The Vespiary

Main Topics => Drug Synthesis & Extraction => Topic started by: tregar on September 10, 2013, 04:29:15 PM

Title: **seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 10, 2013, 04:29:15 PM**

This seed to LSD tek took a few weeks to put together (and even more months of study), Drank lots of coffee during the research and compiling, enjoy. I have not had time to post the salting and N2 notes yet.

Modern Coupling-Reagents File attached, see 5th page for illustrations of all available couplers as their molecular self. Piglet mentions in the paper that all acid is made via peptide coupling now-a-days...a difficult method of coupling is given in note 3.

seeds-->LSD

Step 1: Defat the seeds and extract the lysergic acid amides

Step 2: Hydrolysis of extracted ergot alkaloids to Lysergic Acid

Step 3: Distillation of DEET to Diethylamine

Step 4: Coupling of LSA with Diethylamine via modern peptide coupling agents

Step 5: Cleanup

Step 6: Salting

Note 1: the mysterious "Method X" (no coupling agent needed) there is a reference in the Chem Abstracts and a patent granted, but it is mentioned only once and there are no other confirmations that it works..it uses a constructed "pipebomb" and high temperature achieved using an adjustable heating mantel with oil suitable for very high temp use to hold and heatup the pipebomb while it sits in the oil for 24--60 hours. Only included this since director of sound makes a passing reference to this, as does Fester in his book "Practical LSD production". Piglet sniggers at the Method X method, as would imagine just about everyone else (included). But I refuse to throw out the baby with the bathwater just in case there is some slight chance the method actually has some merit.

Note 2: A few comments about rotovaps

Note 3: a difficult coupling procedure

Note 4: How to setup and use an N2 tank

Step 1: defat the seeds and extract the lysergic acid amides

A--500g yields 1.9g of miscellaneous ergot alkaloids (director of sound)
B--850g yields 2.5g of miscellaneous ergot alkaloids (makoeyes)
C--Alkaloid extraction (short method) by Michael Valentine Smith--yields yellow oil
D--Alkaloid extraction from Otto Snow, "LSD"
E--Alkaloid extraction by Moxley/Webster--yields light colored syrup: glows in blacklight
F--Alkaloid extraction per Uncle Fester

A--Per director of sound:

director of sound, 2009:

I talked to my FOAF today and she said that the 300mg of lysergic acid used came from 500g of woodrose seeds (aprx 4000 seeds, 8 seeds = 1.028g). The total LAA's that came from that 500g was 1.88g which was converted to 487mg lysergic acid in the hydrolysis.

No epimerization process was used so she still has ISO-LAA's in the mother liquor that could give her another 200mg or so of lysergic acid. my FOAF hopes to grow a rye field accidentaly contaminated with ergotas ergot contains 2%+ LAA's rather then .3% like the other seeds. she hopes to find a few dozen ergot kernels this fall that she will fruit on agar in the spring and make several gallons of liquid culture to accidentally spray on her rye field when the rye is flowering towards late june/early july...she said that 1 acre of contaminated rye could yield 225--400g LAA's after
extraction...that could give her roughly 60-120g lysergic acid--that means 45--90g of LSD-25.

director of sound, Nov 2010:

50-100g wouldnt make alot and you'd have to do it on a micro scale with 25-50ml glassware because after you convert the LSA to lysergic acid you wouldnt have too much to work with. youd get maby 350-400mg LSA from 100g HBWR and depending on how you convert that it could end up being 100-250mg lysergic acid.... it could be done though. just get all your ducks in a row first before attempting anything otherwise you'll loose product by the hour once its extracted.

director of sound, Oct 2010:
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actual fresh hawaiian seeds, i have a friend on maui that can aquire them for me every now and then. when working with something like lysergamides from seeds (other than ergot) a larger scale is much more feasable when you think of it. first if you were to extract say 50g of seeds you would only get about 200mg of mixed lysergamides that on hydrolysis to lysergic acid would only give about 60mg mabey a little more if you were to make the hydrazide (150mg +/- 25). so you wouldnt be able to do very much with 150mg or less so if you can get your hands on half a kg or better you might be able to make something from it.

the average hawaiian baby wood rose seed weighs 125mg so there are 8 seeds

per a gram, go to a site like that shamanjungle bullshit place where they sell by seed count, 500 seeds would be a little over 60g for about $16 (not hawaiian seeds though) its gonna cost you $125+ to get 500g of seeds that might give you 1.5-2g of lysergamides to play with. my suggestion is that if you are gonna go as far as to mabey attempt this, it dosent cost that much to go to hawaii in September. make some friends, find a big old hbwr vine and ship seeds back in coffee bags. just make sure they are the foil lined ones and vacuum seal them before you ship. a few weeks squating on the pipeline for free will let you scroung up several KG pretty much for free (well you plane ticket S&H at least). so now when you fly home you will have 10, 20 whatever kg you gathered in your time there waiting for you. now you have something to work with, if you were to go the hydrazide and POCl3 route 10kg might get you 10-15g..... now would that be worth a $500 plane ticket?

director of sound, Nov 2010:
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as for bromocriptine, if you think you can pull off a grinard, go right ahead as i found 2.5mg pills in a 30ct for $24 online. ill have to find the link again though but it looked like it was OTC and nothing said an rx was needed to buy it.

[Here we go.....]

Per director of sound, original forum 2009:
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this will work for those of you that have rye fields near by too.

1. saturate the seed/ergot powder with a saturated solution of methanol and tartaric or maleic acid and let it soak for a day,

2. add more methanol as the seeds soak it up.

3. drain the methanol off and run 4 times the amount collected more.

4. drain that then press the rest of the methanol from the seeds

5. collect and evap it all down to a greenish-yellow syrup/goo.

6. extract the goo with enough diethyl ether (starter fluid if you have to) to dissolve the goo and 500ml water per each 250ml of ether.

7. keep your ether it still has useful Iso-clavine/Iso-ergoline alkaloids that can be put through hydrolysis...

8. the water portion should be extracted a few more times with new ether or another NP solvent like hexane (bestine rubber cement remover)

9. then the water made basic with strong ammonia

10. and extracted X3 with ether.

11. the ether is evaporated

12. and the residue dissolved in a minimum ammount of a saturated solution of methanol and tartaric acid.

13. then add ether to the solution until it stays cloudy and put it in the freezer overnight.

14. there will be Xtals of ergot/clavine alkaloids (mixed lysergic acid amides (LAA's) waiting there for you.

15. now the ether you saved from your 'de-fat' is evaporated

16. and the residue is dissolved in methanol and made basic with a saturated sodium carbonate or bicarbonate solution

17. then extracted with ether

18. which is then evaporated

19. and dissolved in methanol/tartaric acid again

20. and forced to crystalize with ether in the fridge overnight.

I have more steps to convert these alkaloids into D-lysergic acid monohydrate ((+)-lysergic acid monohydrate) very easily but from 10g mixed LAA's you might get 3-5g D-LA.

conceivably if you started with 1kg of hawaiian baby woodrose seeds you could end up with close to 2.5-3g LAA's ergot on the other hand will give you around 15g/kg.

Per director of sound, secondary forum, Oct 2010:

I have read a few synths (with out any real data though) that use a solution of lysergic acid and diethylamine or another tritary amine in an alcohol sealed in a 'pipebomb' fashion that supposedly works, ill have to dig up the data i have on that and post it later. but for now here is some stuff that might help. i have performed both and can assure you that they work.

[editors note: director of sound is referring to method x (which uses only LSA and diethylamine with no coupling agent whatsoever) refer to Note 1 at end of paper.]

Production of Pure D-lysergic acid monohydrate from LAA/Ergine containing plant sources:

All steps to be done under a red photographic dark room light.

1.*** 8 kg of HBWR seeds are ground to a fine powder with the assistance of dry ice or liquid nitrogen.

2. to the seeds is added a chilled (30*F) acidic methanol (80% MeOH/DH2O) solution of tartaric acid (saturated). the ammount needed is just enough to cover the powder with 1cm of fluid and is allowed to stand cold for 24 hours in the dark. more acidic methanol may be added as the seed powder absorbs it.

3.*** after 24 hours the methanol is removed by vaccum to complete dryness.

4. the seeds are then made into a slurry with DMF/DCM and loaded into a large column,

5. the DMF/DCM is allowed to drain through and more is added until test drops evapurate with no residue.

6. save the DMF/DCM filtate.

7.*** the residual DMF/DCM is then removed by vaccum

8. and the seed powder loaded back into the column and filled with chilled MeOH and allowed to stand for 2hr before draining.

9. this is repeated 4 times <----->

10. and the methanol extracts combined, filtered and reduced under vaccum to a small volume.

11. cold ether is added to the methanol solution untill cloudiness does not dispell and is placed in a very cold fridge for the crystals to set overnight.

12. save this solution. this should afford 6-10g mixed LAA's as the tartarate salt.

13.*** the crystals are dissolved in 200ml MeOH with 11g KOH and the methanol is removed immediately by vacuum as soon as the amide is dissolved.

14. the residue is dissolved in 200ml 8% KOH in DH2O and heated with a stream of nitrogen passed through it, ammonia gas will be evolved and titrated with HCL to follow the reaction (HCl fumes profusely in the presence of ammonia gas).

15. as soon as it is complete, cool the solution and slowly add 2 or 3N sulfuric acid until a PH of 3 is obtained, this will result in precipitation of crude D-lysergic acid monohydrate. save the solution.

16.*** the crystals should be dissolved in 200ml abs.EtOH containing a few ml of strong ammonia (or gassed with anhydrous ammonia untill the solution has gained 3g)

17. the solids that do not dissolve within 1 hr are inorganic and can be discarded.

18. filter the solution through a column packed with 3in of 100 grit basic alumina/silica wetted with anhydrous EtOH.

19.*** the alcohol solution is again acidified with 2-3N sulfuric acid untill a PH of 3 is obtained and is placed in the fridge overnight to crystalize.

20. the crystals are filtered out and dried in a vaccume desiccator or centerfuge. this will yeild 4g to 8g D-lysergic acid monohydrate.

21.*** there is still Iso-lysergic acid and amides in the solutions you saved.

22. remove the solvent in all three by vaccum using only heat from a hot water bath under 80*C.

23. dissolve the residues in a mixture of EtOH/MeOH 3:1 and combine.

24. basify with a saturated NaHCO3 solution and extract with ether or chloroform,

25. remove the solvent by vacuum

26. and process the residue through steps [13-20] for additional yield.

27.*** dissolve the crystals in abs. EtOH and THF 2:3 (alt use benzene and chloroform 3:1) and process through a chromatography column following the blue band with a weak long wave UV light.

28. collect that fraction and remove the solvent by vaccum to get pure D-Lysergic acid monohydrate.

29. the D-LA should be cold stored -0*C in the absence of light and oxygen.

director of sound, 2009:

In the post I made it's not really a true titration but rather you hold a beaker of conc. HCL in the off gas stream coming from the refluxing amides in water and KOH.
The conc. HCL will fume profusley in the presence of ammonia gas so when the fuming
stops the rxn is done. The reaction takes usually around 1.5--2 hr for 10g of the amide.
Only a steam bath should be used, a hot water bath of no more than 86 C (186 F) should be
used, also try to keep the temp of the refluxing solution at least 10 c (18 F) below that.

jon responded:
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director of sound your technique is superb.the only weakpoint is the fact that hydrolysis of lysergamides yeilds 35% theoretical lysergic acid and the pocl3 process 77% not too great.
hydrazine is dangerous to handle but yeilds 70+% of a stable intermediate. a third option would be to chromatigraphically isolate ergine from the clavines along with the carbinolamides ergonovine and subject them to a milder hydrolysis in alcohol using a weaker base which would yeild higher (ergine) then alcoholysis in methanolic hcl would yeild the d-epimer in quantitaive yeilds this could be then reacted with diethylamine in a bomb to yeild 60% or reacted with trimethyl amuminum to form an activated diethylamide complex which would yeild quantitatively. other options include esterification with pentachlorophenol and reaction with diethylamine at stp to give quatitavie yeilds. a bit more tedious but the yeilds are higher by a factor of 2.

director of sound, Oct 2010:
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i also dreamed up and idea for a large scale vacuum soxlet type extractor to use when extracting large ammounts of plant material. it would cut down the needed 50gal or so of solvent to about 10L mabey less and the whole thing under an aspirator with a hot water bath for heat would avoid the high temps that would destroy most lysergamides. ill have to post up the schematics when im done with

them.

director of sound, Oct 2010:
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all i posted was a novel rout to lysergic acid and diethylamine, you could use one of many methods after that to make your fluff. SO3, trichloroacetic anhydride, lithium lysergate, PyBOP, POCl3, grignard its your choice baised on what you have available and what kind of lab experience you have.

im still working on the vacuum soxlet schematics now. but there would only be one custom piece that would have to be made. that would consist of a large hopper/tank made from stainless steel tubing (30x80cm about a 57L capacity) fitted with a suitable head that has a valve and fritted discs for filters. it would essentialy be a manual soxlet so it would have to be constantly watched while running but it would allow you to process close to 100lbs of ergot/seeds at a time using only 5L or so of a chloroform/methanol/ammonia mix. with vacuum from an aspirator applied you would only need the heat from a hot water bath to drive the distillation.

as for the labware you would need: at least a 20L 2 or 3 neck flask, 400mm graham/friedrich condenser, 400mm allihn condencer, vacuum adapter, tubing and water aspirator vacuum probally totaling close to $300 and the hopper/tank i could see coming close to $200 so mabey $500-600 over all which is chump change if you are gonna process that much material and make LSD from it. just add a heating mantle, 500ml flask and some rubber septums and you would also have every thing needed to preform the synth its self.

