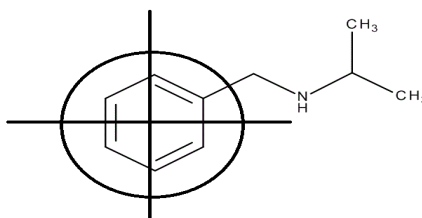
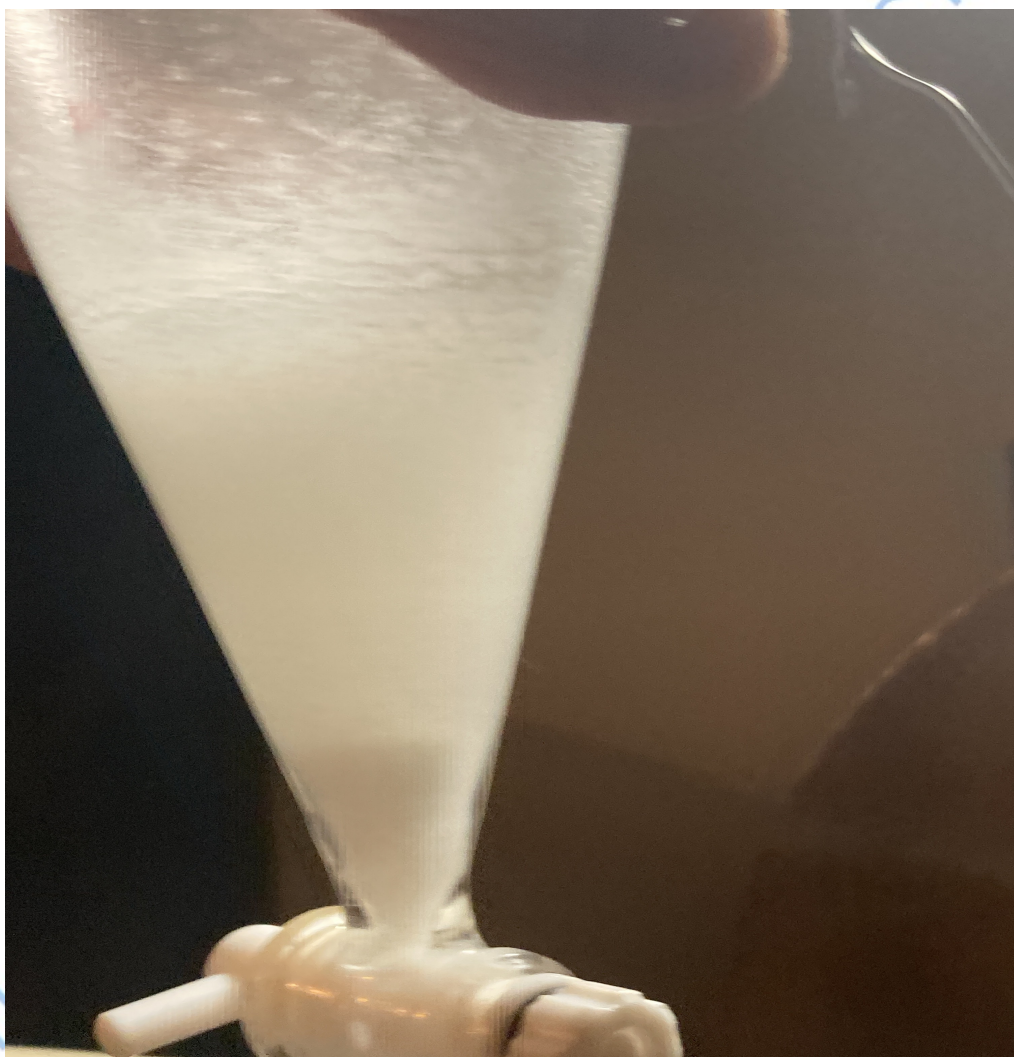


Steam Distillation of Hydrolyzed Methamphetamine

a laboratory write-up by
The n-Isopropylzodiac Killer



STEAM DISTILLATION OF HYDROLYZED METHAMPHETAMINE
A LABORATORY WRITEUP BY THE N-ISOPROPYLZODIAC KILLER
INTERNAL COVER

PUBLISHED BY
**THE NATIONAL STEAM DISTILLATION SOCIETY OF HEATED ALKALINE
HYDROLYSISTS**

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TABLE OF CONTENTS

1. BACKGROUND INFORMATION
2. THINGS YOU WILL NEED
3. SELECTING AND PURIFYING AN ORGANIC SOLVENT
4. INITIAL DEPROTONATION AND HYDROLYSIS
5. STEAM DISTILLATION
6. PURIFICATION OF THE ORGANIC SOLUTION
7. PROTONATION
8. COLD WATER EXTRACTION OF THE PROTONATED PRECIPITATE
9. RECRYSTALLIZATION
10. REFERENCES AND ACKNOWLEDGMENTS, MATERIAL SAFETY DATA SHEETS
11. TRANSPOSITIONAL HOMOPHONIC SUBSTITUTION CIPHER

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BACKGROUND INFORMATION

Abstract: High temperature, pressurized Alkaline Hydrolysis is carried out upon a sample of adulterated street Methamphetamine, and the resultant freebase amine is steam distilled directly into a purified organic solvent, from which it is protonated, evaporated, extracted with cold water and recrystallized.

