETRAHYDROCANNABINOL,  
RESULTING IN GREATLY INCREASED POTENCY.  
CONVERSION OF LOW-ROTATING THC TO THE  
HIGHER-ROTATING ISOMERIC FORM.**

The oil produced by alcohol extraction and purification with petroleum ether contains tetrahydrocannabinol, two other compounds closely related to THC but non-psychoactive (cannabidiol and cannabinol), and several other compounds which contribute the taste and smell of the oil. The quality and quantity of the THC in the oil is determined by the quality and potency of the starting material. The oil from very strong cannabis material contains a much higher percentage of THC than the oil from marijuana or hashish that is less potent. The quality of the THC and the characteristics of the effect (high) are determined by the relative positions of the double bonding in the THC molecule. The higher-rotating forms are more potent than the low-rotating and produce a higher, more psychedelic and spiritual effect. Methods for converting THC from low- to high-rotating follow.

The quantity of cannabidiol in the oil is important, as it may be converted to THC, thereby increasing the potency of the oil proportionally. Experience has indicated that the quantity of cannabidiol is usually at least equal to the quantity of THC. This enables one to at least double the strength of the oil through isomerization, and in some cases potency may be increased five to six times.

By using the correct chemicals and methods to convert the cannabidiol to THC, it is possible simultaneously to convert the THC (that which occurs naturally in the oil and also that which has been produced from cannabidiol) to higher-rotating forms. The highest benefit to be obtained is by starting with material high in cannabidiol, isomerizing the cannabidiol to THC, and converting the THC to its higher-rotating form. Both the potency of the oil and the quality of the high are greatly increased. The operation is carried out as follows:

Dissolve the oil from the ether extraction in absolute ethanol or pure methanol in the ratio of one gram of oil to ten grams of solvent. The ethanol may be denatured, but must not contain water. Add one drop of 100 per cent sulfuric acid to the alcohol-oil solution for each gram of oil. Add the acid slowly and stir well. Pure sulfuric acid is very strong and will cause severe burns. When working with it, wear safety glasses, long rubber gloves, and clothing that covers as much of the body surface as possible. If any acid touches the skin, wash immediately with water and bicarbonate of soda. The sulfuric acid should be kept in a safety bottle made by

permanently fitting a glass bottle with a screw top in a styrofoam-lined metal can.

Place a Pyrex pot containing the oil-alcohol-sulfuric acid solution in the refluxing apparatus originally used for refluxing the material in alcohol. Pyrex is substituted for the stainless steel pot because of the reactive nature of the sulfuric acid. Place the rig in the boiling water bath and reflux for two hours. At the end of this time, place the stew pot in an ice-water bath and then open it. Pour the solution into an equal amount of water and extract with petroleum ether as was done in removing the ether-soluble oil from the alcohol extract solution. Then pour the ether solution into four volumes of water and gently invert twenty-five times, taking care to release the pressure each time. Let separate, siphon off the ether-oil layer and discard the water. Pour the ether-oil solution into four volumes of five per cent bicarbonate of soda solution in water. Mix, then separate and siphon off the ether-oil layer. Discard the bicarbonate of soda solution and repeat the previous step (washing with pure water) twice. Evaporate the ether from the ether-oil solution as was done previously in the first purification, using the stew pot apparatus. Collect the pure ether in the pan held in the colander. The oil now contains a much higher percentage of THC (determined by the amount of cannabidiol originally present). The THC is of the high-rotating isomeric form and all of the toxins have been removed from the oil.

### THC ACETATE

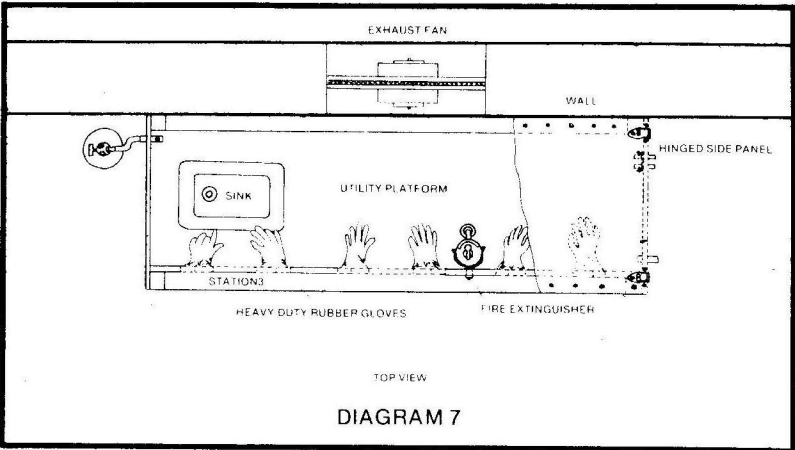
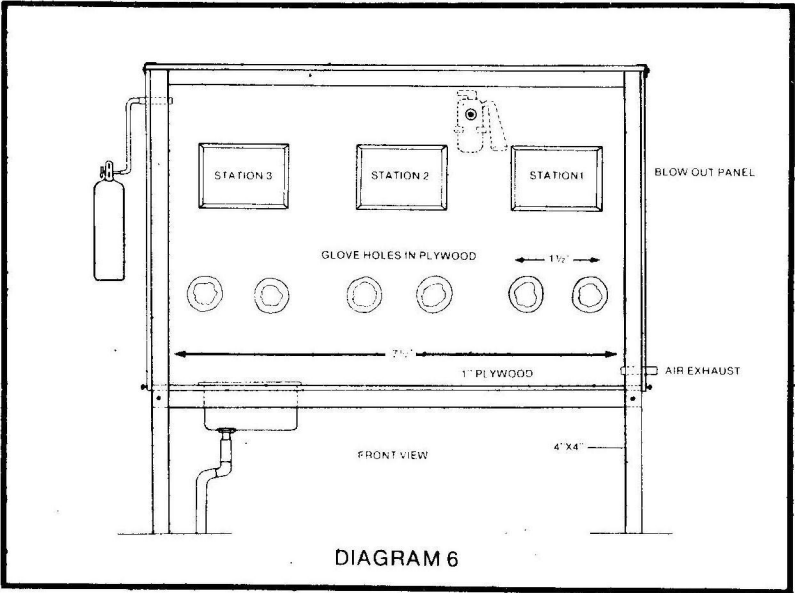
THC acetate has twice the potency of THC. On the Adams scale THC = 7.3, while its acetate = 14.6. Furthermore there is a twenty-five per cent increase in weight after adding the acetate structure. The effect of the acetate is more spiritual and psychedelic than that of the ordinary product. The most unique property of this material is that there is a delay of about thirty minutes before its effects are felt.