B--Per Makoeyes:

Makoeyes method of practical LSD production:

Step 1

A/B extraction of HBWR seeds (850g)

1. Wash/dry seeds...Powder 850g of seeds in a clean blender or coffee grinder. Air dry
   the resulting powder.

2. Submerge powder in VM&P Naptha (petroleum ether) in a flask, stopper flask, shake vigorusely,
   let sit for 2 days in a dark place shaking every once in a while.

3. Vacuum filter, save/dry resulting mush (dry in vacuum)

4. Submerge powder in anhydrous MeOH in a flask, stopper flask, let sit 2 days in a dark place
   shaking every once in a while.

5. Vacuum filter, save the MeOH this time. Remove solvent in vacuum.

This is the gunk that morons across the web will debate all day, is it LSD? (no) is it lsa?
(kinda) Is it an amine or an amide? (amide) Is it active? (kinda) It made my

friend vomit!
(your friend is a dick) It must be cyanide!! (you're a dick)...on aleged toxicity: to make along
argument null, yes there is something like cyanide in the seed, is it cyanide?...no, it has some
cyanogenic glycosides in the seed coat, alot of seeds (apple) do. will it kill me?...(no) will it
make me sick? (yeah, probably, that's why we extract) what if I'm lazy? (don't eat a lb of raw
plant material, you will be fine)

% of Total alkaloid % dry seed weight
Ergine 22.68, 0.136
Isoergine 31.36, 0.188
Ergometrine 8.20, 0.049
Lys alpha-OH-ethylamide 5.79, 0.035
IsoLys. 3.98, 0.024

Well, since we started with HBWR, there is some LSA-111 present, not alot, (~.03%) most of it is
ergot-type alkaloids, many of these are amides (meaning they are derivatives) of LSA, after you
split them with water (hydrolyze) your left with LSA-111 and ISO-LSA-111.

Lysergic acid means the organic acid you get when you split ergot. The aformentioned crap yielded
from the procedure above will be gunky and off color...if you proceed with it, you won't yield a
whole lot. But, maybe you don't want alot, maybe you just wanted enough to trip on, whatever,
sounds like a waste of money, and forget about selling it. I suggest purification of this rough
extraction for better yields down the line. JiJo, SiCo and the like...(junk in Junk out Shit in
Shit out) "You can't piss into a Mr. Coffee and get "Tasters Choice"--commentary on Waterworld
(stupid movie...I kinda liked it...SHIT!...)

C--per Michael Valentine Smith, "LSD"

Alkaloid Extraction (short method)

Method 2

1. Add 100ml petroleum ether to 100g finely ground seeds and let soak about 2 days
2. Filter
3. Discard petroleum ether and let seeds dry
4. Add 100ml methanol to the seeds and let soak about 2 days.
5. Filter,
6. Repeat extraction with another 100ml methanol
7. and evaporate in vacuum the combined methanol extracts.
8. The residual yellow oil contains the alkaloids

Method 1

1. Finely grind seeds (preferably woodrose) and add NaHCO3
2. Extract with ethyl acetate by soaking about one day
3. Filter
4. and extract the ethyl acetate with tartaric acid solution.
5. Basify the extract with NaHCO3
6. and extract it with ethyl acetate.
7. Dry and evaporate in vacuum the ethyl acetate to get the alkaloids.
8. Repeat this procedure on the seeds until no more residue is obtained.

D--Per Otto Snow, "LSD"

Extraction of Ergoline Alkaloids from seeds

Method A

1. Pulverized seeds (100 grams) must be defatted before extraction of alkaloids.
2. Naptha or petroleum ether are suitable solvents for fat extraction of the seeds.
3. The seeds can be refluxed in the solvent or they can be refluxed in a Soxhlet extractor.
4. The seed mash is then filtered from the solvent.
5. Total extraction of fats is accomplished when new solvent extract leaves no greasy residue on
   evaporation.
6. The seed mush is then allowed to dry of solvent,
7. mixed with 500 mL of 10% ammonium hydroxide (strong ammonia water)
8. and extracted with ether or appropriate solvent.
9. Evaporation of the solvent leaves the alkaloids. Reference (Genest 1965)

Method B

1. 100 grams of pulverized seeds is mixed with 50 grams of sodium bicarbonate and 100 mL of water.
2. 100 grams of anhydrous soldium sulfate are mixed to leave the mass dry and granular.
3. The mass is extracted x 3 times with one liter of ethyl acetate.
4. The ethyl acetate solutions are combined and evaporated to leave the alkaloid residue.
   Reference: (Marderosian 1966)

All extractions should be done under inert atmosphere. Ergot alkaloids will decompose in light,
heat and air. Tartrate and maleate salts are less susceptible to destruction.

E--Per Moxley/Webster, lysergic acid amides syrup extraction from seeds:

We have slowly been building up a stock of seeds of the "Heavenly Blue" morning glory, Ipomoea violacea, which grows widely in this area of Mexico. A short drive out of the city in any direction leads to the discovery of some extensive stand of the plant, and we look for groups of boys playing and gather them 'round for a short lesson on economic realities. It seems that we offer hard pesos for anyone who will gather these funny little black seeds for us, and be here on this spot in

exactly one week. Returning after a week we usually find only one or two of the boys has taken us seriously and actually collected even a coffee-can full. But when the scales come out of the back of the pickup, and hard cash changes hands for what would seem to all excepting gringos a worthless commodity, eyes widen with dreams of transistor radios. Mexico is a tragically poor nation, and our harvest of seeds has, upon last inventory, attained rather amazing levels with very little expense.

A second goal for our work would be to try to obtain pure lysergic acid from the seed extracts by chemical hydrolysis. A rather large industry had evolved since the turn of the century which produced the alkaloid ergotamine from a laborious process of growing the ergot fungus on rye grass.

Ergotamine had been a widely used lysergic acid alkaloid for decades, but recently other derivatives of lysergic acid had been found to be more useful, and to produce them, the ergotamine yield from ergot was first hydrolyzed to lysergic acid, then appropriately reacted with various amines or other compounds.

It was work of this type that had led Hofmann to synthesize LSD by reacting pure lysergic acid, via an intermediate, with diethylamine. We intended to evaluate the possibility that morning glory seeds might someday provide an alternate, or even better source for lysergic acid than the ergot/rye process.

We would at the same time be determining if it were possible for an underground chemist, using morning glory seeds instead of ergotamine (which was tightly controlled and difficult to obtain), might produce small amounts of LSD with very little risk. I say small amounts, because the alkaloid content of morning glory seeds had been assessed at barely 0.06%, and assuming normal losses and other factors it would therefore be necessary to process perhaps a hundred kilos of seeds or more to produce even a gram of LSD. Still, due to the vanishingly small effective dose of LSD, such a process was far more a practical possibility than that necessitated by the required minimum dose of peyote extract, more than two thousand fold that of LSD.

The extract of Ipomoea violacea that we had prepared radiated power, just sitting there in its flask. A light amber, odorless syrup which, in the darkened laboratory fluoresced brilliantly blue under ultraviolet light.

it was an extreme contrast with the series of messy, difficult to purify, dark-colored and discouraging volumes of intermediate sludge we had treated, and brought to mind the Curies and their arduous separation of a few tiny crystals of glowing radium from a mountain of pitchblende.

The difficulties had, however, taught us much about ways in which we would modify our processes for future work. As for the extract, the following day would see the first test of its activity, with myself as the guinea-pig.

The morning-glory extract provided not a nasty surprise, but a powerful surprise none the less. It was by far the most powerful experience I had yet encountered. Perhaps the methods of our extraction had yielded a product more representative of the shaman's recipe than the preparations obtained by other investigators, who reported only modest psychedelic effects.

The experience of that day was hardly modest, from the beginning moments it

certainly did not fail to inspire reverence and humility, no matter what the direction to which I managed to guide it. The colors and geometric patterns, the rippling waves so often seen in watching clouds in the sky, the slowing of time and other typical effects so frequently described in the literature had some time ago become only minor and unattended aspects of psychedelic experience for me. Certainly, I still noticed these effects, if I took the trouble to pay attention to them. But the psychedelic experience had become for me far more an arena for the Herculean task of attempting to achieve the truly original perspective for viewing the fundamental questions that man has posed since the beginning of time. It was the task of freeing oneself completely from preconceptions, from habits of thinking that affected the outcome of seeking in unknown and unconscious ways. And of course, it was paradoxical, if not impossible to erase these filters of comprehension completely. To a very significant extent, comprehension consisted of these filters. Nevertheless, the psychedelic experience seemed to go quite a good distance in providing this ability, if one were ready to use it. Particularly the experience of that day.

Ingesting ~2mL of the light colored syrup from the vial containing the extracted lysergic acid amides was obtained a full on psychedelic experience remarked equally as strong as high dose LSD trips I had experienced. The syrup in the vial fluoresced bright blue under UV, which is characteristic of simple lysergic acid amides.

1. The procedure involved extracting finely powdered Heavenly Blue Morning Glories w/ Ethanol.

2. Most ethanol was evaporated off in a rotovap,

3. added a bit more water,

4. basified the messy solution w/ ammonia (to make sure alkaloids weren't in the salt form)

5. & extracted 3x w/ DCM (dichloromethane)

6. combined extracts were extracted with water containing 5% tartaric acid.

7. The alkaloidal-tartarate solution was basified w/ ammonia

8. and extracted w/ dichloromethane.

9. Evaporate DCM leaving a light coloured syrup which contains all the lysergic acid amides from the seeds, that fluoresced bright blue under blacklight.

F--per Uncle Fester:

1. Defating is a very important step in the isolation of pure alkaloid. The fats and oils present
in the crop must be removed because if they were left in, a tenacious emulsion would form during
the extraction of the alkaloid, and you could forget about ever getting even close to a pure
amide extract.

2. Defatting can be done with any one of several very common and easily available solvents. For a
200 lb crop, one can count on using at least one, and possible two 55 gallon drums of solvent.
The defatting can be done with either hexane, petroleum ether (not ethyl ether) mineral spirits
or naptha.

3. The preferred procedure for small scale extractions is to put the ground-up, solvent-soaked
crop into a burette, and then keep dripping fresh solvent onto the top of the material until
the solvent coming out at the bottom of the burette does not leave a grease stain on filter
paper when the solvent dries.

4. This is easily scaled up for one 200 pound crop by replacing the burette with clean pipes
about 4 feet long, with suitable valves and filters at the bottom to prevent everything from
falling out.

5. When all the fasts have been removed from the crop, the best procedrue is to evaporate the
remaining defatting solvent from the crop under a vacuum. This is not practical for a large
crop, so letting the reaminder drip out of the bed over a period of a few hours is called for.

6. With the fats removed, the ergot alkaloids can be extracted from the crop. Note here the word
alkaloid. This is the key to all variations of the extraction procedure. There is a piperidine
nitrogen atom in the lysergic portion of these molecules that possesses basic properties
similar to ammonia and amines. This atom allows the lysergic molecules to form salts with acids
and also causes the solubility characteristics of the molecule to change depending upon whether
the molecule is in acid or basic solution. It further allows the lysergic amides, including
LSD, to form crystals from solution.

7. The naturally occurring ergot alkaloids come in isomeric pairs. The carboxyl grouping of the
amides can be in the desired and very physiologically active configuration. Or they can be in
the inactive "iso" configuration. The amides in the "iso" configuration will not form
crystalline precipitates with tartaric or malic acid. More on this later.