Claim: Mysterious unknown cutting agents are destroyed in the procedure, allowing for a thorough isolation of Methamphetamine Hydrochloride regardless of which adulterants were added by the manufacturer in an attempt to increase profits and potentially damage the health of their consumers.

Disclaimer: The information compiled in this write-up is a product of many countless laboratory sessions, and, as such, the photography within may not necessarily come from one single purification, but instead, many different extractions and distillations separated by days or weeks. These photographs **do** portray an accurate example of the written content, and are used as substantiating evidence for the credibility of the author, but otherwise they may show extractions of varying quantities or materials throughout. The italicized writing beneath them is true, accurate and honest to the fullest extent, and to the best of the author's knowledge, and can be relied on absolutely. They have been redacted with blacked out backgrounds for the operational security of the author.

The activities described and portrayed by this publication are an extremely serious crime in most civilized countries of the world, and care should be taken by anyone wishing to perform the acts described.

This document is **not** for educational purposes only.

THINGS YOU WILL NEED

LABORATORY GLASSWARE AND EQUIPMENT:

1. 24/40 (250-1000mL) single necked round bottom flask.
2. 24/40 3-way distilling adapter (with optional thermocouple)
3. 24/40 (200mm-300mm) Liebig condenser
4. 24/40 vacuum take off adapter
5. 24/40 (250mL-500mL) separatory funnel with PTFE stopcock
6. Temperature variable electric hotplate (with optional magnetic stirring)
7. Thermometer
8. Heating mantle, heavy duty aluminum foil, steel wool or copper gauze
9. Submersible fountain pump and associated hoses
10. 0.5" steel adjustable hose clamps
11. Ice water bucket
12. Various borosilicate glass vessels of differing heights with threaded PP5 lids
13. PTFE thread-sealant plumbers tape
14. Hazardous liquid waste disposal container(s)
15. Various glass droppers and HDPE pipettes
16. Unbleached coffee filters or filter paper.
17. pH indicator strips
18. Shallow-walled, large surface, Pyrex evaporation dish
19. Tupperware
20. Ice
21. Picture frame glass plates
22. 5/16" polyvinyl hose
23. 2x HDPE applicator bottles
24. Scissors

CHEMICALS, SOLVENTS AND DRYING AGENTS: (see ch. 10 for MSDS)

1. Distilled Water
2. Sodium Hydroxide ^[1]
3. Organic solvent (Toluene or Diethyl Ether) ^[2]
4. Sodium Chloride
5. Non-Indicating anhydrous Silicon Dioxide or 3a/4a molecular sieves
6. Concentrated Sulfuric Acid ^[3]
7. Sodium Bicarbonate ^[4]
8. Calcium Chloride (optional) ^[5]
9. 2-Propanol / anhydrous Isopropyl Alcohol ^[6]
10. Anhydrous Acetone ^[7]

SELECTING AND PURIFYING AN ORGANIC SOLVENT

Before this procedure can be applied to adulterated street Methamphetamine, an effective organic (non-polar) solvent must be chosen, and cleaned thoroughly before dissolution of freebase Methamphetamine can occur.

In the opinion of the author, there are only a small handful of sufficient solvents capable of effectively dissolving the freebase amine, and thus far, Toluene and Diethyl Ether have demonstrated the best possible performance in dozens of extractions and distillations of the amine. These two have their advantages and disadvantages, and many factors play a part in these. Disregarding regional or geographic-related availability here are some of these data points.

Keep in mind, these points can be subject to influence of the *opinions* of the author, and as a result, some information might be debatable or disputable. Entries marked with (**) indicate that certain points can be utilized as either advantages OR disadvantages depending on the intentions of the chemist handling the solvent.

TOLUENE

ADVANTAGE	DISADVANTAGE
Stability (can be stored)	Higher boiling point** [2]
Non-Polarity	"Dirtier"
Insolubility of Hydrogen Chloride** [8]	Availability/Accessibility

DIETHYL ETHER

ADVANTAGE	DISADVANTAGE
Lower boiling point** [2]	Hazardous Instability
Ease of accessibility	Solubility of Hydrogen Chloride** [9]
"Cleaner"	Miscibility with water

**

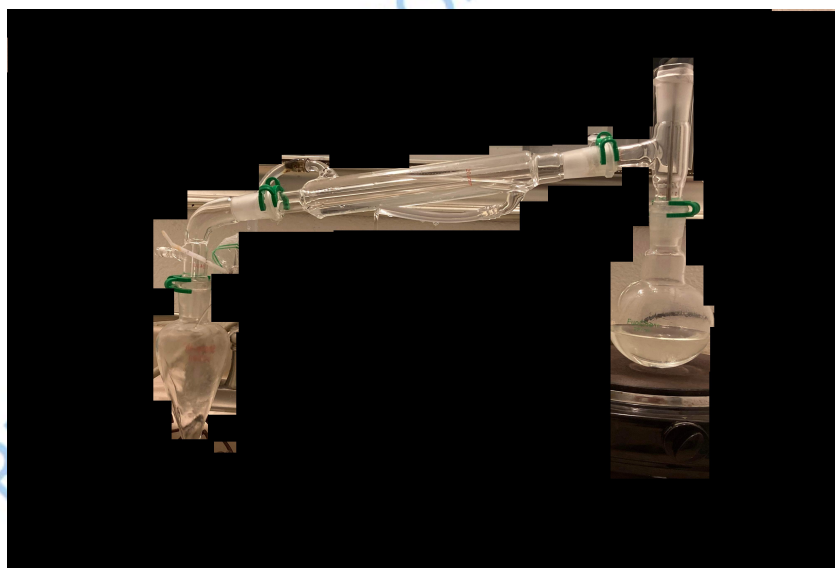
Toluene's higher BP can be used to preserve quantity of available solvent remaining.	Hydrogen Chloride's insolubility in Toluene could lead to difficulties achieving thorough protonation.
Ether's lower boiling point can result in loss of available organic solvent quantity.	Hydrogen Chloride's solubility in Ether can be utilized for easier protonation.