#### **Building a safety box in which to convert high-rotating THC to its acetate.**

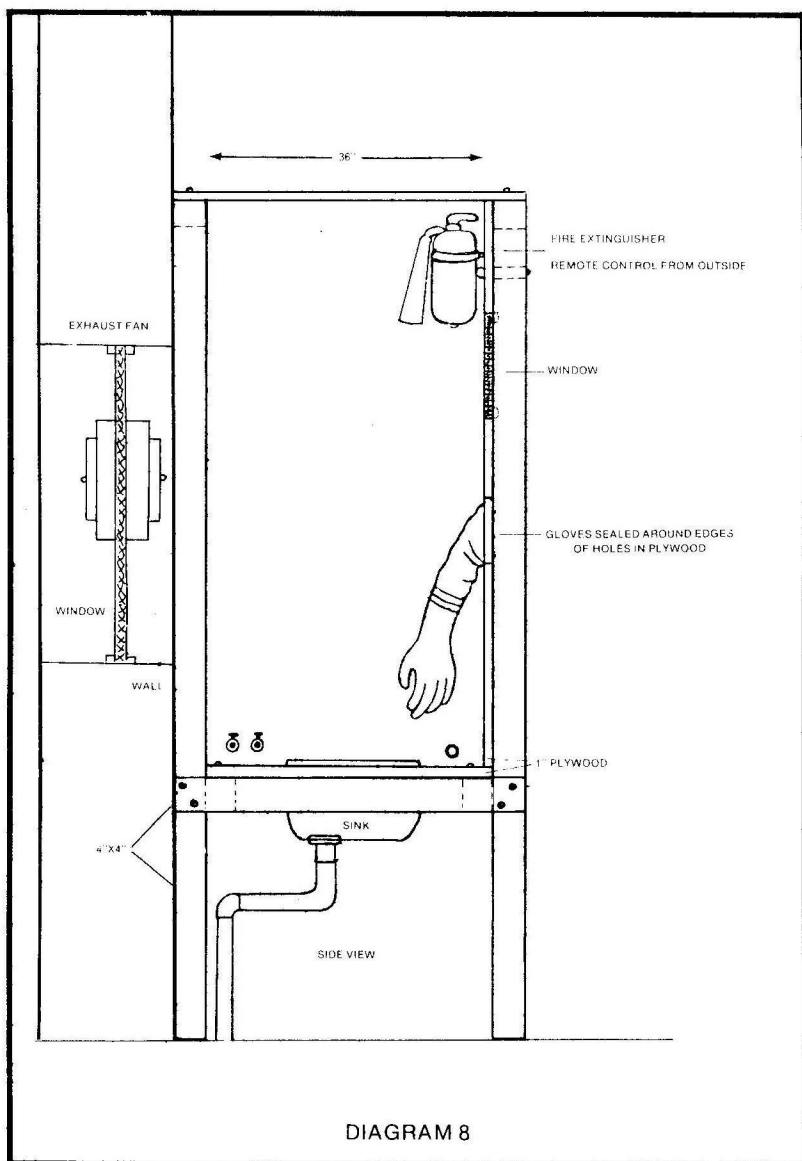
Because this conversion utilizes a very dangerous chemical, acetic anhydride, it is necessary to construct a safety box in which to perform this operation. This places a shield between the chemist and the apparatus, and has the operation taking place in a separate atmosphere. The fumes from heated acetic anhydride are very flammable and poisonous. Inhalation of the fumes is one of the most unpleasant and dangerous experiences we have had, and that is why we feel it is necessary to present this section on building a glove box. Acetic anhydride is so difficult to handle safely that it is a necessity to use standard laboratory equipment and procedure. The reaction is monitored and controlled from outside of the box by observing the equipment through a safety-glass window and manipulating the apparatus with long gloves sealed to the shield. The box should have an adequate exhaust fan with a sparkless electric motor to quickly evacuate any fumes that occur while transferring solutions or from a spill or other mishap. A fire extinguisher should be mounted inside of the box. In case of fire or explosion, the chemist is protected by the thick front piece of the box and by its structural design. The box can also be used for any other chemical operation requiring an artificial atmosphere to avert fire or explosion.

An artificial atmosphere is created by replacing the air in the sealed box with anhydrous nitrogen gas. This makes flame or combustion (oxidation in general) impossible. The nitrogen is introduced into the chamber through an opening in one side, near the top of the box. The displaced air is removed through a valve near the bottom of the opposite end. Success here is determined by attempting to strike a match inside of the box. When oxygen has been removed, this becomes impossible. The exhaust fan is then used only when it becomes necessary to evacuate the atmosphere in the box.

Diagrams showing details of construction follow:







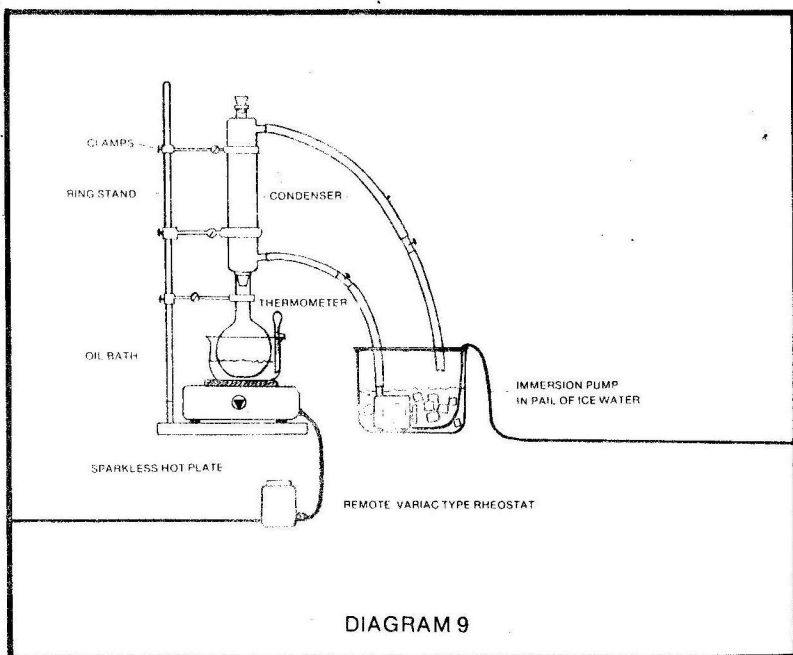
The three stations are utilized as follows: The equipment and bottled chemicals are put into the right-hand side of the box by lifting the hinged side piece. The apparatus is prepared at this station and operated in the middle at station two. The bottles are opened at station one, using the gloves, from the outside of the box after the side flap is closed and the atmosphere has been replaced with anhydrous nitrogen gas. The operator need never be exposed to the dangerous chemicals except within the controlled atmosphere of the box.

The side walls of the box are made of thin plywood and hinged at the top. This serves two purposes: access to the box is available from both ends and in case of explosion (which is unlikely if the chemist is alert and following directions), the force would be expended through the side panels while the thick reinforced front boards protect the chemist.

### Conversion of THC to its acetate.

THC is converted to THC acetate by refluxing for two hours with acetic anhydride. Collect the following apparatus to be assembled as illustrated in the diagram:

1. 500 ml Pyrex round-bottom boiling flask with a ground glass fitting.
2. Tubular type condenser with ground glass male fitting that matches the fitting on the boiling flask.
3. Metal pot of at least 2000 ml as hot oil bath for heating boiling flask.
4. Thermometer for monitoring the oil bath temperature.
5. Sparkless electric hotplate.
6. Rheostat to control temperature of hotplate from outside the box.
7. Ring stand and proper clamps for securing flask and condenser.
8. Cottonseed oil.
9. Acetic anhydride.
10. Immersible water pump, bucket, and hoses for filling condenser.