8. The lysergic amides as found in our crop are tied up in the plant material in association
with acidic substances. To get the amides to extract out in a solvent, this salt

must be
free-based. There are two preferred solvent and basing agent combinations. Choice number one
is used in the USP procedure. This combination is ammonia as the free-basing agent in a solvent
of chloroform. The other preferred combination was used extensively in Europe. This combination
used MgO (magnesia) as the basing agent with a solvent of ethyl ether or benzene.

9. The USP method allows the much simpler procedure that follows: The extraction solvent is made
up by adding one-tenth gallon strong ammonia (28% NH3OH; 56% NH4OH) to nine-tenths gallon
methanol. After mixing, this is added to nine gallons of chloroform to give 10 gallons of
extraction solvent. The use of methanol is necessary because without it the ammonia does not
mix into the chloroform. Instead, it would float on top of the chloroform giving an
unhomogenous mixture.

10. The extraction is done by trickling this extraction solvent into the top of the bed of crop,
allowing it to flow downward through the crop, and collecting the extract as it flows out
the bottom of the pipe. This extract must be protected from light to prevent its destruction.
The extraction of a 200 pound crop requires about 150 gallons of solvent.

11. One can monitor the extraction by catching a little bit of the solvent coming out the bottom
of the pipes in a watch glass, and shining a black light upon it in a darkened room. The
lysergic amides in the crop fluoresce a bluish color. When this color no longer appears in
the extract, the extraction is complete.

12. Next, the approximately 150 gallons of solvent must be evaporated down to a more convenient
amount. The evaporation is continued until the extraction solvent has been reduced to one-
fifteenth its original volume. For the 200-pound crop, the 150 gallons of extraction solvent
has been reduced to 10 gallons.

13. When the chloroform has been reduced to 1/15th of its original volume, it must be diluted
with ether. The reason for this is that the next step is extraction of the ergot alkaloids
into a TARTARIC-ACID solution, and it has been found that this is very difficult from pure
chloroform. When the solution is predominantly ether, the transfer of the alkaloids into

the tartaric-acid solution can be done efficiently. For the drum-sized batch, add 30 gallons
of ether and two gallons of alcohol. Similarly, for smaller batches add three volumes of
ether and a little alcohol.

14. The serious experimenter may wish to try substituting toluene for ether, since it is not
now on the mandatory snitch list. Ether starting fluid would work fine for smaller batches.

15. The alkaloids are next extracted out of the ether solution into decimolar (15 grams per liter)
tartaric acid in water. The alkaloids form a salt with the tartaric acid that is solube in
water, and leave the extraneous plant compounds in the ether.

16. This extraction should be done x 4 times with a volume of tartaric-acid solution tht is one
seventh the volume of the ether solution. For example, with about 40 gallons of ether solution
in a drum, extract with about 6 gallons of tartaric acid solution x 4 times. This means a fresh
6 gallons on each extraction. If a stubborn emulsion forms, the addition of a little alcohol
to the mix will break it.

17. Tartaric acid is the preferred acid for this extraction because the tartaric acid salt of the
alkaloid is relatively stable in light. A .2N solution of sulfuric acid can be used instead
if precautions are taken to protect the solution from exposure to light. This method may be
preferable because it can be a hassle to buy tartaric acid sometimes.

18. The tartaric-acid solution containing the alkaloids should now be free-based, preferably with
ammonia. The ammonia should be added slowly with vigorous stirring until the pH of the solution
reaches 8 to 8.5. A higher pH must be avoided, since at these pHs racemization to the inactive
iso form of lysergic occurs. This conversion is an equilibrium reaction in which only partial
reversal to the iso form will occur. Heating is also needed to get the reaction moving, but
it should be avoided.

19. The free-based alkaloids can now be extracted out of the water solution into ether. The
extraction should be done x 4 times, each time with a volume of ether 1/4 that of the water
solution. The combined extracts should be dried over some magnesium sulfate previously wetted
with ether to prevent it from absorbing alkaloid during the drying process.

20. Finally, the ether is evaporated away under a vacuum to yield a residue of fairly pure alkaloids. The alkaloids in this form are very fragile, and must be immediately transferred
to a freezer for storage.

21. Now as we mentioned previously, the lysergic amides occur in pairs in nature. This extraction
procedure was designed to isolate the "active" members of the pairs and leave behind the
inactive "iso" alkaloid. Hunting for this "iso" material should double one's yield of
product whether one is extracting ergot or seeds.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 10, 2013, 04:30:33 PM**

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step 2: Hydrolysis of extracted ergot alkaloids

A--500g seeds yields 1.9g of alkaloids hydrolyzed to 300mg of Lysergic Acid (director of sound)
B--850g seeds yields 2.5g of alkaloids hydrolyzed to 1g of Lysergic Acid (makoeyes)
C--20g ergotamine yields 9g maximum of Lysergic Acid (Michael Valentine Smith)
D--cleave the amides to lysergic acid--Uncle Fester

A--per director of sound, 2009:
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1. The Lysergic Acid Amide crystals are dissolved in 200ml MeOH with 11g KOH and the methanol
is removed immediately by vacuum as soon as the amide is dissolved.

2. The residue is dissolved in 200ml 8% KOH in DH20 and heated with a stream of nitrogen
passed through it,

3. ammonia gas will be evolved and titrated with HCL to follow the reaction (HCL fumes profusely
in the presence of ammonia gas). AS soon as it is complete, cool the solution and slowly
add 2 or 3N sulfuric acid until a PH of 3 is obtained, this will result in precipitation of
crude D-Lysergic acid monohydrate. Save this solution.

4. The crystals should be dissolved in 200ml abs. EtOH containing a few ml of strong ammonia
(or gassed with anhydrous ammonia until the solution has gained 3g) the solids

that do not
dissolve within 1 hr are inorganic and can be discarded. Fiter the solution through a column
packed with 3in of 100 grit basic alumina/silica wetted with anhydrous EtOH.

5. The alcohol solution is again acidified with 2-3N sulfuric acid until a PH of 3 is obtained
and is placed in the fridge overnight to crystallize. The crystals are filtered out and dried
in a vaccum desicator or centerfuge. This will yield 4g to 8g D-lysergic acid monohydrate.

6. There is still Iso-lysergic acid and amides in the solution you saved. Remove the solvent in
all three by vaccum using only heat from a hot water bath under 80 C. Dissolve the residues
in a mixture of EtOH/MeOH 3:1 and combine. Basify with a saturated NaHCO3 solution and extract
with ether or chloroform, remove the solvent by vacuum and process the residue through steps
1--5 for additional yield.

7. Dissolve the crystals in abs. EtOH and THF 2:3 and process through a chromatography column
following the blue band with a weak long wave UV light. Collect the fraction and remove
the solvent by vaccum to get pure D-Lysergic acid monohydrate.

8. The D-LA should be cold stored -0 C (32 F) in the absence of light and oxygen.

B--Per Makoeyes:

Makoeyes method of practical LSD production:

Step 2

Hydrolysis of extracted Ergot alkaloids:

1. Dissolve 2.5g of the alkaloid in 22 ml of 1M methanolic KOH solution (this is made
by dissolving 1.4g of KOH pellets in 25ml of dry methanol).

2. In a 1 L evaporation flask (heavy walled construction) immediately evaporate the methanol off.

3. Add 40 ml of 8% aqueous (water) KOH solution to the residue and boil for one hour under a slow
stream of nitrogen that is allowed to flow through a small orifice for exhausting purposes.

4. Cool, acidify with dilute sulfuric acid, and shake in a sep funnel with 1 L of dry ether.
Keep the lower aqueous layer.

5. Vacuum filter. Wash the precipitate with 2ml of dilute sulfuric acid.

6. This is LSA and iso-LSA (about 20% iso) time to break out the column. I'm not gonna go into
how to operate a column here, your big boys, there will be 2 major fluorescent bands (oh yeah
you need a long wave black light) don't leave it on the whole time or anything, just check
your bands. The greater is LSA, the lesser is Iso-LSA. Collect them both in seperate collection
flasks, you can convert the Iso-LSA to LSA or just discard it, whatever, point is you want
LSA for the rxn, not it's isomer.

C--Per Michael Valentine Smith, LSD:

Ergot Alkaloid Hydrolysis

1. Dissolve 20g of the alkaloid (e.g., ergotamine) in 200ml 1M KOH in methanol (this is made
by dissolving 56g KOH pellets in 1L 100% methanol)

2. In a 1L heavy walled vacuum flask, evaporate in vacuum the methanol at room temperature.

3. To prevent the solution from cooling, and thus greatly prolonging the evaporation time,
put the flask in a pan of water kept at room temperature by gentle heating or by
running wqrm water through it.

4. Add 400 ml 8% KOH in water to the residue and boil for one hour (under N2 if possible,
this can be done by filling the flask with a N2 stream and loosely stoppering or by
allowing a gentle stream of N2 to low through during heating.)

5. Cool, acidify with dilute sulfuric acid and shake in a separatory funnel with 1 L ether.

6. Discard the upper ether layer and filter with vacuum the aqueous suspension of lysergic acid.

7. Wash precipitate with 20ml dilute sulfuric acid.

8. It is unnecessary to purify, but this can be done as follows: dissolve 9g in 20ml

NH4OH, filter and concentrate in vacuum at room temp to precipitate. After filtering,
the grey crystals can be further purified by dissolving in boiling water and cooling
in an ice bath to precipitate.

D--per Uncle Fester:

Method number one uses easily available KOH and methanol to cleave the amides to lysergic
acid.

Method One

1. 10 grams of lysergic amides extracted from the crops are dissolved in 200 ml of methanol containing 11 grams KOH.

2. The methanol is then removed at once by distillation under a vacuum.

3. To the residue in the flask, then add 200 ml of an 8% solution of KOH in water.

4. This mixture should then be heated on a steam bath for a few hours under a nitrogen atmosphere.

5. Next, the reaction mixture should be cooled, and sulfuric acid added to it slowly until it
reaches pH 3.

6. This results in the precipitation of crude Lysergic Acid having a dark color.

7. The beaker should be allowed to sit overnight in the fridge to let the crystals fully form.

8. Then the crystals of crude D-lysergic acid should be filtered out and rinsed with a little ether.

9. These crude crystals should be transferred to a beaker, and taken up in solution with two
200 ml portions of ethyl alcohol containing a few mls of strong ammonia.

10. The residue which does not dissolve within an hour of stirring is inorganic, and can be
discarded.

11. Filtration or letting the beaker sit to settle the sludge followed by decantation is the way
to remove this insoluble material.

12. The alcohol solution of Lysergic Acid should next be acidified to roughly pH 3 using diluted
sulfuric acid once again. 2 or 3N sulfuric acid is roughly the proper range of dilution of
the sulfuric acid.

13. The ph of the solution is best tracked using ph papers which have been moistened with some
water. Then a glass rod is dipped into the lysergic acid solution, and a bit put onto the
indicator strip of the ph paper. Meters don't work well in alcohol solution.

14. The crystals of lysergic acid will form while the beaker is sitting in the fridge

overnight.

15. Then filter or centrifuge, and rinse with some ether.

16. This yields about 3 or 3.5 grams lysergic acid.

17. It should be dried in a vacuum dessicator, then stored in the freezer. The lysergic acid
even after vacuum-drying holds one molecule of water as part of the crystal structure.
This is not a problem if the method given in Chapter 6 is used. Other synthesis methods
require the removal of this water of crystallization, and it is tough. A vacuum of 2 mm Hg
and a temperature of 140 C is needed to remove it. Such methods are best avoided if possible.

18. To increase the yield, it would be worthwhile to pull out the isolysergic acid and convert it
to lysergic acid. The isolysergic acid should still be in the filtered mother liquor from
paragraph two of this section. If this ph 3 solution is based with bicarb and then extracted
with ether or chloroform, the isolysergic acid should extract out. Removal of this solvent
under a vacuum followed by another heating in alcohol solution containing KOH should then
give a fresh portion of D-lysergic acid. Then add water and cool the solution and slowly
add sulfuric acid solution to ph 3. Remove the alcohol under a vacuum, but leave a good
portion of the water remaining. Then chill overnight in the fridge and collect the crude
crystals.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 10, 2013, 04:31:53 PM**

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step 3: Distillation of DEET to Diethylamine

A--175ml of 98.1% commerical grade mixed with 40ml of 70% EtOH then distilled yielded 57ml
of freebase diethylamine (director of sound)

B--yield of 1g (makoeyes)

A--Per Director of Sound, original forum 2009:

OTC Hydrolysis of N,N-diethyl-meta-toluamide (DEET) to Diethylamine and M-toluic acid

1. To a 250ml autoclavable media bottle is added in order, 175ml N,N-diethyl-meta-toluamide (98.11% comerc grade, $15) followed by 40ml 70% EtOH.