Whichever solvent is chosen, unless the chemist has access to fresh, analytical or reagent grade materials, it is necessary to water wash, distill and dry the solvent before it can be used for elution of the amine.

If Diethyl Ether is the solvent of choice, Butylated Hydroxy Toluene (BHT) should be added (0.1% by wt.) if the chemist intends to store this Ether^[10]. Failure to do so can lead to the formation of highly dangerous peroxides, which are shock, heat and friction sensitive explosive precipitates which can detonate^[11] from even slight disturbances and are created when Diethyl Ether is exposed to oxygen for sustained periods of time, the duration of which varies depending upon the source.

The author has managed to prepare extremely high quality anhydrous Toluene from commercial lacquer thinner using the following approach:

1. Brine **NaCl(aq)** wash, followed by distilled water wash to remove water soluble impurities and Sodium ions from the brine.
2. Removal of additional solvents in the mixture by simple distillation, and distillation of the final fraction (Toluene) to obtain only volatile solvent material.
3. Drying over a suitable drying agent (SiO₂, MgSO₄, NaCl, CaCl₂, etc.)
4. Second distillation over a suitable drying agent.
5. Drying over a suitable drying agent.
6. Assessment of the boiling point of the target distillate to verify the presence of the known scientific attributes and properties of Toluene.



Distillation of a commercial lacquer thinner to isolate Toluene.



Fraction 1



Fraction 2

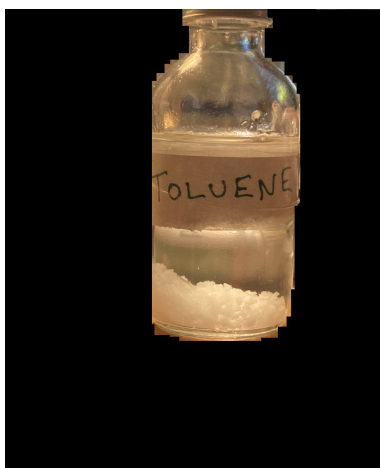


Fraction 3

By receiving these fractions directly in a separatory funnel, they could be preserved or discarded as desired.



Additional Distillation was carried out of the final fraction of the Lacquer Thinner, this time with a drying agent in the distilling flask. It was conducted very slowly and gradually, with careful attention to detail to ensure the cleanest Toluene possible.



Twice-distilled Toluene stored over baked Sodium Chloride. Non-indicating, anhydrous silicon dioxide or 3a/4a Molecular Sieves are far superior solutions as non-reactive

drying agents for this particular application. By the time the author began using SiO₂ as the default drying agent, the switch from Toluene to Diethyl Ether had already been made, but Sodium Chloride will work nicely for the photographed application if it has been dried thoroughly with a microwave or conventional oven first. As Toluene becomes closer to dryness, the characteristic cloudiness caused by micro-emulsions will disappear, and the Toluene will become progressively more clear in its transparency.

There are many routes to a clean organic solvent, if the chemist does not have access to laboratory grade supplies, and the approach can be varied or altered and experimented with as desired. These three basic principles will be irreplaceable for the clandestine drug chemist:

1. Water washes
2. Drying agents
3. Distillation (simple and fractional)

There are many available commercial sources for Toluene and Diethyl Ether if the chemist is limited to such sources. If these sources are utilized, it is *imperative* that the chemist expends considerable effort in the purification of these solvents, because during this procedure, they will make direct, sustained contact with freebase methamphetamine in solution and the potential for contamination is extremely high. These sources include, but are not limited to:

Diethyl Ether	Toluene
Engine Starting Fluid	OTC Toluene
Production via H ₂ SO ₄ +ETOH	Lacquer Thinner
	Premium, unleaded 91 Gasoline

Some form of boiling bath should ideally be used during distillation, but a heating mantle will suffice under certain conditions if necessary. Whenever an organic solvent is obtained from a commercial product or solvent mixture, the resultant distillate should have its boiling point accurately tested to verify the quality of the solvent. Diethyl Ether should not be distilled to dryness.

Regarding OTC sources: The author recommends Engine Starting Fluid for Diethyl Ether, and OTC Toluene (such as Kleanstrip or Sunnyside Toluol) in US states with fewer VOC restrictions. Otherwise, some brands and formulations of Lacquer Thinner may contain a substantial percentage of Toluene. The MSDS should be consulted for any product to verify the presence of the desired hydrocarbon.

INITIAL DEPROTONATION AND HYDROLYSIS

An aqueous solution of street Methamphetamine (8.5g) was prepared via dissolution in fresh, Distilled Water in excess, and – upon complete dissolution at room temperature – the solution was filtered to a clean glass vessel with a threaded PP5 lid. This vessel's threads were then wrapped with PTFE tape to ensure an air-tight seal, and an electric hotplate was configured for the hydrolysis.