The principle of the refluxing operation is approximately the same as was used for isomerizing the cannabidiol to THC with the kitchen apparatus. The explosive and noxious nature of the acetic anhydride necessitates the use of the safety box. Although it is not necessary to use a glove

box for the operations of extraction and isomerization using the kitchen method previously described, these steps may also be done in the box if it is convenient, as an added measure of safety.

The solution of acetic anhydride and cannabis oil is boiled in the round-bottom flask. The fumes rise into the icewater-cooled condenser, where they are condensed back into liquid, thus relieving the pressure created by boiling. The drops then fall back into the solution.

Before assembling the apparatus, it is necessary to take these factors into account: The temperature of the hotplate must be controlled from the outside of the box. This necessitates a variac type rheostat in the power line to the hotplate. The pail containing the immersion pump which circulates the icewater coolant through the condenser should also be outside of the box. There should be two small holes in the safety box for the icewater input and return hoses. Although the sink at the left station of the box seems handy for the coolant pump, this would necessitate opening the side panel while the refluxing is in progress to add ice and remove water.

Assemble and operate the equipment in this manner: Open the right side panel and assemble at station one the boiling flask, condenser, oil bath, and hotplate, as illustrated in diagram 00. Each item is secured to the ring stand with adequate clamps. Position the flask at least one-half inch above the bottom of the oil bath. Do not make electrical connections or secure the coolant hoses to the condenser yet, as the entire apparatus will be moved over to the center station before beginning.

The boiling flask, prior to being put in the safety box, should contain a measured amount of cannabis extract. Also place in the safety box an unopened bottle of acetic anhydride, an empty graduated beaker, a beaker containing sufficient cottonseed oil to fill the oil bath to a level slightly above that of the cannabis oil-acetic anhydride solution that will be in the flask, and an empty, open-top container of the same height as the boiling flask and of a slightly larger diameter. This container is for holding the boiling flask safely when the apparatus is dismantled. Loosen the clamps holding the condenser and slide them up the ring stand so that the mouth of the boiling flask is accessible for addition of the acetic anhydride. Close the right side panel; fill the chamber with nitrogen, then, using the gloves through the front board at station one, open the bottle of acetic anhydride. Pour an amount into the graduated beaker equal to three times the volume of the cannabis oil in the boiling flask. Replace the cap on the acetic anhydride bottle and carefully pour the acetic anhydride from the graduated beaker into the boiling flask. Securely replace the condenser on the boiling flask and loosely fit a solid rubber stopper to the top of the condenser.

Open the side panel and carefully move the apparatus to the center station. Connect the input icewater hose to the lower fitting of the condenser. The return hose should run from the uppermost fitting (assuring that the condenser is always filled with circulating water) through a hole in the safety box to a pail containing the icewater and immersion pump. Secure the fittings on the condenser with twisted wire or automobile-type hose clamps. The power wire for the hotplate is also run through a hole in the safety box and connected to the rheostat. The oil bath temperature should be monitored with a thermometer which should be adjusted for observation through the safety glass window. Add the cottonseed oil to the bath. Remove the empty beaker and the closed bottle of acetic anhydride. Close

and secure the side panel. Now, bleed more nitrogen into the box until you are fairly sure that the air has been completely replaced by nitrogen. Striking a match is not a good idea for a test at this time, but if the test is tried a few times prior to beginning the operation, you should have a good idea of how long it takes to drive out the air completely. Fill the pail containing the immersion pump with water and ice, turn on the pump, and fill the condenser with circulating icewater. Begin heating the oil in the oil bath by turning on the electric hotplate. Slowly raise the temperature indicated on the thermometer, giving the solution in the flask time to heat to the temperature of the bath. Notice when the solution of cannabis oil and acetic anhydride begins to fume and droplets of pure acetic anhydride form in the condenser and fall back into the solution. Continue slowly raising the temperature until the solution in the flask begins to boil. Stabilize the bath temperature at this point. Note the time and continue to boil for three hours. Be sure to keep ice in the container with the immersion pump. After three hours of refluxing, turn off the electricity to the hotplate and allow the solution to cool to room temperature. Be certain to keep icewater circulating through the condenser. After the solution has remained at room temperature for at least two hours, check the rubber stopper at the top of the condenser. It should form a perfect seal, but not be too tightly jammed into the condenser. Next turn off the immersion pump and let the apparatus sit at room temperature for another hour. At the end of this time loosen the clamps holding the condenser and slide it up the ring stand as before, giving access to the aperture of the boiling flask. Remove the rubber stopper from the top of the condenser and fit it tightly in the top of the boiling flask. Loosen the clamp holding the boiling flask and remove the flask from the oil bath. Wipe the flask clean of oil and set it into the empty open-top container which was set in the box earlier. Open the side panels and dismantle the equipment.

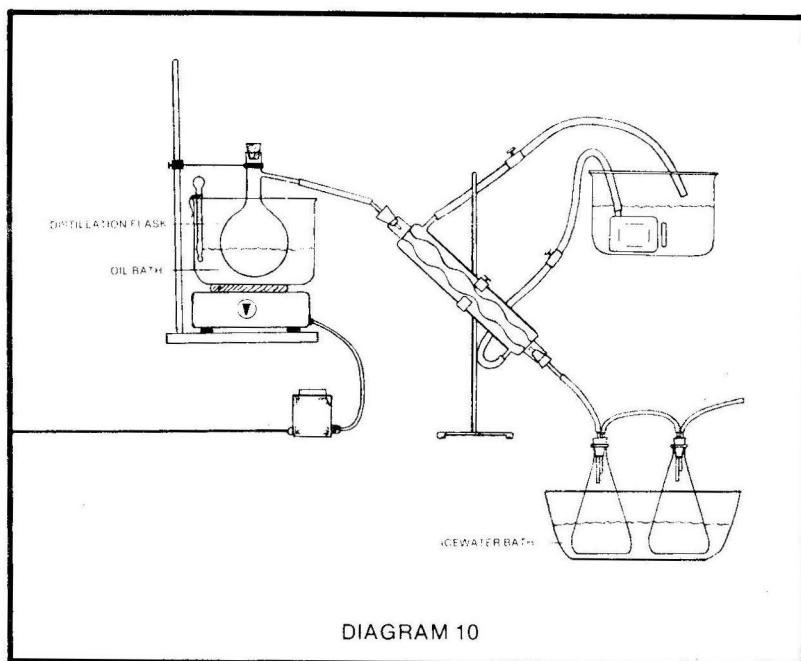
Removing the acetic anhydride by distillation is the next step; see diagram 10. The distillation requires the following equipment not used for refluxing:

1. A Pyrex distillation flask of the same capacity as the boiling flask, and two Erlenmeyer flasks, also of the same capacity.
2. An assortment of glass tubing, flexible tubing, and rubber stoppers.
3. A large pan to be used as an icewater bath for the Erlenmeyer flasks.
4. Several more ring stands and equipment clamps.