2. The media bottle is capped and vigorusly shaken for one minute.

3. The EtOH and DEET should now be a homogenous mixture.

4. To this solution is added 40g NaOH and once again capped. the media bottle containing the mix is now sealed and heated in a hot water bath (crock pot set on low) added to the bath at 55*C (125*F) ending at 70*C (160*F) periodicly agitating for the legnth of time it takes to compleatly desolve the NaOH plus 1.5hr.

5. The solution should now be a deep yellow color

6. The crock pot is turned off and allowed to cool to RT.

7. It is removed from the bath and allowed to stand at RT for one day minimum.

8. The bottle will then be carefuly opened after cooling in the refridgerator for 1hr.

9. Observed was the strong smell of diethylamine, no longer did it smell of DEET and EtOH.

10. The solution is fractionaly distilled (dont forget boiling chips or mixing bar, this stuff likes to bump)

11. collecting the fraction along a 9 degree tempeture arc centered on 55*C (51-59*C).

12. the solution may solidify twords the end of the distilation and a small ammount of DH2O can be added to liquify the waxy mass of M-toluic acid and NaOH.

13. total yeild form aprx 225ml of solution was 57ml of crystal clear diethylamine in freebase form. 25.3%

Notes:

* MeOH can be used in a pinch but i like EtOH, it mixes better with the DEET and has just enough water for the hydrolysis but not enough to cause separation.

* Diethylamine C4H11N 73.14g/mol boiling point 55.5*C (131.9*F)

* N,N-Diethyl-M-Toluamide C12H17NO 191.27g/mol boiling point 288-292*C (550.4-557.6*F)

* M-toluic acid C8H8O2 136.15g/mol boiling point 263*C (505.4*F) (melt at 111-113*F)

Per director of sound, oct 2010 (secondary forum posting):

Diethylamine

1. make a solution of 60g KOH/NaOH (NaOH works better) in EtOH (50ml)

2. its okay if it does not all dissolve.

3. add 175ml DEET (98.11%) to this solution in an autoclavable media bottle (can withstand high pressures) and place in a crockpot (sealed) set on high.

4. let it get to 80°C (176 F) and stay there for 1.5-2hr.

5. over this time the solution will progress from a clear soln to a dark piss yellow soln.

6. after the 1.5-2hr remove and cool the bottle before you open it or you'll spray diethylamine vapor everywhere.

7. fractionaly distill the solution with the addition of 20g lye to the distillation flask.

8. the diethylamine will come over at 55.5°C (132°F)

9. the ethyl alcohol will come over at 78.4 °C (173 °F)

10. the M-toluic acid dosent boil till about 230°C (446 F) so you wont be distilling any of that or the lye over.

11. since undoubtedly some of the EtOH and a little water came over you will have to distill again but distill over NaOH (25g per 25ml of distillate) and play close attention to the temperatures as the fractions start coming over.

12. the temp will continue to rise untill it hits the boiling point of the diethylamine and stop there. when it starts to climb again all of the diethylamine is across and the EtOH will start coming over next would be the water if it didnt get trapped by the lye.

13. you can expect to get about 30g (or 90 ish ml) form 175ml of DEET, that is enough for a few small batches as you only need about 7g per 3g of lysergic acid...

question from not a troll, 2009:

How long does the Naoh usually take to dissolve? My monkey tells me after 4 hrs it's not even half
dissolved. The first batch was heated between 50 and 60 C (122-140 F) and produced a deep yellow solution after about 2 hours. The second batch was overheated (to about 75 C (167 F), the water bath evaporated faster than

expected) and was pink by 4 hours. Neither batch dissolved even half the Naoh (pellet form). All starting materials were reagent grade with exception of the DEET, which was labeled 98.11%.

director of sound replied, 2009:

A bit of the Naoh won't dissolve. Mine was finely powdered rather than pellet form so the majority
(minus about 5g) dissolved. Your pink solution should progress to a dark yellow soon, it takes a
little time. The initial heating just starts the rxn. It progresses to completion over the next
day. Normally just DEET and Naoh or KOH would be combined and heated in a flask distilling the
diethylamine as it is formed.

That is more suited to a large scale production working with liters of DEET, by tomorrow you can
distill the rxn. mixture and get your diethylamine. A simple distillation before the fractional
is better yielding and gets a relatively pure distillation of the M-toluic acid will solidify,
that is your hint that there is no more that you can fractionally distill to get pure dry
diethylamine.

not a troll said:

The pink solution never turned yellow, and changed from pink to purple when distilled. The solution
sat for about 30 hours before being distilled. It distilled off at 74 C (165 F) so I'm assuming there's no significant amount of DEA in it. I'll try again with more careful temp control and use powdered Naoh (as opposed to pellet form).

director of sound replied:

That is odd, swim has never had the solution turn pink...was it just lye/DEET/ethyl alcohol?
or was there something else in there?

not a troll said:

Just the 3 magic ingredients. I think I'll blame it on overheating, there's a lesson to you all
on temperature control.

Winder, June 2012:

I have worked with aromatic amines (anilines) that were protected with acetyl groups.
If anything, these were rather easily hydrolyzed, since the aniline had a resonance structure available that was not so important for the acetanilide, thus making the process favorable to the product formation.

Of course, DEET is a amide of a benzoic acid, so -C(=O)NEt2 is on the aromatic ring, not -NHC(=O)R
I do not see why a benzamide should be so resistant to hydrolysis though. Diethylamine is fairly strong base and should be happy to take a proton.

When I did perform hydrolysis of acetanilides, they were alkali-driven reactions. With aromatic amines as the products, loss of the product by vaporization was not an issue.

With DEET as the starting material, alkaline-driven hydrolysis will make free-amine.Diethylamine is very volatile, so loss is an issue. Maybe a decent yield would be possible by distillation while reacting.

Consider refluxing DEET with NaoH in an ethanol/water mixture, to enhance the solubility of the DEET. The products would be the sodium salt of the toluic acid, which would have low volatility, and free amine form of diethyl amine, which would have high volatility. After some refluxing period, a side arm could be used to take off the diethylamine into ice-chilled dilute hydrochloric acid.

B--Per Makoeyes:

Makoeyes method of practical LSD production:

Step 3

Distillation of DEET to Diethylamine.

1. Mix DEET with an excess of 10--20% aqueous Naoh
2. Distill, collect the distillate in dilute HCL.
3. Evaporate HCL to get Diethylamine Hydrochloride (to circumvent this stage just buy some
   Diethylamine HCL, it's pretty suspect tho)

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 10, 2013, 04:32:46 PM**

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step 4: Coupling of LSA with Diethylamine via peptide coupler reagent with stirring
at room temperature in DCM for 3 hours

A--yield of Lysergamides, general method using PyBOP, David E. Nichols
B--yield of 3.13g, and 5.55g LSD, using PyBOP, Casey William "Freeblood" Hardison
C--yield of LSD from Proposed synthesis procedure using BOP-reagent
D--yield of LSD from Proposed synthesis procedure using HBTU/HATU
E--yield of LSD from Proposed synthesis procedure using DCC in presence of HOBt
F--yield of 1.7g D-Lysergic Acid Diethylamine, using PyBOP (makoeyes)
G--a few notes on coupling

A--Per David E. Nichols, "Lysergamides of Isomeric 2,4-Dimethylazetidines Map the Binding Orientation of the Diethylamide Moiety in the Potent Hallucinogenic Agent N,N-Diethyllysergamide (LSD)",Journal of Medicinal Chemistry, 2002, Vol. 45, No. 19, 4344--4349:

Synthesis of Lysergamides. General Method.

1. Lysergic acid monohydrate (obtained from NIDA) (200mg, 0.75mmol)
2. PyBOP (426 mg, 0.82mmol)
3. and the appropriate 2,4-dimethylazetidine (109mg, 0.9mmol)
4. were all suspended in 20 ml of CH2CL2 (DCM)
5. Diisopropylethylamine (193mg, 1.5mmol) was added, and the reaction was stirred for 3h.
6. The reaction was then quenched by the addition of 20 mL of 7.5 M concentrated NH4OH,
7. The CH2CL2 layer was separated,
8. and the aqueous phase was extracted with 10ml of CH2CL2.
9. The organic layers were combined
10. and washed with H20 (2 x 30ml)
11. and washed with brine (15 ml)
12. and then dried with (mgSO4)
13. the solvent was then filtered
14. and the solvent then removed by rotary evaporation under reduced lighting
15. followed by drying under high vacuum
16. produced a light golden foarm
17. this crude product was then subjected to purification by centrifugal thin layer preparative
chromatography (Chromatotron, Harrison Research) over a silica rotor and elution with
4:1 Ch2CL2/hexane under an N2--ammonia atmosphere.
18. The faster-moving blue fluorescent band was collected
19. and the solvent was removed under vacuum in the dark.
20. the normal lysergamides were then dissolved in tert-BuOH and combined with 0.5 equiv of
(+)-tartaric acid dissolved in a minimum amount of PrOH.
21. The solutions were stored at 0 C (32 F) overnight.
22. The crystalline products were collected by filtration and dried overnight at 60 C (140 F) under high vacuum.

B--Per Casey William "Freeblood" Hardison, "Novel Condensation of D-LA into D-LSD via PyBOP
The Entheogen REview, Volume XIV, Number 1, Page 94:

A recent publication by Dr. David E. Nichols (Nichols et al. 2002) on the isomeric lysergamides
of dimethylazetidine catalyzed a revolution in the realm of clandestine LSD synthesis. I do not
know if Dr. Nichols is to be credited with the first use of PyBOP for lysergamide condensation,
as theoretical discussions on the use of a variety of peptide-coupling reagents have been
occurring on The Hive and Rhodium web sites since 2001.

In early 2004, I engaged Dr. Nichols in a theoretical discussion as to his expected limits on
scaleability and it was clear that he did not know, as he is limited to NIDA quantities of the
lysergic acid, i.e. < 250mg.

After studying Dr. Nichols papers and the Internet, and doing further book research on peptide
synthesis (Coste et al. 1990), I conducted a series of experiments to determine the limits and
parameters of the reaction, i.e., the best solvent, the best tertiary scavenger amine, the best
sequence of introducing the reagents, and the most effective reaction time.

I worked with several solvents, but I found CH2CL2 (DCM) to be most suitable, as it evaporates
easily and keeps the reaction temperature low.

I worked with several tertiary amines, but N,N-diethylmethylamine added slowly after the dry
lysergic acid gave the most effective results and work-up.

I varied the reaction time between 30 to 120 minutes; however, I am of the opinion that the
reaction completes in less than one hour. All reactions were conducted under a 15w red light,
in an Argon atmosphere, and with dried Sigma-Aldrich solvents and reagents.

Experimental

1. 2.80 grams of lysergic acid was added to 100 ml of magnetically stirring CH2CL2.
2. To this was added 1.81 grams N,N--diethylmethylamine and the solution was allowed to stir
   for 5 minutes.
3. Then 5.70 grams of PyBOP was added and the solution was allowed to stir for an additional
   5 minutes.
4. Then 0.84 grams of diethylamine was added and the reaction was allowed to stir at room temp
   for 60 minutes.
5. The reaction mixture was quenched with 100ml of 7.5M concentrated NH4OH
6. The layers were separated
7. and the aqueous phase was x3 times extracted with 30ml CH2CL2,
8. the organic layers were combined
9. and rotary evaporated at 35 C (95 F) under high vacuum.
10. The residue was dissolved in 40ml of cold saturated NaHCO3,
11. and extracted x 3 times with 20ml EtOAc,
12. the organic layers were combined
13. and washed with deionized H20
14. then washed with brine
15. then dried over MgSO4
16. then filtered

17. and rotary evaporated at 40 C (104 F) under high vacuum to a constant weight
18. Yield: 3.13 grams LSD before chromatography, 93%
19. Another run of 5.12 grams lysergic acid with the same amines, equivalents, and times,
yielded 5.55 grams after chromatography, 90%

C--Per KCN (the Hive):

Coupling with BOP-Reagent

Proposed LSD synthesis procedure:

1. 1eq. LSA is dissolved in a suitable solvent (must be fairly dry) at room temp,
2. 1.05 eq BOP-reagent is added
3. 2eq. of diethylamine is added and the rxn is stirred at RT until it goes to completion which takes (15min--2hr).
4. The solvent is removed under vacuum
5. and the residue partitioned between EtOAc (or other suitable solvent) and saturated NaHCO3 (or Nh4OH).
6. The layers were separated
7. and the organics were washed with NaHCO3 (or NH4OH)
8. washed with H20
9. washed with saturated NaCL
10. dried over MgSO4
11. filtered
12. and concentrated in vacuo to remove the solvent and excess diethylamine
13. The crude LSD, which should be fairly pure, is then further purified by chromatography and
converted to the tartrate salt.