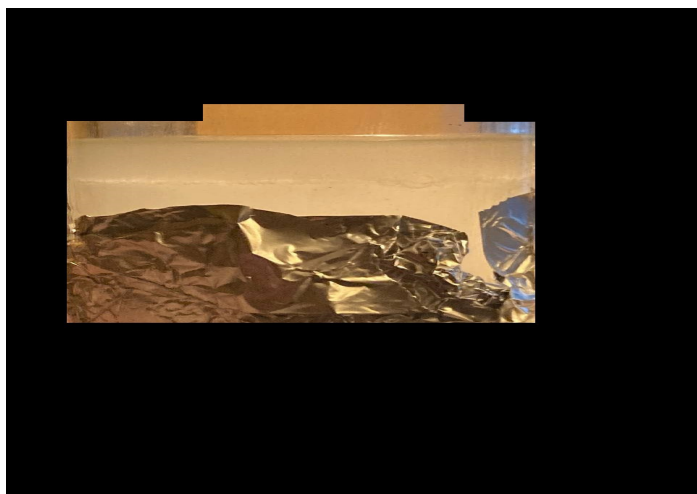
In a separate vessel, a large excess (~25-50g) Sodium Hydroxide was dissolved into a generous quantity of distilled water, so that the concentration of NaOH was over 30%, and the pH was tested to indicate highly alkaline (>13.5+).

Slowly, the aqueous Methamphetamine solution was simmered on moderate heat with the lid **removed** and thickly wrapped aluminum foil to prevent catastrophic failure of the vessel's structural integrity. This initial heating was performed in an effort to steam away any potential volatile P2P-related impurities prior to deprotonation^[12] of the amine and warm the hydrolysis reactor to aid in the acceptance of Sodium Hydroxide for thorough liberation of the amine.

Gradually, NaOH(aq) was added dropwise to the hydrolysis reactor, and this continued until two conditions were met:

1. pH of the reactor contents reached highly alkaline (>12.8+)^[13]
2. Full liberation of the amine (by visual assessment) was achieved.

Upon the establishment of these conditions, a thick, cloudy freebase formed atop the water, and at this time, the reactor was sealed with its PP5 lid and threaded tightly to avoid volatile losses of the organic material in the reactor.



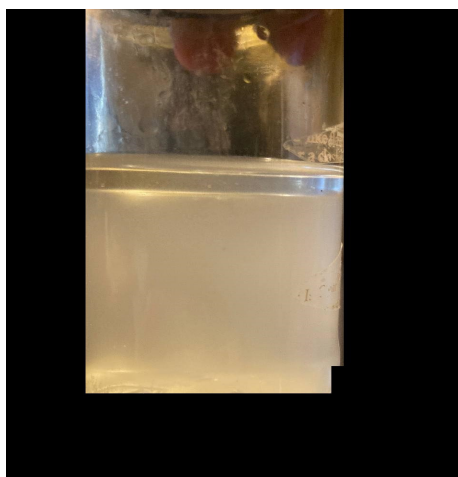
Initial liberation of the freebase. Note the cloudy and murky discoloration.

Intermittent swirling and removal from heat was required to ensure a safe hydrolysis, as over-pressurization and thermal shock can lead to total catastrophic, structural failure of the reactor if the chemist is not careful. Considering the extremely dangerous situation of an exploding container of hot Sodium Hydroxide, such precautionary measures should be taken to the utmost, and the reactor should be observed and attended to at all times. Proper PPE should be donned in the vicinity of hot alkali solutions such as this. The hydrolysis should only be carried out in thick-walled glassware designed for heating.

Note: In experimental efforts to avoid a pressurized hydrolysis and the associated risks, the author has done many of these in a traditional reflux configuration with a water-cooled reflux condenser and boiling flask, but in all instances, the yield of purified methamphetamine was **always** substantially lower, averaging between 10-15% by weight from the starting material volume. Consequently, the author began conducting these hydrolyses in a sealed reactor and has done so *dozens* of times without a failure of the reaction vessel on any occasion. The resultant yield could then be expected to range from 29-39% by weight from the starting material volume after all purification steps were completed.

Visually, there were indicators of a complete hydrolysis of the base amine. It is known that Methamphetamine freebase is a volatile, **clear**^[14] oil. It was then the assumption of the author and his mentor that any discoloration or opacity in the material floating atop the water in the reactor – provided Sodium Hydroxide was not the cause – could only be caused by an impurity or deliberate adulterant. Few chemicals can survive being heated under pressure with Sodium Hydroxide, and in fact, the process is used to destroy *entire human bodies* in a process known as “liquid cremation”.^[15]

After approximately 45 minutes, the hydrolysis reactor was removed from the hot plate and allowed to cool to room temperature, still sealed tightly.



Hydrolyzed Methamphetamine Freebase. Note the clearly apparent transparency in the upper phase layer, and the “shrinkage” (the word 'reduction' was intentionally avoided here) of the aforementioned upper phase layer. Some rough preliminary measurements in various tests have indicated a ranging 30-50% “loss” in volume by measuring the height of the upper phase itself relative to the hydrolysis vessel.

It is likely important to remember that some “murky” discoloration of the upper phase could potentially be caused by the presence of undissolved Sodium Hydroxide, and the chemist should exercise caution with regards to not “over-hydrolyzing” this base oil.