As the solution in the distillation flask is heated, the acetic anhydride vaporizes; the fumes rise and travel down the side arm of the distillation flask into the condenser where they are cooled to liquid. The recondensed acetic anhydride is collected in the receiving flask at the end of the condenser. This flask and a back-up flask used for safety are immersed in an icewater bath.

Assemble the equipment at stations one and two as illustrated in diagram 10. Be certain that the condenser is at a great enough angle that no acetic anhydride can lie between the condenser bottom and the exit tube. The glass tube for the introduction of the recondensed acetic anhydride should extend deeper into the flask than the exit tube. The same holds true for the back-up flask, even though it is unlikely that any fumes or liquid will go beyond the first flask. The tube leading from the back-up flask should be open at the end.





Close the side panel, replace the atmosphere with nitrogen gas as before, and proceed as follows: Using the gloves, pour the cannabis oil-acetic anhydride solution from the boiling flask into the distillation flask. Use a funnel with a tube long enough to extend past the side arm of the distillation flask. This eliminates the chance of any solution running into the condenser. Secure the rubber stopper to seal the top of the distillation flask. Add the cottonseed oil to the oil bath and the ice and water to the ice bath. Turn on the immersion pump and the hotplate. Slowly raise the temperature indicated by the thermometer in the oil bath to that used for the refluxing. Retain this temperature until no more acetic anhydride distills. Continue this process until certain that all acetic anhydride is evaporated and collected. Note that the volume of cannabis oil acetate now in the distillation flask will be up to twenty-five per cent more than the volume of the oil prior to acetylation. Retain the oil temperature for one hour after the last traces of acetic anhydride have been removed. Turn off the hotplate, and, with the water still circulating through the condenser, allow the oil to cool to room temperature. Drain the water from the ice bath and wipe the basin completely dry. Using the gloves at station one, remove the two-holed stopper from the Erlenmeyer flask and replace it with a solid rubber stopper. Loosen the clamp and remove the flask from the ice bath. Very thoroughly dry the outside of the flask, removing any traces of water. Carefully pour the acetic anhydride from the flask into a safety container like that used for the sulfuric acid, a glass bottle fitted into a metal can.

Remove from the safety box the flask containing the cannabis oil acetate. Slowly, one drop at a time, add several volumes of pure alcohol to dis-

solve the oil. Pour this solution into five volumes of water and extract with petroleum ether as was done in the purification techniques following the isomerization. Evaporate the ether in the stew pot apparatus as was done before and collect the ether. Redissolve the resultant oil in alcohol and pour once again into water. Extract again with petroleum ether, which is evaporated and collected as before. The resultant oil contains THC acetate and may be consumed in any of the customary manners.

### **PREPARATION OF HASHISH FROM THE INTENSIFIED CANNABIS OIL**

Hashish may be prepared from the extracted cannabis oil by mixing it with finely powdered marijuana. The oil may be at any stage of refinement. Extremely strong hashish is obtained by using oil which has been isomerized, acetylated, and refined through removal of non-psychoactive compounds. Along with the potency of the oil itself, the ratio of oil to powdered marijuana determines the strength. In order for the hashish to be the proper consistency, a minimum of fifteen per cent oil must be used. This gives a product with the same consistency as powdery Moroccan or Lebanese hash. Fifty to sixty per cent oil (about equal parts of oil and powder) is the maximum amount of oil usable to give a product with hashish consistency. This product will be very strong and resemble in appearance and consistency the sticky, pliable charas of Nepal and India.

Powdered marijuana of the finest consistency is obtained by the following method: Clean, very dry marijuana is pulverized in a high-speed blender. The material may be dried in a preheated oven (250° F.) for fifteen-minute intervals. A very fine dust will collect on the blender top. This is sifted through a piece of nylon stocking or a very fine mesh screen.

Many times the taste of the hashish is improved if the oils giving the marijuana its taste and smell are removed from the dust. This is accomplished by extracting it with alcohol in the stew-pot apparatus as described earlier. Further extraction of the compounds contributing to the taste and smell is accomplished by boiling in water. Be certain that all solvent is removed from the dust first, as the fumes might present a fire hazard. Filter the water from the dust and repeat the process with fresh water until the water remains clear, indicating that all soluble substances have been leached from the cellulose material. The cannabis dust is thoroughly dried, and is then ready to be mixed with the oil.

The mixing is facilitated by first heating the dust and oil and then working them together in a large mortar, or by kneading the mass with the hands. Thin, flat, hand-pressed patties like those from Afghanistan may be fashioned, or one may mold clumps of "fingers" or round "temple balls" such as are found in Nepal. Flat sheets and blocks may be formed by pressing the mixture between two heated steel plates in a vise.

### **REMOVING THE OIL FROM INTACT MARIJUANA FLOWERS, INCREASING ITS POTENCY, AND REPLACING THE INTENSIFIED OIL BACK ON THE ORIGINAL INTACT FLOWERS**

Reflux the cannabis material in the same manner as was done with the fine powdered cannabis material previously, except that when processing intact flowers the material is first placed in a cheesecloth bag. The oil is

then extracted from the marijuana in the usual manner. The oil is purified by re-extraction with petroleum ether and then isomerized and acetylated. The tars and resins left behind from the ether extraction remain dissolved in the alcohol-water layer in the extraction jug. Evaporate and collect the alcohol in the usual manner and evaporate the water in an oil bath at 220°F. The tars and resins thus obtained are mixed with the intensified, purified oil and dissolved in the exact amount of alcohol that the completely dry flowers will absorb. This amount is determined by adding clean alcohol to the dried flowers until they will absorb no more alcohol but there is none lying in the bottom of the pan. The saturated flowers are then put into a distillation apparatus and all the solvent is removed and collected. This amount of alcohol is then mixed with the purified, intensified oil and the tars and resins. Using an oven baster type syringe, equally saturate the flowers with the oil-bearing solvent. The saturated flowers are then put into the appropriate apparatus and the solvent removed. A small amount of water is then sprayed on the flowers. A steam iron or Sears wrinkle remover works fine. They are then placed in an oven which has been preheated to 250° and then turned off. As the solvent evaporates at a much lower temperature than the water, when the flowers begin to dry out you can be sure that no traces of solvent remain. The flowers are now coated with the intensified oil and may be over twelve times their original potency.

## **PREPARATION OF OIL CAPSULES FOR ORAL CONSUMPTION**

Capsules of oil for oral ingestion (sometimes called pot pills) are prepared by first mixing the purified oil with an equal amount of butter. The butterfat carries the oil through the membranes of the stomach and intestine. The oil and butter mixture is buffed into two volumes of marijuana, parsley, lactose, or any edible inert powder, and then stuffed into large gelatin capsules.

## **SMOKING OIL BY DIRECT VAPORIZATION**

There are several customary methods for smoking of oil by direct vaporization with heat. The most common is the glass oil pipe, or vapor pipe. The oil is placed in the glass bowl of the pipe and the pipe is heated from below with a flame similar to the method for smoking opium. This is a very efficient method, as very small amounts of oil may be vaporized at a time.

Another method for smoking oil straight is to place a tiny dab of it on a piece of aluminum foil. The foil is then heated from below with a match and the smoke is inhaled from above the boiling oil through a tube or funnel. This is essentially the same as smoking in a glass pipe except that a new spot may be used each time and there is no build-up of residue at the point of vaporization. Before using, heat the foil in a gas flame to burn off any part of the foil which might also vaporize.