D--Per KCN (the Hive):

Coupling with HBTU/HATU

Proposed LSD synthesis procedure:

1. 1eq. LSA is dissolved in a suitable solvent (must be fairly dry) at room temp,
2. 1.05 eq HBTU/HATU is added
3. 2eq. of diethylamine is added and the rxn is stirred at RT until it goes to completion which
takes (15min--2hr).
4. The solvent is removed under vacuum
5. and the residue partitioned between EtOAc (or other suitable solvent) and saturated NaHCO3 (or Nh4OH).
6. The layers were separated
7. and the organics were washed with NaHCO3 (or NH4OH)
8. washed with H20
9. washed with saturated NaCL
10. dried over MgSO4
11. filtered
12. and concentrated in vacuo to remove the solvent and excess diethylamine
13. The crude LSD, which should be fairly pure, is then further purified by chromatography and

converted to the tartrate salt.

E--Per KCN (the Hive):

Coupling with DCC in the presence of 1-Hydroxybenzotriazole (HOBt)

Proposed LSD synthesis procedure:

1. 1eq. LSA is dissolved in a suitable solvent (must be fairly dry) and 1.05 eq HOBt is added.
2. The solution is cooled to 0 C and 1.05 eq. of DCC is added.
3. The rxn is stirred at 0 C for 30 min.
4. 1.05 eq of diethylamine is added and the rxn is stirred at 0 C for 30 min,
5. and allowed to warm to room temp
6. and is stirred until the rxn goes to completion (0-24hr)
7. The rxn is cooled in the freezer to precipitate out the maximum amount of DCU.
8. The precipitated DCU is filtered and washed with solvent.
9. The solvent is removed under vacuum
10. and the residue partitioned between EtOAc (or other suitable solvent) and saturated NaHCO3
(or NH4OH).
11. The layers are separated
12. and the organics were washed with NaHCO3 (or NH4OH)
13. washed with H2O
14. washed with saturated NaCL
15. and dried over MgSO4
16. filtered
17. and concentrated in vacuo to remove the solvent and excess diethylamine
18. The crude LSD, is then further purified by chromatography and converted to the tartrate salt

F--Per Makoeyes:

Makoeyes method of practical LSD production:

Step 4

Coupling of LSA with Diethylamine via PyBOP.

1. Dissolve 1g LSA in anhydrous Toluene at RT
2. Add 1.05 g PyBOP.
3. Add 2g of diethylamine, stir reaction at RT until it goes to completion (15min--2hr).
4. Remove the solvent under vacuum.
5. Load residue from step 4, Toluene and saturated NH4OH into a sep funnel.
6. Wash the organic layer with NaHCO3 (or NH4OH) and H20.
7. Saturate with NaCl.
8. Dry over MgSO4.
9. Filter and concentrate in vacuum to remove the solvent and excess diethylamine.
10. Convert to the tartrate salt.

Let's talk about purification. Obviously your chromatography column is your best friend, mine

is...we play poker on thursdays, him and my sep funnel.

G--a few notes on coupling:

Dr. Bob (2013):

If you are looking for a simple peptide coupling agent, EDC and HBTU are the most commonly
used for simple work. HATU is much more expensive and normally only used for problematic
couplings, as well as PyBOP. EDC is the least hazardous of them, generally, and easiest to
use, as you just mix amine, acid, and EDC in DCM, THF, or similar solvent, stir a while and
then wash the reaction with water, dil acid, dil base, and the amide is left in the organics
to dry and concentrate. If you have chiral acid, then HBTU might work better, but produces
more by-products which are harder to remove, so chromatography is often needed.

Just make sure NOT to let the coupling agent sit with the amine without the acid present,
as that often leads to side products. Best to add acid, then coupling agent, mix and stir,
then add the amine. If not chiral, then mix the acid and amine then add the coupling agent--
that works in most cases.

Piglet (2006):

Ergotamine is NOT the best start material. Cabergoline is commercially available and leads to a legal & stronger analogue. BOP leaves some toxic shit, hence PyBOP. More expensive, but lots safer. Trade secrets prevent me from revealing my route but anyone with the asbility to make it will easily find a good 3 stepper from simple, essy to buy compounds. The mild conditions means that no isoLSD is formed.

Forget ALL published books (even Shulgin) and go for the journals. Perdue is the home of the chemistry....BTW DEA isn't THAT watched. I also invite interested parties to consider other amides & substitution of the 8 nitrogen. Just don't use the Hoffman degredation; Ethyl chloroformate is MUCH better.

Nobody EVER grew ergotamine to make LSD! It's total BS from a 4th rate chemist. ET (and other useful compounds) can be bought. Eastern Europe also has LOTS lying about. The problem is knowing what condition it is in! One hint I happily give is NO book gives a good route to the amide. I mean, jesus, SO3, Hydrsazine or Phosgine! Method X (snigger). Anyone who can do this reaction will know about PyBOP or other coupling agents developed SINCE the early 70s! That's how it's all made now. Why not more? Bulk prices are low, massive distribution for a non-addictive drug.... May as well make something like 4-methyl fentanyl (easier than 3, not controlled)

Using modern methods, I can reassure people that a 2 step route with over 50% yield is available. You don't need toxic OR controlled chemicals. The biggest myth is that diethyl amine is hard to get. Well, diethyl formamide + NaOH gives 100% yield of it. DEF is EASY to get...

And what side-products would you expect? Also, how to shift that equilibrium? Well, what's the BP of DEA? OK, it's a 99%+ reaction! Also, HBTU HAS been used & PyBOP used for some analogues (isobutyl amine). Cabergoline synthesis is a GOOD place to look. Actually, it's a 3-stepper including saponification...

BTW there's toxic and there's toxic. I already mentioned the compounds I consider risky. DEA is smelly, but an extractor fan & fume hood work fine. You messed with SO3 or N2H4? Instant lungfuck & cancer with the latter. That's toxic! I wouldn't 'swim' in most any chemical, so it doesn't worry me... do you?

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 10, 2013, 04:35:13 PM**

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step 5: Cleanup

Per David E. Nichols, J. Med. Chem 2002, 45, 4344-4349, April 10, 2002:

1. reaction then quenched by adding 20ml of 7.5M concentrated NH4OH (ammonium hydroxide)
2. The Ch2CHL2 (DCM, Dichloromethane) layer was seperated
3. Go back to the aqeous phase and back-extract with 10ml of DCM to get any left
behind product
4. Combine all the organic layers (DCM)
5. Wash the DCM with H20 (2 x 30ml)
6. Wash the DCM with Brine (salt water, 15ml)
7. Dry the DCM with MgSO4 (magnesium sulfate)
8. Filter the solvent
9. Evaporate off the solvent under vacuum or use a rotovap under reduced lighting
10. Dry under high vacuum
11. Left behind a light golden foam. This crude product is LSD.

Per Casey William "Freeblood" Hardison, Entheogen Review, Volume XIV, Number 1, page 94:

1. The reaction mixture was quenched with 100ml of 7.5M concentrated NH4OH
2. The layers were seperated
3. The aqueous phase was then thrice extracted with 30ml CH2CL2 (DCM)
4. The organic layers were combined
5. The solvent was then rotary evaporated at 35 degree C (95 F) under high vacuum.
6. The residue was dissolved in 40ml of cold saturated NaHCO3 (sodium

bicarbonate)
7. Then extracted thrice with 20ml EtOAc (Ethyl acetate)
8. The organic layers were combined
9. The organic layer was then washed with deionized H20
10. The organic layer was then washed with brine
11. This was then dried over MgSO4
12. Filter the solvent
13. Rotary evaporate the solvent at 40 degree C (F) under high vacuum to a constant weight
14. Yield 3.13 grams before chromatography, 93%
15. Another run of 5.12 grams lysergic acid with the same amines, equivalents, and times,
yielded 5.55 grams after chromatography, 90%

Per KCN (the Hive):

1. The solvent is removed under vacuum and the residue partitioned between EtOAc
(or other suitable solvent)
2. Saturate with NaHCO3 (or NH4OH).
3. The layers were seperated
4. The organics were washed with NaHCO3 (or NH4OH)
5. The organic then washed with H20
6. The organic then washed with saturated NaCL
7. Dry the solvent over MgSO4
8. Fiter
9. Concentrate in vacuo to remove the solvent and excess diethylamine
10. The crude LSD, which should be fairly pure, is then purified by chromatography
11. Convert to the tartrate salt

Note 1: Method X

---

note: melting point of LSD is 80 C, 176 degree F.

note: Lysergic acid melting point is 240 C, 464 degree F.

[editor's note: wouldn't the LSD be destroyed in the pipebomb as it is being created due
to the high temps?]

[Here we go.....]

From "Practical LSD Manufacture" by Uncle Fester, Pages 67-74. Method X refers to: Chem Abstracts Volume 69, entry 106934 (1968) and Czech Patent 125,498.

This method uses an entirely novel route to LSD. It is mentioned in this reference and never again. One general method of producing amides, such as LSD, from organic acids such as lysergic acid is to
simply heat the organic acid and an amine inside a sealed tube. In this case the

reaction would be between lysergic acid and diethylamine to give LSD, and they claim yields of 90 to 100%!

There is one caveat on this procedure. They used the hydrogenated form of lysergic acid
in which the double bond at the 9-10 position has been reduced. It may be that this form of lysergic acid tolerates the very high temperatures used in the reaction better than natural lysergic acid.
There are some very high temps used in this process, but they are below the melting point of lysergic acid. The natural acid decomposes at its melting point of roughly 240 °C, and this process gets close to that temperature.

In their Patent, the inventors took some threaded stainless steel pipe and securely attached a stainless steel cap onto the bottom of the pipe. They put an excess of amine into the pipe along with a couple volumes of acetone, then added the lysergic acid. They bubbled nitrogen down into the solution in the pipe so that all oxygen would be flushed out, then they screwed on the top cap of the pipe. They next proceeded to heat this mixture at 195 to 200 °C for a prolonged period of time. For benzyl amine they heated for 8 hours, and for cyclohexylamine they heated for 60 hours. They never tried diethylamine in the experimental results published in Chem Abstracts, but I would think that 24 to 60 hours should be enough for diethylamine. It's not as reactive as benzylamine, but it should be pretty similar to cyclohexylamine. The temperatures used are below the critical temperature of diethylamine, so if the natural lysergic acid holds up to the heat, this method would be the winner of the simplest, best, and highest yielding recipe contest. There is no mention in the Patent as to whether the lysergic acid should be the baked dry form. There is water formed as the result of the reaction anyway, so it seems that small amounts of water carried in from the lysergic acid would be OK.

To isolate the product, simply cool the tube and evporate off the excess diethylamine under a vacuum. Take up the residue in chloroform, and wash it with dilute ammonia or bicarb water. Then
the chloroform extract can be evaporated down and either chromatographed or directly crystallized as the tartrate

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Note 2: A few comments about rotovaps

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jon:
---
rotovaps (2-3000 at least) are expensive but for molecules as touchy as this pretty much mandatory.

solidstone:

Rotovaps are essentially a warm water bath. A airtight swivel(used for glass blowing, easily made). A motor to rotate. A vacuum source. and a cold finger.

A functional one can be made for much less then 2-3000.

With a rotovap one could keep re-using ones solvents, cutting down on suspicious aquisitions.

jon:
---
right i'd love to see a blueprint. save us all a bundle. lysergamides really don't like to be in solution as freebases for very long very prone to oxidation.

vesp:

I believe I have seen smaller - questionable ones for around 150 to 200 dollars. I do not know if they would be of any interest, but I guess this is a topic for another place and another time.

jon:
---
well a rigged up alternative is to run a n2 capillary through a magnetically stirred solvent to be evaporated it's called "sparging" works pretty well to carry off solvents.

overunity33:
----
does the capillary gas really make it evaporate that much faster? Could you just rig a room temp distillation setup with a few ambient-air capillarys, stirring and have the condensor running room temp water, have this thing sitting in the corner distilling your solvents slowly but surly?

jon:
---
umm you want an inert gas co2 at the very least yes it speeds up evaporation considerably.
a tapered tube allows for smaller bubbles and greater surface area of gas and faster evaporation.

trips:

Why is Director Of Sound's post cut off? Where is the rest? Also, Buchi rotovaps can be obtained complete and fully functional with Glassware off ebay used for ~500 bucks if you're patient.