AUTHORED BY THE N-ISOPROPYLALCOHOLIC KILLER

5 STEAM DISTILLATION

To a 250mL 24/40 separatory funnel, there was added approximately 150mL Toluene or Diethyl Ether. This organic solvent had been thoroughly water washed and dried in the same separatory funnel prior to this step of the procedure. The separatory funnel had then been rinsed with excess distilled water, degreased with anhydrous 2-propanol, then dried with anhydrous acetone before the organic was added.

A simple distillation set-up was constructed, wherein a 250mL round bottom flask was charged with 100mL distilled water, then fitted with a 3-way distilling adapter (which was stoppered), a 200mm Liebig condenser and a 24/40 vacuum take-off adapter. The vacuum take-off was mated to the separatory funnel, the condenser was fitted with hoses – through which room temperature water was circulated from bottom to top – and the distillation could begin.

Quickly, so as to avoid prolonged exposure to ambient air, the hydrolysis reactor was emptied into the round bottom flask through the *joint atop the flask, but not through the 3-way stopper joint*. This was done to avoid the inadvertent pouring of impure hydrolysis reactor contents down the interior of the condenser, which might contaminate the distillate as it traveled along the distillation path. The 3-way was then connected back to the round bottom, and the stopper was verified tightly sealed.

The distilling flask was then heated very slowly, and not to the boiling point of water, but rather a “hot simmer”, probably ranging from 75-85 degrees Celsius.



During steam distillation of the methamphetamine freebase.

By receiving directly into a separatory funnel, the aqueous phase in the receiver could be drained in real-time as it formed. It was collected in a secondary vessel charged with Toluene (or Diethyl Ether) to preserve any freebase which might have inadvertently been drained from the receiver, but subsequent tests of this secondary receiver have never showed a significant presence of misplaced freebase.

This distillation should be carried out until the condensate in the Liebig Condenser appears to be water by visual assessment. The chemist might benefit from some experience with the procedure in making this visual identification, but the base oil is notably distinct, as it will appear thicker and more viscous than water distillate, and it will move in a different way entirely along the distilling path.

The base oil may appear cloudy or slightly opaque early in the distillation path, subsequently clearing up as it nears and lands in the receiver. This is normal, and probably ideal.

The distilling flask may still contain a relatively thin organic phase atop the aqueous contents of the flask. This, too, is normal, and it is the opinion of the author and his mentor that there is often a freebase compound present in the distilling flask which simply does not follow steam through a distilling system. Methamphetamine **will** steam distill, so the chemist should not expend much energy or time trying to distill the entire organic phase over to the receiver.



Steam Distillation of Methamphetamine Freebase as seen from below the distilling flask looking upward. Note the base amine collecting at the start of the Liebig condenser towards the right of the photograph.

It may be advantageous to the chemist to continually add fresh distilled water to the distilling flask throughout the distillation to ensure sufficient production of steam, but if this is done, the receiver *must* be a separatory funnel, so that the accumulating aqueous phase can be drained as the distillation proceeds.

When the chemist is satisfied that the base amine has “come over” completely to the organic solvent in the receiver, the heat source can be disengaged, and the assembly is cooled to room temperature with the stopper still in place. The only way for pressure to exit the system should be through the vacuum take-off port, which will vent steam throughout the

process normally.

Finally, the receiver was disconnected, and the stopper was cleaned thoroughly, then fitted to the separatory funnel. The distilling flask was disconnected from the assembly, and its contents were quickly poured into a large storage container charged with an arbitrary, large volume of Toluene.

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PURIFICATION OF THE ORGANIC SOLUTION

The separatory funnel, filled with the steam distillate, Toluene and a residual aqueous phase from the distillation was unstoppered. With sustained swirling, the aqueous phase was slowly drained via the stop cock until no visible water could be seen in the Toluene or below it.

0.5g Sodium Chloride was then added to the separatory funnel to flocculate Sodium Hydroxide bound to the organic solution, and the organic solution was washed with 3x 50mL distilled water. Each water washing volume was allowed to sit and settle with the stopper of the separatory funnel open for some reasonable period of time. During this time, the separatory funnel was swirled numerous times to assist the water in dragging the sodium salts downward towards the stop cock.

Each wash of Distilled Water was slowly drained from the separatory funnel. After the final water wash, the separatory funnel was swirled several times, and this allowed small deposits of water to descend from the solvent and collect at the stop cock, where they, too, were slowly drained.

When no further aqueous phase would form, a drying vessel was prepared, containing 30% (vol.) of anhydrous, non-indicating Silicon Dioxide. The entirety of the organic solution was slowly drained into this drying vessel, its threads were sealed with PTFE and a PP5 lid was threaded tightly onto the vessel and it was allowed to sit for *at minimum* one hour.

The separatory funnel was then rinsed with 200mL Distilled Water, then *thoroughly* degreased with 2-Propanol and dried with anhydrous Acetone. After no visible residual solvents were seen in the separatory funnel, the ground glass joint was sparked in a controlled, safe environment with a lighter to ignite residual solvent vapors, which then burned in a very calm manner with a small blue flame.

The separatory funnel was then set into a DIY ring stand, the stopper secured and the stop cock temporarily left open to allow any possible solvent traces and water to drain from the stop cock.