## **PREPARATION OF TRANSLUCENT (HONEY) OIL**

One of the most potent and popular of the cannabis oil preparations is a thick, translucent, amber oil which has been extracted from Afghanistan

hashish. This consistency is obtained by removing the colored impurities from cannabis oil which has been purified by re-extraction with petroleum ether.

The purified cannabis oil (which may or may not be isomerized or acetylated) is dissolved in ten times its volume of pure alcohol. An amount of granulated activated charcoal (Norit) equal to half the weight of the oil is added to the solution. The solution is filtered through fine filter paper and the alcohol is removed by evaporation. The residue is a translucent amber oil with the appearance and consistency of dark honey.

### **PREPARATION OF "REEFERS"**

The term "reefer" has sometimes been used to describe a marijuana cigarette which has been impregnated with cannabis extract. This may be accomplished by working the cannabis oil in with the marijuana or tobacco to be rolled, painting the paper with oil before rolling, dipping the rolled joint in tincture of cannabis and letting it dry, or injecting the rolled joint with cannabis tincture and letting it dry.

### **METHODS FOR CONSTRUCTION AND OPERATION OF A HIGH-VOLUME EXTRACTION APPARATUS**

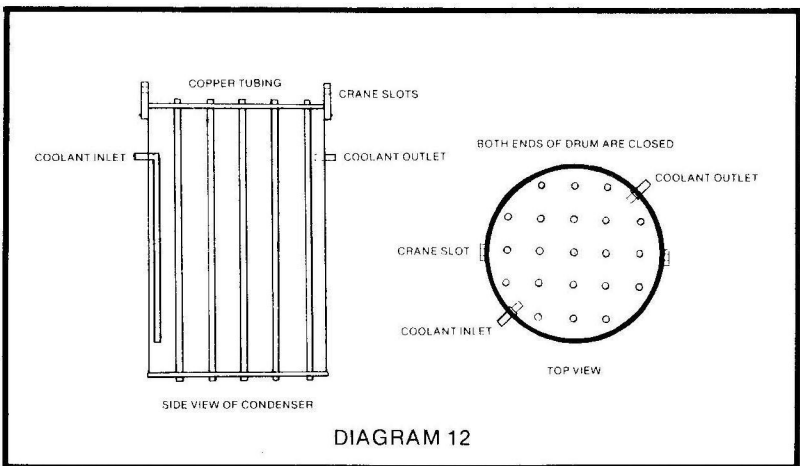
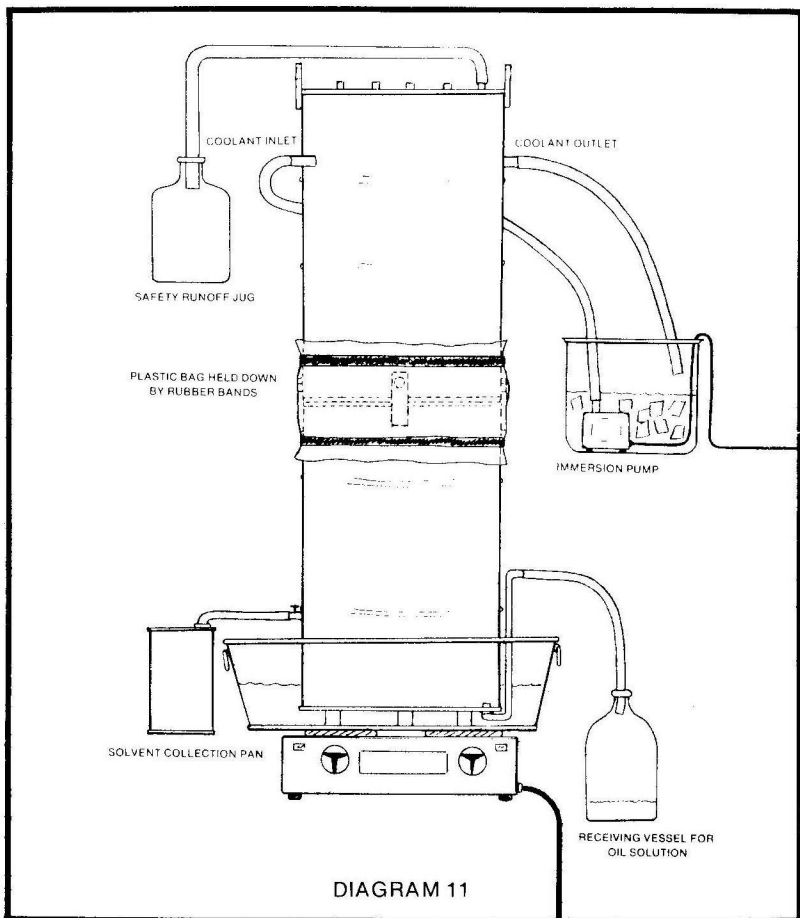
An apparatus may be constructed utilizing two 55-gallon oil drums and equipment purchased from a hardware or surplus store with which very large amounts of marijuana or hashish may be extracted and processed.

The apparatus is appropriately designed to meet the unique problems inherent in high-volume extraction and contains the necessary safety features to prevent mishaps with flammable solvents. The apparatus may be used to perform all the operations: refluxing, soxhleting, distilling, and collecting of solvents.

Even though the apparatus has many safety features (pressure relief valves and construction design which would prevent a minor mishap from being disastrous), the rig must be carefully watched and tended at all times. When working with the large quantities of inflammable solvent required, extreme safety measures must be exercised at all times.

The solvent and cannabis solutions and mixtures are heated, via the tub of boiling water, in the lower oil drum. The upper drum acts as a giant condenser; it is filled with circulating icewater so that when the solvent fumes contact its surfaces, they recondense into liquid and fall back into the lower drum. The tops of the tubes, except for one which acts as a safety pressure relief valve, are closed with rubber stoppers. The solvent fumes condense inside these tubes, as they do on contact with the bottom surface of the condenser drum. The bottom surface of the condenser drum acts as the top surface of the lower boiling drum.

Begin construction of the condenser drum by marking corresponding four-inch grids on the top and bottom surfaces of the oil drum. Drill a hole at each point where the lines intersect and run pieces of one-inch copper tubing through the drum lengthwise. Each tube should protrude at least one inch from the surface on both top and bottom. Solder or weld a watertight seal around the outside of each tube where it passes through the surface.





Inlet and outlet fittings for the circulating icewater coolant are fitted to the top of the barrel. Two metal straps are attached to the top of the drum on opposing sides. The straps have large holes through which a chain is to be run. This allows the apparatus to be lifted with a small overhead crane.

The removal of the top is the first step in the preparation of the lower oil drum. Sturdy legs are attached to the bottom of the drum for maintaining a space between the bottom of the drum and the bottom of the boiling water bath into which the drum is set. A copper drain tube with an on/off valve is attached to the bottom of the drum, enabling the solvent cannabis solution to be drained by siphon. Four metal straps are attached to the top of the drum. These protrude above the top of the drum and are used to secure the upper drum to the lower. A fitting with an on/off valve is run through the side of the drum approximately halfway between top and bottom.