Note 3: A difficult coupling procedure

Director of sound (2010):
Quote

On Phosphorus oxychloride, POCl3 whatever you want to call it. it is accually quite easy to make at home with materials you can get down the street at the pool store. glassware most of us have if you are indepth with the feild of organic synthesis, or you can get it cheap on ebay... so here it goes:

There are 2 choices for your chlorine production, one is wet and the other is so wet it can hurt your yeild if not dried well. (2ft section of PVC filled with anhydrous MgSO4 works good). The first which is prefrable uses Trichloroisocyanuric acid (TCCA) tablets (or powder) which you can find at a pool store, it is used as a chlorinating agent. The other uses household bleach (remember its only 5% the other 95% is water). Both use HCl to strip the chlorine gas off the other moleclues. I prefer the TCCA as it generates

much more chlorine than the bleach/HCl method and it is much dryer. The next draw back is that you will not be using the chlorine as it is generated, it must be premade and stored so your Cl2 generation vessle must be able to withstand at least 3-5 bar (40-70 psi) and safley store it for some time. A heavy walled 1000ml round bottom flask (with threaded necks) will hold that pressure easely the trick is getting the HCl into the flask either through a septum under one of the caps or squirting it in there and closing it quickly. My setup uses the said round bottom flask with three threaded necks one has the septum under a cap with a small hole drilled in it, the central neck has a pressure guage connected to it through a gland cap and tha last neck has a peice of tubing (also through a gland cap) leading to the PVC drying tube equiped with a valve at the other end. therefore when i introduce the HCL the pressure builds in the flask and the drying tube so the Cl2 gas is instantly at my disposal when i need it with out having to bleed the lines. The next step is to charge your rxn flask with dry RP powder, it can be safley baked at 200*F in your oven for a few minutes to dry it out. Use any ammount you can scrounge up as it goes a long way to have more than you expect to need (yields most often are not 100%). Just dump it in your three neck rxn flask dosent have to be a huge 5000ml or more but at least 500ml. after the rp is in the flask lead the Cl2/helium line through one stopper (use cork POCl3 attacks rubber stoppers) the center one is equiped with a glass stir bar that reaches the bottom with atleast 5cm to spare, the end in the flask has had a short section of glass rod melted to it crossways. The last neck has a hose leading out a window or to a hood if you have one. The flask is now charged with helium, not helium for baloons, that stuff is only about 35% helium and the rest air. get a small cilinder from a welding store, nitrogen will work in a pinch but you want a light gas. If you were to use argon it would blanket the RP and prevent the Cl2 from getting to it. Now that your flask in inerted slowly (REALY SLOWWWW...) turn on the supply of Cl2. The trick here is to let the chlorine 'trickle' in in small ammounts (PPH ammounts). If you put too much in the RXN goes exothermic and will produce flame, possably an explosion if you have a large enough ammount of RP or melt through your flask creating a dangerous chemical fire. If you do it right you can feel the flask by the RP and it will be warm possably hot but not flaming. Gradualy the RP will start to look 'wet' as the phosphorus trichloride starts to form you may have to break up and stir the RP from time to time to get it all to react. Eventially you will have a puddle of PCl3 with some RP that did not react. Now you can turn up the chlorine a little bit and start stiring to react the rest. Once the liquid is as clear as it is gonna get flush the flask with helium again and insert a section of glass tube into the liquid. You now have to pass pure O2 through it for several hours 2 is enough with a heavy stream but it can get real hot. a small stream of bubbles in a heavely stirred mixture (toss your magnetic stir bar in now if you have one) over night will be perfect. Next in a dry room (turn on the dehumidifyer the night before) extract your POCl3 and transfer it to a smaller distillation flask. This will be a somewhat high temp fractional distillation so use a air condencer before your allihn or graham condencer or it could crack. The first fraction that should come over will start around 70*C (158*F) this is the unreacted PCl3 once that stops (probally around 85*C (185*F) continue with heating untill the next fraction comes over around 100*C an stops when the distillation flask is dry. Congratulations you just mad some very pure (in the area of 98%) phosphoryl chloride!!! Use it wisely, it will fume in moist air and react violently with water forming phosphoric and hydrochloric acid throwing it everywhere. You dont want to get it on you either as you are mostly water it can act as a superacid and eat through your skin like a batch of conc. boiling sulfuric acid...

POCl3 boiling point 105.8*C 222.4*F

PCl3 boiling point 76.1*C 168.8*F

Note 4: How to setup and use an N2 tank

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Tsathoggua** on **September 10, 2013, 09:41:53 PM**

Bromocriptine is not OTC..at least not here in the UK.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Polonium** on **September 11, 2013, 05:13:45 AM**

Fantastic review of the current known experimental data. Good job tregar!

Just a quick note on Piglets discussion of Cabergoline. Hydrolysis and subsequent peptide coupling of this substrate will result in AL-LAD, an equipotent LSD analog which has recently become available on the online RC market. It looks like someone ordered a few grams on this legal substrate in from China and is turning a nice buck on it. Anyone seriously looking to succeed in this route might get the reactions and techniques locked down with this more available starting material before moving on to the valuably LA.

Maybe Piglet is far ahead on this route and has been producing AL-LAD as LSD since before 2006? A brief qualitative comment from it's TiHKAL entry is very positive.

Quote

> (with 150 µg) "Simply beautiful. Erotic and music absorption after second hour. Clear thinking with superb imagery and good interpretation. Easy, gentle sleeping. Next day -- serene, clear-thinking peacefulness. One of the best materials ever."

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **planckunit** on **September 11, 2013, 08:08:03 AM**

Quote from: Polonium on September 11, 2013, 05:13:45 AM

> Just a quick note on Piglets discussion of Cabergoline. Hydrolysis and subsequent peptide coupling of this substrate will result in AL-LAD, an equipotent LSD analog which has recently become available on the online RC market.

it's missing the 9,10 double bond of AL-LAD

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Polonium** on **September 11, 2013, 02:05:34 PM**

Sorry, you're completely right. I must have got a rush of blood to head and didn't really look properly. None the less AL-LAD seems like a promising compund

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Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **myCH3** on **September 11, 2013, 04:17:51 PM**

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piglet says its a three stepper yet it wouldn't hydrolysis followed by peptide coupling just be two steps? Could the omitted third step be the way to add 9-10 double bond? Is that even possible?

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 11, 2013, 05:42:03 PM**

Thanks for the kind words Polonium....and everystinger for your comments...none of this would be possible were it not for the heroic, intelligent, top notch research work of director of sound..he gave us all hope...on the other hand just check out the 20 plus page thread at sciencemadness of deet-->diethylamine, reading that is like watching 2 hours of bad commercials....reminds me of the Beatles song "no-where-man"...the whole thread goes nowhere, mere speculation for the most part, with no heroic nuts and bolts.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**

Post by: **Sneak** on **September 12, 2013, 10:33:20 AM**

Great stuff Tregar. Enjoyed reading through this. I have learnt a lot!

Good to see your still posting also.
Keep it up brother.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 13, 2013, 03:00:46 AM**

Thanks Sneak, appreciate the kind words.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 13, 2013, 03:52:26 AM**

That was a funny read when Webster asked those kids in Mexico if they wanted to go out and collect 100kg of morning glory seeds in coffee cans for dineros so they could then turn around and buy themselves transistor radios :) Ahhh but a few of them did it!

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Sneak** on **September 13, 2013, 01:50:50 PM**

That part also stuck in my mind

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 13, 2013, 03:48:55 PM**

How is Jon doing? Is he still active in the forum?

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Mango** on **September 14, 2013, 12:47:30 AM**

Not to be a hater but this is basically a copy-paste of 5,6 "papers", some of them of very questionable quality.

Hydrolysis via TiKHAL, coupling via Nichols. Done. Sourcing ergot alkaloids was always the most difficult part.

Like Piglet said you would be better off reading the original journal work than theoretical interpretations (Hive, Makoeyes?) or science fiction (Fester's method X, dihydro is not didehydro!).

If you have the material and the means for seeds to LA, great! Post a detailed tek with pictures, get people excited about it.

If you don't then this is all, in your own words mere speculation for the most part, with no heroic nuts and bolts.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 14, 2013, 07:01:16 AM**

> This is precisely how I get you to think....question everything and pick and choose the pieces that make sense to you, omit was is not valid....i'm not going to give the pics and all the answers, that is for you to do, i've done plenty of that over the years...you do the homework and you decide...go work Mango, precisely what I was looking for, it shows you took the time to read and question. You say sourcing ergot alkaloids was always the most difficult part...tell us more. The Hive is not science fiction, the Hive, as Casey pointed out, was talking about the use of peptide couplers way before Dr. Nichols even put then to use in his research, KCN had this down years before the good doctor did. I used to be a part of the Hive, got my training wheels there, all the research at the Hive was not for not, years of sweat and tears.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Mango** on **September 14, 2013, 07:17:39 AM**

> All that KCN did was theoretically apply peptide coupling principles to LSD (not to downplay the ideas, it is good theory).
>
> If you want to do the same get a peptide reagent promo flyer, look up at experimental, exchange amine with diethylamine and carboxylic acid with lysergic acid. Now you have LSD synthesis.
>
> Not.
>
> It might work (or not), it sounds plausible, but it ignores the real problem, that being the availability of ergot alkaloids/lysergic acid.
>
> When lysergic acid will be available for experimentation then this kind of debate will be fruitful.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 14, 2013, 08:22:15 AM**

> Thanks for the tips Mango!
>
> I apologize for my writing style, and even calling this a "tek" you are right though, in the past
> all of my "teks" have had lots of pictures and real world steps done with real world bolts and nuts,
> this compilation of papers (broken down into steps) was meant to be a starting point or jumping
> off point, picking pieces here and there that made sense, omitting the bad, and going from there,
> same way I start all my research....it's more a comprehensive pulling together of the minds, and you are right, fester is complete science fiction in my book! I only include him in there to show how bad he looks right upside all the other minds, and makoeyes is also uncomplete, those 2 are in there to show you how bad things can look, yet each of them still have a small bit of useful information, so that's why they were still included...yet omitting leads straight to alot of what

those 2 say. But as an aside, the peptide couplers can be acquired from china if so desired, easy as ordering hordenine or any other such best-seller from chinese site, N2 tank easy enough to get at your local town gas shop, just make sure to pick up a regulator meant specifically for N2, and the adapter that connects the two together (important) & some plumbers tape for a great seal, may also need a business card to request a tank.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **ImAMANGUYS** on **September 16, 2013, 03:59:48 PM**

Quote from: tregar on September 10, 2013, 04:30:33 PM

C--Per Michael Valentine Smith, LSD:

Ergot Alkaloid Hydrolysis

1. Dissolve 20g of the alkaloid (e.g., ergotamine) in 200ml 1M KOH in methanol (this is made by dissolving 56g KOH pellets in 1L 100% methanol)

2. In a 1L heavy walled vacuum flask, evaporate in vacuum the methanol at room temperature.

3. To prevent the solution from cooling, and thus greatly prolonging the evaporation time, put the flask in a pan of water kept at room temperature by gentle heating or by running wqrm water through it.

4. Add 400 ml 8% KOH in water to the residue and boil for one hour (under N2 if possible, this can be done by filling the flask with a N2 stream and loosely stoppering or by allowing a gentle stream of N2 to low through during heating.)

5. Cool, acidify with dilute sulfuric acid and shake in a separatory funnel with 1 L ether.

6. Discard the upper ether layer and filter with vacuum the aqueous suspension of lysergic acid.

7. Wash precipitate with 20ml dilute sulfuric acid.

8. It is unnecessary to purify, but this can be done as follows: dissolve 9g in 20ml NH4OH, filter and concentrate in vacuum at room temp to precipitate. After filtering, the grey crystals can be further purified by dissolving in boiling water and cooling in an ice bath to precipitate.

Can anyone theorize on why the author suggests that the amides first be hydrolyzed in methanolic KOH before the aqueous hydrolysis? I'm trying to understand what the point of that is... couldn't the amides be solely hydrolyzed in water such as in the *LSD* entry in TIHKAL?

Thanks for anyone who'd chip in, I'm sure I'm just missing something simple.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Baba_McKensey** on **September 16, 2013, 04:29:54 PM**

Is the presence of water necessary to hydrolyse amides, or can an amide bond be split with KOH in MeOH?