When the separatory funnel was fully clean and dry, the stopper was removed, the stop cock was closed and two small sections of an unbleached coffee filter were fitted securely into position in the ground glass joint opening. This filter was then treated with clean, dry Toluene to pre-soak the filter paper, and the organic solution was transferred carefully from the drying vessel back into the separatory funnel. The stopper was resecured.

7 PROTONATION

*It is the rigid opinion of the author and his mentor that, regarding protonation **dry Hydrogen Chloride gas must be used.***^[16]

An HCl generator was prepared by outfitting an HDPE applicator bottle with a 5/16" polyvinyl hose. This hose was then connected to a secondary HDPE applicator bottle (to serve as an inline drying chamber), and this secondary HDPE applicator bottle was then outfitted with an additional length of 5/16" poly vinyl hose, and into this hose, a glass dropper stem was connected, so that the dropper tip faced retrograde back into the hose, and the larger opening of the dropper stem faced away from the generator.



Example photograph of a DIY Hydrogen Chloride generator and in-line drying chamber.

The generator output hose was led to the Toluene via the separatory funnel joint opening, but the output hose was ideally not allowed to make direct contact with the organic solution so as to avoid catastrophic suck-back of the organic solution during the protonation.

If the chemist opts to submerge the generator hose, it is recommended to include a 5/16" check valve between the generator output hose and the glass dropper stem affixed to its tip. This is not fully necessary, but is likely the most effective way to prevent any catastrophic suckback. Others have fitted their output hose with an inverted HDPE funnel, but the author cannot attest to the effectiveness of this method, and it presents other complications when the protonation is performed directly in a separatory funnel in the manner described by this writeup.

To the inline drying chamber, there was added a significant volume of concentrated sulfuric acid, to act as a drying agent for the Hydrogen Chloride gas.

To the primary (first) bottle of the generator, there was added (approximately 30-50% vol) dried Sodium Chloride. 6mL concentrated sulfuric acid was dripped into this primary generator bottle, and evolution of Hydrogen Chloride was instantaneous.

This gas was then hand pumped, fed with an air pump, or simply allowed to evolve through the system and into the separatory funnel, whereupon precipitation of the amine's salt conjugate began rapidly in the funnel. Throughout the duration of the protonation, the separatory funnel was swirled numerous times to allow the hydrochloride salt to form, falling from the Toluene and collecting at the stop cock barrel.



Protonation sequence. 1

2

3

This particular sequence was photographed using Diethyl Ether as an organic solvent, but the general principle remains the same utilizing both Diethyl Ether or Toluene as the protonation medium/substrate. It is the opinion of the author that protonation in Diethyl Ether is probably cleaner than Toluene, but subsequent purifications and recrystallizations are very effective towards cleaning this precipitate so that any indication of which solvent was chosen will likely no longer be present.

The protonation was allowed to proceed patiently, and the solvent was repeatedly gassed with dry Hydrogen Chloride, swirled, allowed to settle and gassed further until no precipitate formed in the solvent.

After cessation of protonation, the funnel was allowed to settle in the ring stand for no less than 30 minutes or an hour.

In a very small glass jar, 15mL fresh distilled water was brought to a simmer on an electric hot plate.

The hot water was then dumped into the separatory funnel, and with the stopper still open, the amine was allowed to elute to the aqueous phase. A small drop of this aqueous phase was drained from the separatory funnel via the stop cock and tested with pH indicator paper, where it indicated acidic.

In a shallow-walled, large surface Pyrex evaporation dish, 2-propanol was added, then hit with a light gassing from the HCl generator. The aqueous phase was slowly drained into this 2-propanol, and it was quickly tested with pH paper to verify that no residual freebase had followed this first aqueous pull. If the evaporation dish tests alkaline to litmus paper after draining the first aqueous pull, it is imperative that the chemist gasses the evap dish directly with HCl, until no reaction is observed in the dish. Residual freebase in the evaporation dish can be a catastrophic loss of yield upon evaporation.

The separatory funnel was then gassed further with Hydrogen Chloride to verify no more precipitate would form. Once the Toluene remained fully clear of any precipitate formation, another small volume of Distilled Water was added to the separatory funnel, then it was stoppered, shaken vigorously, allowed to settle and its aqueous phase was drained to the evaporation dish.

Once all aqueous pulls were collected in the evaporation dish, it was hit with dry Hydrogen Chloride until no precipitation could be seen occurring in the dish, then it was doused with a generous quantity of anhydrous Acetone to assist the evaporation. Finally, the dish was again pH tested to indicate acidic, and the evaporation was allowed to complete with the dish loosely covered by an unbleached filter.

The generator and drying chamber was neutralized with Sodium Bicarbonate.

After evaporation of the protonated precipitate, the crude Methamphetamine Hydrochloride was collected with an HDPE scraping and scooping tool, and it was chopped up finely on a large glass picture frame, where it was allowed to dry fully.

AUTHORED BY THE NABP/ANAC/ASAP

COLD WATER EXTRACTION OF THE PROTONATED PRECIPITATE

Technically, it is the assertion of the author that this step is not 100% necessary for the completion of this procedure, but it is *highly recommended* to include this in the overall process for the reader, as it will greatly assist in the removal of any potential non-polar impurities which are likely to follow aqueous pulls from the protonation performed in the separatory funnel.