During extraction a chamber containing marijuana or hashish is placed between the drops of recondensed solvent falling from the condenser and the boiling solvent solution. The distilled, recondensed solvent runs through the cannabis material, washing out the oil. The soxhlet chamber is removed and many holes are punctured in the bottom with a nail. A hole is cut in the side of the drum at a point slightly higher than the on/off valve in the side of the lower drum. A plate is fashioned to close the hole when necessary.

Solvent may be removed from a solution and collected by catching the recondensed drops of solvent in a funnel as they fall from the copper tubes. A tube leading from the funnel through the hole in the soxhlet chamber to the on/off valve in the side of the lower oil drum transfers the pure distilled solvent to the outside of the apparatus where it is collected in a metal can.

The following additional equipment is necessary for operation:

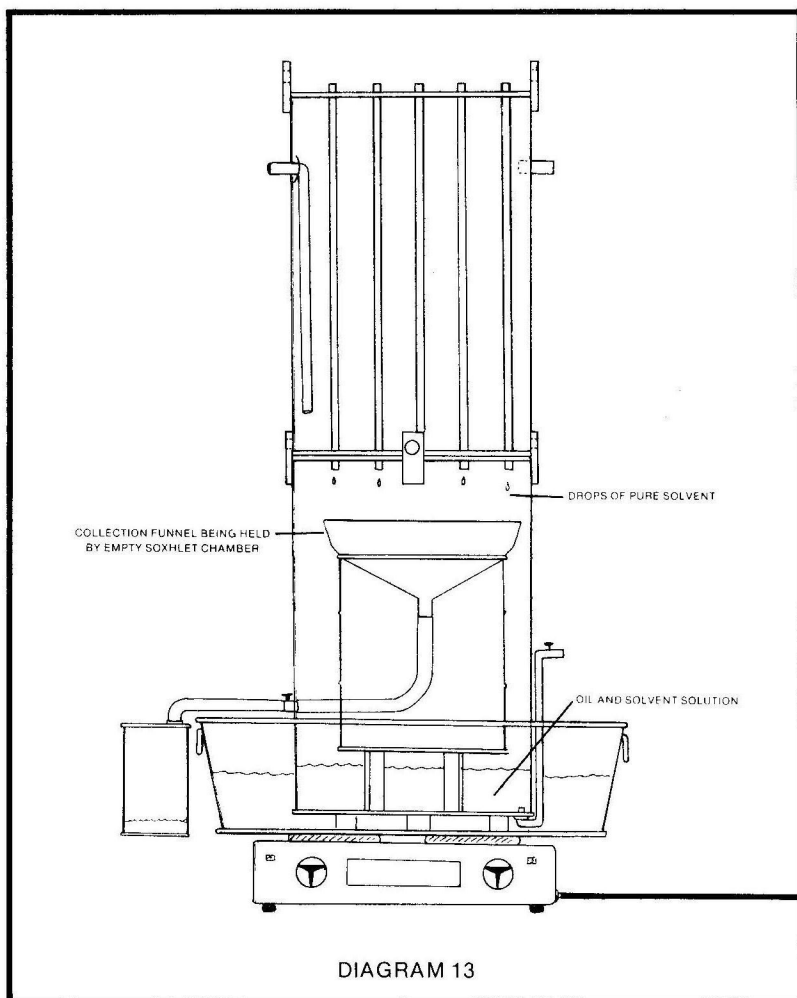
1. A large deep tub, at least twice the diameter of the oil drums, for a boiling water bath.
2. An immersion pump and a large pail for ice and water.
3. A long piece of hose which runs from the top of one of the copper tubes to an empty jug.
4. Rubber stoppers for closing the tops of the remaining copper tubes.
5. Large thick polyethylene trash bags and several giant rubber bands, fashioned out of inner tubes, to fit around the drums.
6. A large funnel of nearly the same diameter as the large drum and a piece of tubing to connect the spout of the funnel to the on/off valve on the side of the lower drum.
7. An overhead chain winch or locking block and tackle for lifting the components.
8. Three heavy-duty sparkless electric hotplates.

The principles of each operation parallel those in the basic extraction method using kitchen and hardware equipment.

Prepare the marijuana or hashish for refluxing by grinding it to a fine powder. Fill the lower drum to approximately one-third full with the powdered cannabis material and add solvent to half fill the lower drum. Be certain that the valve on the side of the drum is closed. The funnel and soxhlet chamber are not yet used. Secure the upper drum to the lower. The crack between the two oil drums is sealed in the following manner:

A large trash bag, with the bottom cut out, is slipped over the apparatus and positioned at the joining of the drums. Inner-tube rubber bands secure the plastic to the drums both above and below the joining. This arrangement acts as a safety valve. Should any pressure build inside the apparatus, the operator will notice the plastic puffing out.

Place the entire apparatus in the tub, which is resting on the three electric hotplates. An immersion pump in a tub of ice and water supplies coolant, which is pumped through the condenser drum. Rubber stoppers are used to seal the tops of all the copper tubes except one. A long piece of garden hose is attached to the tube; the hose runs into a large jug which is set in a place which is safe from flame or electrical spark. As long as ice-water is being circulated through the condenser drum, no fumes or liquid will be noticed entering the jug. If any fumes or liquid are seen coming from the hose, the boiling water should be removed from the bath and ice



and water immediately added. The condenser and coolant system should then be checked.

The electric hotplates are turned on, and allowed to heat the water bath to boiling temperature. This, in turn, heats the mixture of solvent and powdered cannabis material. The solvent fumes rise to where they contact the copper tubes and bottom surface of the condenser barrel, then recondense into liquid and fall back into the boiling mixture.

Reflux the mixture for three hours, then turn off and disconnect the hotplates. Remove the boiling water from the tub, refill with ice and water, and let stand for at least one-half hour. After the apparatus has sufficiently cooled, remove the oil-saturated solvent utilizing a siphon pump to draw the mixture through the drain tube. After removing all the solvent and draining the condenser, open the drums, scoop out all the cannabis material, and store it in a closed drum. Replace the cannabis oil-solvent mixture in the lower drum and assemble for distillation and collec-