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **java** on **September 16, 2013, 05:19:28 PM**

Reference Information

Hydrolysis in the absence of bulk water 1. Chemoselective hydrolysis of amides using tetrahalophthalic anhydrides
Jefferson T. Eaton, William D. Rounds,
Tetrahedron Letters
Volume 29, Issue 50, 1988, Pages 6553–6556

Abstract
The reaction of primary and secondary amides with tetrafluorophthalic or tetrachlorophthalic anhydride gives carboxylic acids in good yield. The reaction is chemoselective in that the amide functionality can be hydrolyzed in the presence of ester groups.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 18, 2013, 05:34:17 AM**

Recent Update: "addition 1" to Step 1 of the rambling tek:

Step 1: Defat the seeds and extract the lysergic acid amides

continued....

G--Per Eye of Ibad "the real morning glory extraction"
H--Per Kash "the advanced Lysergic Acid Amides extraction"
I--Per Halfapint (the Hive)
J--Per KRZ (the Hive)

G--Per Eye of Ibad:

The following extraction TEK contains nothing new. It is a combination of the collective knowledge around the net, with some refinements and additions by myself. You will recognize that the TEK is largely derived from the Dryteks floating around the net. There are many versions, but none of them seem to include adequate details or solutions to problems. There is one that utilizes limonene as a solvent. I have nothing against limonene per se, but I required a solvent that is more available and less pricey. The other parts of the tek are similar to the FASA method, another dry tek. The FASA method takes advantage of the fact that DMT fumarate is not soluble in acetone. I applaud those who have pioneered those teks.

The extraction was originally based on a similar extraction in Otto Snow's book "LSD" which uses baking soda for the base, sodium sulfate as the desiccant, and ethyl acetate as the solvent.

Quote

> [Editor's note, I believe Eye of Ibad is referring to:
>
> D--Per Otto Snow, "LSD"
>
> Extraction of Ergoline Alkaloids from seeds
>
> Method A
>
> 1. Pulverized seeds (100 grams) must be defatted before extraction of alkaloids.
> 2. Naptha or petroleum ether are suitable solvents for fat extraction of the seeds.
> 3. The seeds can be refluxed in the solvent or they can be refluxed in a Soxhlet extractor.

4. The seed mash is then filtered from the solvent.
5. Total extraction of fats is accomplished when new solvent extract leaves no greasy residue on evaporation.
6. The seed mush is then allowed to dry of solvent,
7. mixed with 500 mL of 10% ammonium hydroxide (strong ammonia water)
8. and extracted with ether or appropriate solvent.
9. Evaporation of the solvent leaves the alkaloids. Reference (Genest 1965)

Method B

1. 100 grams of pulverized seeds is mixed with 50 grams of sodium bicarbonate and 100 mL of water.
2. 100 grams of anhydrous soldium sulfate are mixed to leave the mass dry and granular.
3. The mass is extracted x 3 times with one liter of ethyl acetate.
4. The ethyl acetate solutions are combined and evaporated to leave the alkaloid residue.
Reference: (Marderosian 1966)

All extractions should be done under inert atmosphere. Ergot alkaloids will decompose in light, heat and air. Tartrate and maleate salts are less susceptible to destruction.]

I have chosen to call this the Acetone Tek, because it is distinct from the other teks in some ways and it uses acetone as the primary solvent. In fact, it is the only solvent you will need for this tek other than water. The basic idea of the extraction is to freebase the alkaloids with sodium carbonate or related base, dry the plant material, extract with acetone, and precipitate with tartaric acid.

Sodium carbonate was chosen as the base because it is highly available in high purity, it is cheap, and it can be used as a base and as a desiccant. It is also exceedingly safe to use, and it would be difficult for even the clumsiest of stoners to hurt themselves with. It can be prepared quickly and easily on a stove top from baking soda whenever you need it. It is just basic enough to fully freebase most alkaloids that we work with. It is called washing soda, and it is not in the least bit suspicious to have tons of it around because it has a million and one known uses in a normal household.

Tartaric acid was chosen as the acid because it is available at any grocery store that has a McCormick spice rack as cream of tartar. Its salts are non-hygroscopic, and it appears that most or all alkaloid salts of tartaric acid are insoluble in acetone.

Acetone is reasonably cheap, available in gallon quantities pretty much everywhere, and it dissolves almost any alkaloid freebase. It evaporates fast and cleans up nicely with a bit of activated charcoal. It is relatively non-toxic, and the smell isn't terribly offensive, if a bit strong at times.

This Tek is direct, simple, and the result is a solid crystalline product, even in cases where crystals would normally be difficult to obtain (such as with morning glories or ephedra). I will include the teks of how to produce anhydrous sodium carbonate from baking soda and how to make tartaric acid from cream of tartar. You always have the option of finding a homebrew store or doing mail order to get tartaric or fumaric acid (which should be interchangeable in this tek), but I like the idea of getting everything I need from the grocery or hardware store with cash.

Shopping list...

Grocery Store

1) 4 pound box of baking soda
2) Package of cream of tartar

Hardware store

1) Muriatic acid (This stuff is dangerous. If you didn't know that already, it's probably best if you did some reading on the safety of using hydrochloric acid in high concentrations before getting started.)
2) Acetone

That's all! You only need a little bit of muriatic acid to make tartaric acid from cream of tartar. Unfortunately, I have only found it in gallons, but if you can find it in smaller volumes go for it.

Cream of tartar is potassium bitartrate (that's one-to-one potassium and tartaric acid) that forms as a byproduct of the wine making process. All of the tartaric acid contained therein is in the L-Tartrate natural isomeric form, which is the one we want.

****Wear goggles, gloves, long sleeves, long pants, and shoes when you are doing this stuff. When you have concentrations of toxic materials dissolved in acetone, you don't want it to contact your skin (or eyes, mucous membranes etc). Also, use common sense as far as safety, acetone is very flammable and volatile. Only work in well ventilated areas away from sparks or flames.****

Finally, here is the Acetone Tek

1) Grind your chosen plant material to a fine powder and put into a mixing bowl.

2) Saturate a small amount of water with sodium carbonate, and mix it with the powdered plant material thoroughly. Use enough water to make it into a thick paste. Let it soak for a while. I also add a pinch of ascorbic acid at this point to act as an oxygen scavenger and buffer.

3) Stir anhydrous sodium carbonate into the contents of the bowl until the texture is granular and flowing. The sodium carbonate will absorb water slowly, so let it sit for a couple hours or until your mixture is dry and can be re-powdered.

If you haven't already, safety gear must go on after this

4) Put the powdered mixture into a glass or metal container and add enough acetone to cover. Let that sit for a few hours. You may want to add a bit more dry sodium carbonate at this point to make sure it sucks any remaining water out of the acetone.

5) Decant the acetone into a separate container and refill the extraction vessel with acetone until the plant material is covered. Let it sit for a bit. Then decant the second batch of acetone into the first. Make sure the extracts are free of debris. Filter it or decant it repeatedly if you have to. Acetone dissolves most plastics, so be careful.

6) Take a small amount of acetone and saturate it with tartaric acid by stirring it for a several minutes.

7) Pour the tartaric acid saturated acetone into the combined acetone extracts with stirring. Crystals will precipitate immediately. Allow it to sit for a couple

hours.

8. Decant the acetone off of the precipitated bitartrate salts, and rinse them once (or several times if you have enough 'tone) with clean acetone. Decant off the fresh acetone and dry the crystals.

A minuscule amount will precipitate out if you let it go over night, but IME it wasn't a quantifiable amount and probably wasn't worth the wait.

Simple, direct, effective. The result is surprisingly clean. Those lysergamide crystals will glow an oh so bright blueish white under a black light.

The TEK for making tartaric acid from cream of tartar is as follows:

1) 10 grams of cream of tartar, 10ml of concentrated HCl, and 100 ml of water are mixed together until all of the cream of tartar dissolves.

2) Evaporate the solution until most of the water is gone and there are visible solids precipitated out of solution. You want to avoid heating the solution directly. Drying with warm air in the stove or a dehydrator on low heat is fine.

3) Add acetone until the KCl precipitates out (it's obvious when it does) and then continue to stir for several minutes. The acetone is then separated and the KCl put aside to be re-extracted with acetone once more.

4) Evaporate the acetone in a flat bottom dish or plate to yield tartaric acid crystals. CAUTION: Acetone fumes are very flammable! Only evaporate acetone in well-ventilated areas or outside.

The above tek was adapted from the tek contained in the 'One-Pot shot LSD Synthesis' Tek. I used acetone instead of isopropyl alcohol to simplify the shopping list. Iso may have benefits like greater solubility of tartaric acid and less reactivity with the acid itself. See the original tek for more details.

Sodium carbonate is easily prepared from baking soda on a stovetop with a normal sauce pan. I usually make it fresh just before I use it so that I know that it's pure because it came from food grade baking soda and so that I know it's anhydrous because it is fresh off the stove. Anhydrous sodium carbonate will spontaneously absorb water out of the air making it an effective desiccant. Its pH rapidly rises above 11 and hits 11.5 at around 6 grams per 100ml, making it just strong enough for most of the alkaloids we work with.

Here is how to make sodium carbonate from baking soda

1) Dump enough baking soda into a sauce pan to make it about half an inch deep.

2) Turn up the heat to medium.

3) After several minutes, stir the pot by GENTLY swirling it around. If you do it nice and slowly, gasses will gradually be evolved and the baking soda will swirl around in the pot almost like a liquid. If you do it too fast, a big cloud of super heated baking soda, water vapor, and carbon dioxide will fly up into the air. It's a bad thing, stir slowly and gently to avoid it.

4) Turn the heat up to medium high and repeat the process many times.

5) You will know when the baking soda has been fully converted to sodium carbonate when it no longer flows around like a fluid when you stir it. Let it heat a bit longer to make sure it's fully converted and then let it cool.

Done. You now have ready to use, anhydrous sodium carbonate.

It's worth mentioning that this tek will work with other plant materials. I am still honing the TEK for them, so there will be more details to follow, but I will say that the product I got from ephedra was beautiful and potent as hell.

The big addendum to this tek is that step #3 of the extraction may be totally unecessary. If you used a saturated solution of sodium carbonate, all you have to do is cool it down and the carbonate will crash out as a hydrate and suck all of the water out of the mix. Of course, your plant material is a solid block at that point that needs to be ground up a second time.

H--Per Kash:

[editor's note: note similarities of this procedure to A--per director of sound's process, note also Kash's good information related to ph values you want to remain within, see Fester's process for
more information on PH values to remain within]

== Pure LSA Extraction, Visually Active with no Nausea or Bodyload ==

=== Procedure ===

Procedure Main: Clean Liquid LSA Extract

1)Pulverize your seeds into a powder using a coffee grinder or your method of choice.

2)Thoroughly mix in a glass the seed powder and 100 ml of acetone. Mix frequently for 2-3 hrs, the more mixing the better.

3)After this, filter solution and set aside acetone extract in a glass and cover with plastic wrap to prevent early evaporation. Put seed mush back into the original cup and cover with 100 ml new acetone.

4)Repeat steps 3-4 two more times so you have atleast 3 extractions (can do more if desired). . After third extraction, discard seed mush and combine the extracts into an evaporation dish/bowl. Set up a fan blowing on the extraction to speed evaporation. Doesn't take too long. Evap to dryness, making sure there is no more acetone smell.

5)Mix in a glass 100 ml distilled water and a pinch of citric acid (pH 3-5). Add to the dry crude LSA extract and mix well for 10 minutes. Not everything will dissolve. Filter out the solids. There should now be 100 ml of aqueous LSA citrate.

6)Defat this solution with 50 ml naptha, mixing well for 10 minutes. Seperate layers with a 10 ml syringe and discard naptha. Repeat. Make sure the second time that there is no remaining naptha floating in your LSA extract after defat.

7)Now its time to freebase the LSA so it can be extracted from the water layer. Add a small amount of ammonia (around 1 ml) to solution until it changes to a light yellow color. PH should be roughly 9-10.

8]Quickly add 50 ml toluene or DCM to the solution and mix well for 15-20 minutes. After, seperate the layers with syringe and set aside the 50 ml of toluene or DCM in a glass. Repeat this step 1 or 2 more times depending on how scrupulous you want to be. Afterwards, discard your yellow/green water layer.

9)Now with your combined toluene or DCM extracts, set them to evaporate in an evaporation dish/bowl with the aid of a fan. This again shouldnt take too long, and you end up with a pure white crystal residue once dry. This could be consumed, but will oxidize pretty quickly over a few days if left out of solution.