The protonated precipitate was transferred to a tall, cylindrical glass vessel. An HDPE filtration funnel was suspended over a clean, dry evaporation dish, and the filter in the funnel basket was soaked with distilled water before placement into the funnel. Vacuum filtration is highly advantageous here, but it is perfectly possible to perform this step with gravity filtration if fine attention to detail is given.

A salted ice bath was constructed using a tupperware container containing a mixture of 1:1(wt) crushed ice and Sodium Chloride. Anhydrous 2-Propanol was added to the bath to act as a carrier solvent, and a thermometer was optionally inserted into the ice bath, where the temperature was kept as close to -20 celsius as was possible.

A glass vessel containing cold distilled water was added to this ice bath and sealed with a threaded lid to avoid contamination. Next to this vessel, the vessel containing the protonated precipitate was also set onto the ice bath, where ~1-2mL of cold distilled water was added to it, and it was then sealed tightly and set onto the ice bath. If necessary, the vessel containing the precipitate could be intermittently removed from ice to prevent the extraction water from freezing completely solid. After some time, the filter in the funnel was again quickly rinsed with cold water from the secondary vessel on the ice bath.

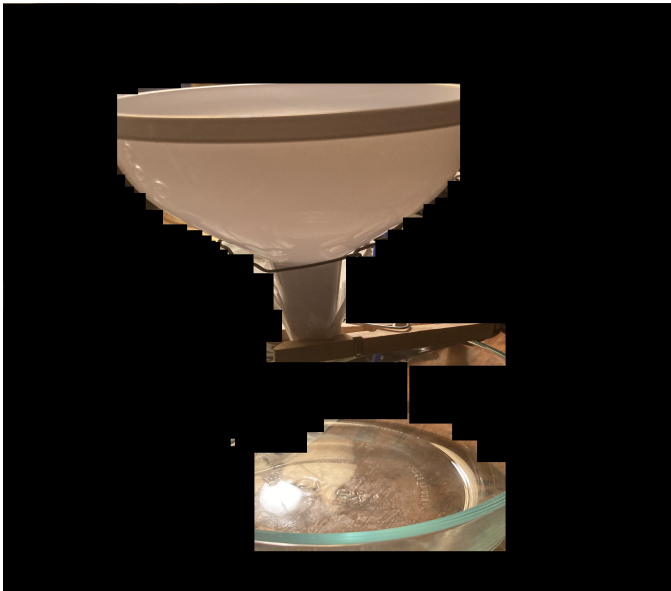
The 1-2mL from the precipitate vessel was then siphoned up using a glass dropper or HDPE pipette, and injected quickly through the filter funnel suspended over an evap dish. It was advantageous to ensure:

1. The pipette made direct tip contact with the filter paper while being injected into the evaporation dish.
2. The filter paper was kept soaked with cold water throughout. This will prevent the extraction water from clogging up or absorbing into the filter, which causes losses.

Immediately following this injection, the filter was quickly rinsed with a small volume of cold, Distilled Water. If the chemist is particularly picky about the avoidance of contamination, the filter in the funnel can be quickly switched out for a new wet filter after every single cold water injection into the evaporation dish, but such precautions are not completely necessary.

This process can be repeated as many times as desired, until the vast majority of the precipitate had been transferred in this way into the evaporation dish. The cold water was quite selective, and most non-polar impurities will have very little chance at making through

such an extraction. It is important for the chemist to be thorough at this stage, as the selectivity of cold water is so extreme, that it may not be entirely easy to extract all of the Methamphetamine this way, but the resultant crystallization will be of substantially high purity if all steps of this write-up have thus far been followed.



An example filtration funnel suspended over an evaporation dish. Heavy redaction was necessary for this photograph.



A cold water extraction vessel containing protonated precipitate is seen on a salted ice bath with 2-Propanol carrier.

After the completion of the extraction and injection into the evaporation dish, the evaporation dish was doused with approximately 1.5x-2x its liquid volume of anhydrous Acetone to spread out the evaporation dish contents and aid evaporation times, and some 2-Propanol was added, in an uncertain attempt to utilize the 2-Propanol/Water azeotrope to further accelerate the evaporation time.

The dish was loosely covered, then allowed to evaporate naturally at room temperature in a well-ventilated, dry area, and sometimes over a bed of drying agents.

RECRYSTALLIZATION

The final step of this procedure serves multiple simultaneous purposes.

1. Final removal of non-polar residual solvents and impurities.
2. Drying of the Methamphetamine Hydrochloride
3. Aesthetic appeal.

A vessel containing 2-Propanol was dried for 24 hours over non-indicating, anhydrous Silicon Dioxide, and it was then decanted to a clean, dry, glass vessel with PTFE thread sealant and a PP5 lid immediately before the recrystallization would begin.

A vessel containing anhydrous Acetone was prepared in the same way.

After the cold water extraction dish had evaporated fully, it was quickly rinsed with a small volume of anhydrous Acetone to remove significant amounts of moisture before recrystallization, and the crystalline precipitate was then collected and placed into a tall, cylindrical glass vessel with thick walls, a PP5 lid and PTFE thread sealant.

To this material, a tiny volume of dry 2-Propanol was added (1-2mL or variable depending on the starting weight).

Slowly, this vessel was brought to a boil, with the lid removed, on an electric hot plate. If, when boiling, all material had not fully dissolved, more 2-Propanol could be added, but no more was added than was necessary to dissolve the entirety of this material *while the solvent was at boiling temperature*.