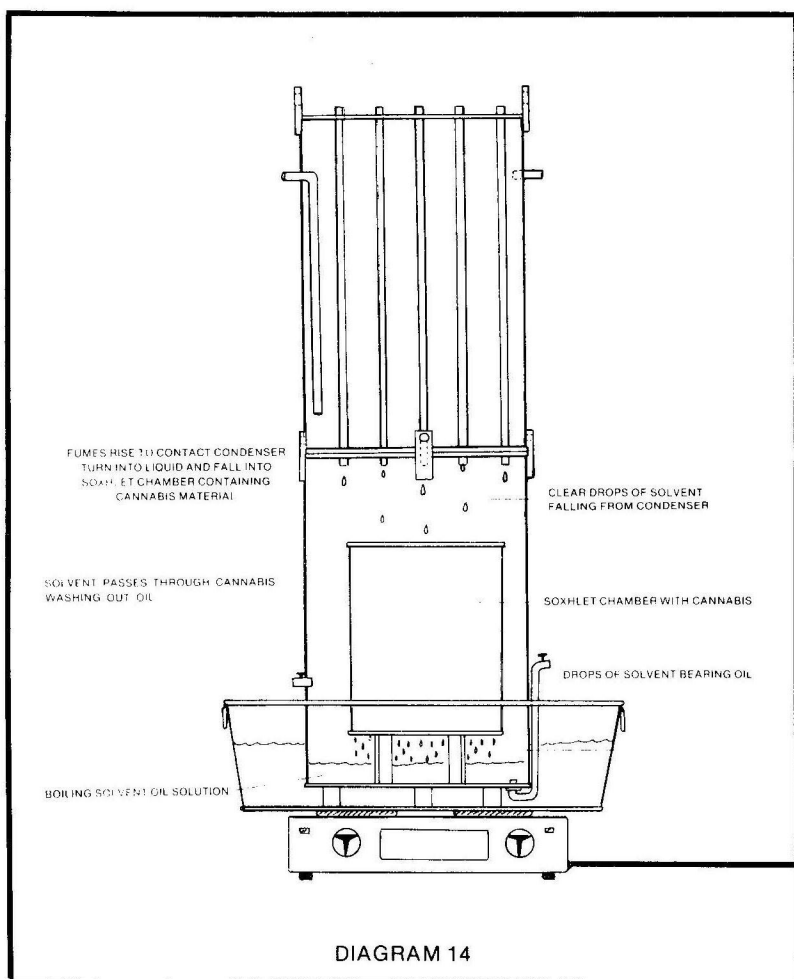


DIAGRAM 14

tion of the solvent. The funnel, which is held by the empty soxhlet chamber, collects the recondensed drops of pure solvent as they fall from the tubes. The liquid runs through a hose to the fitting on the side of the drum. Another hose on the outside takes the solvent to a receiving barrel where it is collected. When all the solvent is evaporated, a heavy film of oil will coat the bottom of the lower drum. Redissolve this in a small amount of solvent and remove the mixture from the drum. Store the mixture in an unbreakable container.

Replace the damp, powdered cannabis in the drum and add the solvent just removed and collected. Assemble and operate the apparatus as before, refluxing for three hours and draining and collecting the oil.

Fit the bottom of the soxhlet chamber with a filter paper and set it in the lower drum. Fill the chamber with the powdered cannabis and add clean solvent to the cannabis until it is saturated and several inches of solvent, which have run through the cannabis material, are in the bottom of the drum. Reassemble the apparatus and bring the water in the bath to a boil. The fumes of the solvent in the lower drum will rise, recondense to liquid in the copper tubes, and fall into and run through the cannabis, washing out the remaining cannabis oil. After several hours of soxhleting, all the oil will be dissolved in the solvent. The cannabis oil-solvent solution is distilled and collected as before. The oil remaining in the drum is dissolved in alcohol and removed. This solution is added to those collected after each refluxing and combined in a large metal pot, the weight of which has been noted. The pot is placed in the dry bottom of the lower drum and the apparatus is assembled for distillation and collection of the solvent. The solvent is removed and the product remains in the pan. If further purification and chemical alteration is desired, proceed according to the methods given earlier.

### ADVANCED REFINEMENT TECHNIQUES

The translucent amber oil produced by charcoal-filtering the ether phase of the extraction and isomerizing the cannabidiol present to THC contains, in most cases, between thirty and sixty per cent THC. Utilizing rather complex and exacting techniques of modern chemistry, it is possible to further refine this oil. Fractional distillation of the oil will yield a product which is up to twice as strong as the ether phase, and can be converted into nearly pure THC. Totally pure THC, a thin transparent oil, can be produced by chemically isolating the pure cannabidiol and then isomerizing it to THC. This is a very complex chemical operation and requires much sophisticated equipment and chemicals. One who is not skilled and practiced in the art of chemistry should not attempt these operations before familiarizing himself with the developments of cannabis chemistry and learning proper safety and laboratory techniques from an expert chemist.

Fractional distillation of the oil requires that the oil be heated to a high temperature in a reduced pressure created by a vacuum pump. This causes the THC and related cannabinoid substances to vaporize. The vapors are condensed back into an oil on contact with a cooled surface. Many of the impurities do not vaporize and are left behind in the flask used for heating the oil. The following is an excerpt of a laboratory method for refinement of crude red oil and purified red oil from the basic extract. The work was done by Roger Adams in 1940 and appears on page 196 of volume 62 of the *Journal of the American Chemistry Society*.

An advanced filtration method may be used to remove non-active elements from the product. This involves filling a tube with a material which retains unwanted constituents of the oil. The oil is dissolved in a solvent and passed through the filtering material. Chromatography of the hexane extract of hashish in the following formula (hexane is a solvent with properties similar to petroleum ether) removed unwanted constituents of the oil amounting to forty-nine per cent of its weight. The chromatographed extract obtained was almost totally composed of cannabinoid elements. After conversion of the non-active elements of the oil, the resultant extract will be nearly all THC. The formula following is taken from the *Lloydia Journal of Natural Products*, page 456, vol. 33, no. 4.

If completely clear THC (a clear, thin, colorless oil) is desired, it is necessary to first isolate pure cannabidiol from the chromatographed oil by converting it to cannabidiol-bis-3,5-dinitrobenzoate. This is then converted back into pure cannabidiol which is now in the form of white crystalline prisms. The formula for this operation is found on pages 456 and 457 of the *Lloydia* volume previously mentioned.

The crystalline prisms of cannabidiol are converted to pure THC utilizing a formula of Roger Adams found on page 2211 of volume 63 of the *Journal of the American Chemistry Society*.

### Fractional distillation

Fractional distillation of the oil requires that the oil be heated to a high temperature in a reduced pressure created by a vacuum pump. This causes the THC and related cannabinoid substances to vaporize. The vapors are condensed back into an oil on contact with a cooled surface. Many of the impurities do not vaporize and are left behind in the flask used for heating the oil. The following is a description of a laboratory method for refinement of crude red oil and purified red oil from the basic extract. The work was done by Roger Adams in 1940 and appears on page 198 of volume 62 of the *Journal of the American Chemistry Society*.

Wild hemp, grown in Minnesota during the season of 1938, was used in the following experiments. The hemp plants were cut after flowering had begun but before seed had set in the female tops; they were stored in a room for six weeks to dry out. A fan was used for circulation and no molding was evident. One-third of the dry hemp plants amounted to stems. These were held and shaken to remove the leafy part of the plant. This clean marijuana was extracted with 95 per cent pure ethyl alcohol. The methods of extraction are described below.

Four twenty-gallon crocks, each with a capacity of 23 pounds of material, were arranged for countercurrent extraction. Each crock held 61 liters of solvent of which 40 were withdrawn at each transfer with 20 liters being retained by the cannabis. After the process had become uniform, the extract of crock #4 at each transfer held approximately 2 gm of solids per 100 cc. Transfers were made once or twice a day as necessary. The most concentrated extract obtained in this manner was passed to a concentrator where most of the solvent was flashed off under vacuum. Never was the temperature raised above 50°C. The evaporation was carried out at 30°C. The concentrated solution contained 23.1 gm of solids per 100 cc 95% ethanol and each 1 cc represented 4.13 gm of hemp.

The red oil from these extracts was obtained by the following methods:



Ethanol extract was poured into a 1-liter Claisen flask with a short, wide neck and filled with glass wool until the flask was two-thirds full. The temperature of the bath was raised gradually from 90° to 140°C. as the pressure was diminished slightly. The distilled ethanol was discarded and the flask again filled to two-thirds capacity. This process was repeated until 1600 cc of extract had been added and the alcohol removed. The temperature was then raised to 200°C. and when the last traces of ethanol ceased, the bath was lowered to 180°C. and the pressure reduced to 30 mm. Care was necessary to prevent the liquid from foaming over. The temperature was raised gradually to 200°C. until distillation ceased.

The bath then was cooled to 170°C. and the pressure reduced to 2-5 mm. The residual product was then distilled. Much care was necessary to keep the bath at the lowest temperature where the oil distilled regularly as there was a marked tendency to foam. The material distilled at between 100° and 220°C. (3 mm) with the bath temperature at 170-310°C. Yield 180-200 gm crude red oil.

This product was dissolved in 500 cc 30-60°C. b.p. petroleum ether and extracted several times with water. The ether layer was distilled and the residue fractionated through a good column having an outside heating unit. The first fraction boiled at 115-120°C. and gave a yield of 70-80 gm. The second fraction distilled at 150-175°C., yielding 25-30 gm. The material remaining in the flask was removed by dissolving in ethanol and filtering from the glass wool. The ethanol was evaporated and the product distilled from a 250 cc flask, b.p. 175-195°C. (2 mm). Bath temperature was 220-270°C. Yield 90-110 gm purified red oil.

### Chromatography

An advanced filtration method known as chromatography may be used to remove non-active elements from the product. This involves filling a tube with a material which retains unwanted constituents of the oil. The oil is dissolved in a solvent and passed through the filtering material. Chromatography of the hexane extract of hashish in the following formula (hexane is a solvent with properties similar to petroleum ether) removed unwanted constituents of the oil amounting to forty-nine per cent of its weight. The chromatographed extract obtained was almost totally composed of cannabinoid elements. After conversion of the non-active elements of the oil, the resultant extract will be nearly all THC. The process following is derived from the *Lloydia Journal of Natural Products*, page 456, vol. 33, no. 4.

### Isolating the cannabinoids from hashish

The National Institute of Mental Health supplied 13 kg of confiscated hashish, origin unknown. The hashish was extracted in a stainless steel pot using 95% ethyl alcohol at 50°C. and was stirred for five hours. A second and third extraction was then completed using 32 liters for 24 hours and 20 liters for 72 hours respectively. The combination of these two mixtures was washed with 5 liters of 50% aqueous ethanol. The solvent was then removed in vacuum at 40°C. to provide a 22.9% recovery, or 3056 gm. This is shown by gas layer chromatography to contain 29.5% cannabidiol, 8.2% cannabinol, and 5.8%  $\Delta^9$ -THC. Florisil (30.5 kg) and methanol (2%) in hexane were used to chromatograph this oil. The resulting dark oil contained 50% cannabidiol, 20% cannabinol, 15%  $\Delta^9$ -THC, and 15% unidenti-

fied components. Although using Florisil (40:1) provided essentially pure cannabidiol by gas layer chromatography, the product could not be induced to crystallize. Crystallized cannabidiol is obtained by using the following modified procedure of Roger Adams.

### **Isolation of pure cannabidiol**

If completely clear THC (a clear, thin, colorless oil) is desired, it is necessary first to isolate pure cannabidiol from the chromatographed oil by converting it to cannabidiol-bis-3,5-dinitrobenzoate. This is then converted back into pure cannabidiol which is now in the form of white crystalline prisms. The process for this operation is found on pages 456 and 457 of the *Lloydia* volume previously mentioned, and a description of it follows.

Cannabidiol-bis-3,5-dinitrobenzoate is made by rapidly adding 300 gm fresh 3,5-dinitrobenzoyl chloride (m.p. 68–69°C.) to a mechanically stirred solution of a chromatographed hashish extract in dry pyridine at 0° under nitrogen. The mixture was stirred for 15 minutes, then warmed in a 60°C. hot water bath for 30 minutes. This mixture was then poured into a mixture of 200 gm of ice and 300 ml concentrated hydrochloric acid and extracted with ethyl acetate (750 ml). The precipitate was filtered and washed with another 750 ml ethyl acetate. The aqueous phase was separated and washed with 500 ml ethyl acetate. The combined organic phases were washed with aqueous sodium bicarbonate (2 × 200 ml) followed by 300 ml distilled water and dried over CaSO<sub>4</sub>. The solvent was removed in vacuum to yield 340 gm of a dark oil. This was purified by crystallization from 1800 ml ethyl ether yielding 194 gm of off-white powdered cannabidiol-bis-3,5-dinitrobenzoate melting at 97–101°C.

Pure cannabidiol is made by adding 220 ml of liquid ammonia to a solution of 288 gm cannabidiol-bis-3,5-dinitrobenzoate in anhydrous toluene (400 ml) at -70°C. in a Parr bomb. The sealed apparatus was mechanically stirred. During five hours the pressure built to 110 psi and the temperature rose to 20°C. The ammonia fumes were released overnight. The product was dissolved in heptane (400 ml) and insoluble 3,5-dinitrobenzamide was removed by filtration. The precipitate was washed twice with 150 ml heptane. The heptane solutions were combined and washed with boiling water (5 × 200 ml) and the solvent removed in vacuum to yield 120 gm of a dark oil. Chromatography on 180 gm of this product on 3400 gm of Florisil and elution with 30% chloroform in hexane yielded oily cannabidiol (140 gm). Crystallization from 30–60° petroleum ether yielded 99.2 gm white prisms and recrystallization gave 94.8 gm pure cannabidiol.

### **Conversion of pure cannabidiol to pure THC**

The crystalline prisms of cannabidiol are converted to pure THC utilizing a formula of Roger Adams found on page 2211 of volume 63 of the *Journal of the American Chemistry Society*. The following is a description of a method for producing pure THC.

### **Isomerizing the cannabidiol with sulfuric acid**

One drop of 100% sulfuric acid was added to a mixture of 1.94 gm of crystalline cannabidiol in 35 cc of cyclohexane. After refluxing for one hour, the alkaline beam test was negative. The solution was decanted from the sulfuric acid, then was washed twice with aqueous 5% bicarbonate

solution and twice with water. It was then evaporated. This residue was distilled under reduced pressure to yield pure THC with a rotation range of 259° to 269°.



*The leaders of the Justice Party and the Republican People's Party, Turkey's two leading political groups, have come out strongly in favor of letting the nation's 100,000 drug farmers grow opium poppy and hashish hemp.*

*Bulent Ecevit, leader of the Republican People's Party, started it all off recently when he called during a campaign speech for an end to the hashish ban. Cheering farmers in the audience ran up and gave him a bouquet of hashish flowers.*

*—From the San Francisco Examiner, October 2, 1973.*

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