A greenish aldol condensation was often observed during this, as residual Hydrogen Chloride was brought into solution with the 2-Propanol. The vessel was boiled with its lid removed until the greenish hue had disappeared almost entirely, and no more Hydrogen Chloride fumes could be seen evolving from the solvent.

Then, the temperature was slowly and gradually lowered, the lid was resecured and sealed tightly, and, once at room temperature, the vessel was set into an insulated area where it could cool as slowly as was possible. Durations of 10-24 hours were tested for the cooling time of the 2-Propanol, and 10 hours seemed quite sufficiently patient to afford excellent crystallization.



Methamphetamine Hydrochloride recrystallizing in cooling 2-Propanol.

Anhydrous Acetone was added slowly, dropwise to the vessel, but the lid was kept in place as much as was possible to avoid humidity and/or airborne contaminants from entering this hygroscopic mixture.

This Acetone addition was carried out as slowly as possible until the vessel was essentially 60% full by volume, and the lid was tightly secured, and crystallization was allowed to proceed.

After 2 hours, the vessel was set onto the electric hot plate, boiled until the crystals appeared as clean as they could be, and again, it was allowed to gradually cool back to room temperature over a period of approximately 12-24 hours.



Methamphetamine Hydrochloride recrystallizing after the gradual addition of anhydrous Acetone to the 2-Propanol recrystallization solvent.

At the end of recrystallization, an unbleached filter was washed first with distilled water, then 2-Propanol, then anhydrous Acetone, and was dried thoroughly. It was then suspended and secured in place over an evaporation dish, and a slight depression was made in the direct center of the filter paper.

The filter was soaked once more in anhydrous Acetone, then the solvent mixture from the recrystallization vessel was poured through the filter until it had been transferred completely to the dish.

The crystals were removed from the recrystallization vessel with an HDPE scooping tool, and they were set onto the filter paper, where they were rinsed with anhydrous Acetone, then quickly transferred to a fresh filter which was set onto a clean, dry, glass picture frame. They were spread loosely out, then covered with a dry filter and the residual Acetone was allowed to evaporate fully over a 6-8 hour period.

The crystals were then collected with an HDPE scooping tool, transferred to a clean, dry, sealable glass storage jar, and they were weighed as ~2.80g, representing a final yield out of ~33%.

Enantiomeric analysis was not performed.



Steam Distilled, Hydrolyzed Methamphetamine Hydrochloride, 2.80g

REFERENCES AND ACKNOWLEDGEMENTS

MATERIAL SAFETY DATA SHEETS:

(See reference numbers as they appear in the body of this publication)

1. Sodium Hydroxide	https://fscimage.fishersci.com/msds/21300.htm
2a. Toluene	https://fscimage.fishersci.com/msds/23590.htm
2b. Diethyl Ether	https://fscimage.fishersci.com/msds/90868.htm
3. Sulfuric Acid	https://fscimage.fishersci.com/msds/22350.htm
4. Sodium Bicarbonate	https://fscimage.fishersci.com/msds/20970.htm
5. Calcium Chloride	https://fscimage.fishersci.com/msds/03900.htm
6. 2-Propanol	https://fscimage.fishersci.com/msds/12090.htm
7. Acetone	https://fscimage.fishersci.com/msds/00140.htm

Various Reference Numbers:

- 8: <https://byjus.com/question-answer/give-reasons-for-the-following-hydrogen-chloride-gas-is-insoluble-in-toluene/>
- 9: <https://www.sigmaaldrich.com/US/en/product/sigald/294837>
10. <https://www.fishersci.com/shop/products/diethyl-ether-99-pure-stabilized-bht-thermo-scientific/AC123990010>
11. <https://louisville.edu/dehs/chemical-safety/chemical-safety-files/peroxide-forming-chemicals-guide>
12. <https://www.thevespiary.org/talk/index.php?topic=20675.msg54223701#msg54223701>
13. <https://pubchem.ncbi.nlm.nih.gov/compound/Methamphetamine#section=pH&fullscreen=true>
14. <https://pubchem.ncbi.nlm.nih.gov/compound/Methamphetamine#section=Color-Form&fullscreen=true>
15. [https://en.wikipedia.org/wiki/Alkaline_hydrolysis_\(body_disposal\)](https://en.wikipedia.org/wiki/Alkaline_hydrolysis_(body_disposal))
16. https://pdfhost.io/v/5NZ56uS2t_Protonation_with_Hydrogen_Chloride

For Acknowledgments:

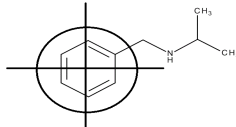
I must endlessly thank my brilliant mentor, /u/spare_me_thy_bs for his creative work pioneering and inventing the heated alkaline hydrolysis and his dedicated quest to destroy n-isopropylbenzylamine while leaving methamphetamine intact within the same vessel, at a time when nobody would admit such a thing was scientifically possible. Without his tireless and inspiring work, and his intimate mentorship, I would not have achieved a fraction of the things I have achieved today in clandestine chemistry. I wish him a lot of success in his numerous endeavors, and I look forward to learning more from him in the future.

To Dolan, thank you for helping get this whole thing started 9 months ago, when we were working in the woods and had no fucking clue what we were doing.

Dr. Snacks and Dr. 15, Rest in Peace. Fly high, forever and always.

In this cipher is my identity. I hope the feds will do what they must do to crack this code

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