10)Once your pure extract is dry and there is absolutely no smell of solvent in your evap dish, add 10 ml distilled H2O and 5 ml 75% drinkable ethanol. Sprinkle in a pinch of tartaric acid and mix thoroughly for 10 minutes. Filter this, and store in a vial away from heat and light.

==== Comments ====
This product of clean liquid LSA extract glows bright blue under black light and '''MUST BE COMBINED WITH A SMALL AMOUNT OF PEPPERMINT OIL''' 20 minutes before consuming for an amazing psychedlic experience. If solution turns milky after peppermint oil addition, add a little alcohol to increase solubility. Inferior extraction recipes can leave you with stomach cramps and vasoconstriction, not to mention nausea, and the effects may not even be very impressive. A pure clean LSA extraction using this tek however... Absolutely no nausea or bodyload. It is a euphoric dreamy feeling psychedelic similar but different than LSD lasting about 6-10hrs, and can produce visuals made up of colors, vibrant pulsing tracers, flashy looking stars and symbols, and intricate geometric patterns. Good luck to all and I hope this tek creates many memorable experiences.

additional comments: === Dosage Information ===
LSA is a mid-duration psychedelic entheogen that lasts about 6-10 hours. The experience varies greatly on how the entheogen is consumed whether it is in the form of raw seeds or extracted. The exact nature of the LSA experience is debated, as experiences vary with each person. LSA tends to produce a dreamlike state with mild to significant visions and can be accompanied by euphoria, sedation, nausea, and vasoconstriction. Raw LSA containing seeds tend to bring on much more of the side-effects than cleaned extracts. Peppermint oil is often combined with LSA extracts leading to a more positive experience, though the exact mechanism for this phenomenon is unknown. Some believe it to be the formation of LSH, an unstable lysergic compound more closely resembling LSD, though this topic is hotly disputed.
It is difficult to accurately measure doses of LSA extract, since potency can vary. In general you can get a rough idea by comparing the ratio of seeds to liquid extract volume. A small amount of peppermint oil should be added to the LSA extract 20 minutes prior to consuming. While dosages vary for everyone, I believe this to be a fairly accurate scale of dosage for the extraction procedure:

* '''Threshold''' < 10 seeds worth
* '''Light''' 10-19 seeds worth
* '''Common''' 20-34 seeds worth

* '''Strong''' 35-50 seeds worth
* '''Heavy''' > 50 seeds worth

Ya you can literally just use a 10 ml syringe to seperate all layers in this extraction becuase it is such a small volume of liquid you are working with.

As for the dosages, they may be adjusted, but it seems you can handle alot more LSA when its not polluted with extra seed alkaloid impurities. Ive tried 20 and 30 seed extract portions so far and had a great time.

pau wrote:

One quick question on step 1 about boiling the MgSO4 until dry: is that boiling it straight from the jar, or boiling after the powder has been first diluted in water?

kash:

Thats cooking a 1/2 cup of epsom salt on a plate in the microwave for a few minutes, until all the water boils out of it.
The mgso4 is a drying agent for the acetone. Any water the acetone would absorb from the air would be only trace amounts and will leave a powder when dried.

If the product you end up with after evaporating your nonpolar solvent is an oil, it is OK as long as there is no more solvent smell. This just indicates you have some impurities in there, or the extract absorbed some water.

kash (on PH):

LSA is a really pH sensitive molecule, you cant just add excess base to it like dmt to get rid of an emulsion, it will start to hydrolyze Im pretty sure. Also, you really shouldnt heat it up at all really when its in basic solution. I also wouldnt recommend using lye. But hey let me know if it all worked out ok. Ive never had to deal with an emulsion in this tek.

Swim just took a 40 seed portion of LSA extract that was mixed for 15 minutes with peppermint oil yesterday and tripped his face off with a friend. Was very clean feeling and relaxed. Rainbows and vibrant fractal energy danced all over the skies and throughout his surroundings and music sounded great. The head-space was very acid like but different. Was a bit intense but he was able to keep it together lol. Whole trip was about 8 hrs long.

High pH 9.5+ will make iso-lsa and lysergic acid I beleive, both of which are orally inactive. When your pH exceeds 9.5, the solution will begin to go from green to blue, indicating that pH is too high and bi-products are forming.

Ethyl acetate can be made relatively easily, though not by the average home chemist since it requires mixing and distillation of ethanol, acetic acid, and sulfuric acid.

The idea behind keeping the acetone anhydrous is to not pull extra garbage that may be soluble in the water but not in the acetone. You could leave out the desiccant if you really want to, wouldnt affect much. However this extraction is primarily meant for purity.

SWIM likes to use a pH meter to keep the pH between 9-10, ideally 9.5. The alkaloids have a very strange way of acting like natural pH dye, if it is based proper the solution turns green (9-10pH), based too much it turns blue(10+pH), though the color takes about 15 minutes to develop.

About the peppermint oil, SWIM has done LSA with only 5 minutes mixing and had a terrible time just feeling sedated and anxious and not being able to sleep. SWIM's other times he has mixed for atleast 15-20 minutes and had a great time, having minor to significant visuals, euphoria, and very minor sedation. SWIM has not tested mixing with peppermint oil for long periods of time (weeks/months) becuase was worried of unforseen chem reactions, but it could probably be done. He also has stored the vial for up to a month with no loss of potency.

Room temp in amber glass, the alcohol acts as a preservative. Doesnt hurt to keep chilled though.

kash:

In response to a few pm's about subbing in materials... This procedure is the way SWIM has used with success and had zero problems every time. The materials can be procured EASILY without any specials licenses or access by anyone, and if you want a smooth extraction, use the materials mentioned.

That being said, SWIM beleives it would be possible to sub in some materials if people want to do so at their own RISK as I have not tried these...

Can recommend substituting ammonia and naptha. Ammonia can be possibly subbed with a decently concentrated sodium bicarb solution or a very carefully made light NaOH (Lye) or KOH solution, each requiring a pH of 9-9.5. If pH gets too high it will destroy the lsa, too low it wont extract. The naptha can be subbed with just about any organic solvent not miscible with H2O.

Tartaric acid could likely be subbed with fumaric acid.

As for the dcm/toluene, SWIM would really recommend just going to the small local hardware store and buying for 10$ in the solvent section... Other things may not extract as well and cause an emulsion. SWIM doesnt like Xylene smells horribly.

[editor's note: 99% D,L tartaric acid is also used in wine making]

=== Pictures ===

Here's a picture of final evap and a vial of clear LSA extract under black light.

I--Per Halfapint (the Hive):

Moekatis et al, Biochem. Physiol. Pflanzen, 1973, 164, 248.

Seeds:
-pulverized seeds defatted in pet. ether for 5 hrs.
-1 g of this material shaken 3X (each for 1 hr.) w/ 20mL of (2g tartaric acid in 30 mL H2O, 70 mL acetone) mix
-combined extracts heated on H2O bath (55°C) to expell acetone

-tartaric acid soln. shaken 3X w/ anhydrous ether, then basified (pH 8-9) w/ NH4OH
-from this soln' alks extracted 3X w/ 10 mL DCM
-combined extracts reduced to 1 mL and chromatographed

Roots, Leaves, Stems:

-pulverized material (15g) wetted w/ 3% NH4OH
-extracted w/ 200 mL DCM in Soxhlet for 6 hrs
-condensed to 20 mL & shaken 4X each w/ 10mL 2% tartaric acid
-total of 40 mL soln. extracted 3X each w/ 10mL anhydrous ether
-tartaric acid extract made basic w/ NH4OH (pH 8-9) & shaken 3X w/ 10 mL DCM
-total of 30 mL DCM extract condensed to 0.3 mL & chromatographed...

J--Per KRZ (the Hive):

Successful Crude Ergot Alkaloid Recovery Bookmark

Put 250g of Claviceps Sclerotia in a blender, added 1L of 5% Tartaric acid and homogenized on low setting for 3 hours. Added NH4OH 10% solution dropwise and waited until pH reached 8.5. Let stir in pH = 8.5 solution for ~30 minutes. Filtered and washed cellular sludge with ~100ml Benzene. Extracted 4x with 125ml of Benzene. Extracted benzene 4x with 125ml of 5% Tartaric Acid in absolute EtOH. Rotovapped down to ~150ml EtOH, placed EtOH in a shallow beaker in a dessicator and left in the dark. Collected prismatic ~2mm long ~1mm wide crystals 2 days later. Weight = 1.81g, stored in a brown glass vial, tightly sealed, wrapped in Al Foil in the refrigerator.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 18, 2013, 07:55:47 AM**

Concerning Step 1 and 2 which are the hardest....I'm really not sure "which of the A thru H teks" will work the best for extracting the amides from the seeds, but I would imagine that converting them to the tartrate salt in the end would allow for the best preservation of the amides for long term in the tartrate form....then store under nitrogen in a freezer....director of sound's A method, along with G and H above sound the most promising...looking among all the practical processes and picking out the similarities and doing comparisons and then going from there is what research is all about, find "all" that is written on the methods, then form a hypothesis, then do the research...the most difficult part of the whole process is collection of the initial material for the extraction, extracting the amides, then the hydrolysis and proper storage of the lysergic acid end result..which is only 35% yield apparently, there being allot of loss during the hydrolysis.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **lugh** on **September 18, 2013, 08:23:56 AM**

This old Hive thread may be helpful:

http://chemistry.mdma.ch/hiveboard/tryptamine/000027907.html

since it includes KrZ's technique:

http://chemistry.mdma.ch/hiveboard/novel/000108007.html

The end results from the effort applied 8)

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 18, 2013, 11:23:42 AM**

Thanks! you are the man lugh, much appreciated.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **harryboberries** on **September 18, 2013, 06:02:42 PM**

Tregar, it is good to see all this information. Thanks for sharing.
I think the hardest part of this would be the precursor. I see you talking about 100g of seed, which can't be easy to continually come by.
The way I figure, if an individual had readily available starting material, the rest would come with time and patience.

If one could do this using seed that would be great, but I think it would still be economically inefficient to use.
It is amazing the price good L can be had for, considering all the steps and chems involved.

In my estimation, the best, cheap, continual source for an obtainable precursor, would be a submerged culture of mutated c. paspali.
I happen to be in a region where this infection is prone, however c. paspali seems to be more difficult to identify than c. purpurea.
From there it would need to be cultured across potato dextrose agar in a petri dish.
A celligen device could be used for spawing the culture into, where supposedly Ergine is produced in heavy ratio.

Because Ergine is produced, instead of ergotamine, there would be no fear of gangrene. That is what I've come to understand.
I've read this method producing multiple grams per liter of usable startup goods.

When it comes down to this method, it would contend with mushroom cultivation in many respects.
If you could master basic mycology, the above should not be much of a challenge.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 19, 2013, 05:19:07 AM**

I totally agree, paspali would be the way to go. Seeds would be an interesting go for personal use, however, it's quite expensive, and there would be considerable loss at every step. Thanks for the info on paspali, I've done reading on it in the past, would love to hear more. This is pretty much all the info I have for quite some time, that and what lugh linked to.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Baba_McKensey** on **September 20, 2013, 03:04:40 PM**

Did you see Morning Glory's posts here?
https://www.hyperlab.info
/inv/index.php?s=02cc313fcbd6c49cf709f8b329390b70&act=ST&
f=17&t=30352&st=0

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 21, 2013, 03:18:57 AM**

Thanks Baba_McKensey. The link took to an "untrusted connection" for some unknown reason. Would you mind posting his method here? I tend to like director of sound's method A, so long as you keep PH values in check--the method is very similar to "H" by Kash (also very good)--both follow the same general procedure. Would love to see the above link if you could post it here. Looking at all the processes, you see the similarities and differences and things to watch (such as PH), that's why I think it helps to have a whole series of methods listed...then the pieces all start falling together.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **lugh** on **September 21, 2013, 05:25:58 AM**

An archive of the Hyperlab report is attached ;) Moonlight et al are old friends from the days of the Hive 8)

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 23, 2013, 06:54:48 PM**

Forgot to mention that morpholine (as an alternative to diethylamine) is readily out there, seen it at many places. Morpholine and many other derivatives are talked about in the attached paper, along with receptor profiling from Dr. Nichols.

This is about all the information I have, it's been an interesting study and read, but that's all it is, a whole lot of rambling and information all in one place.

Quote

> Morpholine could also substitute in place of diethylamine for lysergic acid morpholide, 100ug of it is equivalent to 75ug of acid, however, it only has a 1 hour peak instead of the 4 hour peak of acid...it also only lasted 5--8 hours. It is shorter lasting. "although the morpholide had less than 1/10th the potency of LSD in blocking the action of serotonin, it did however, have nearly 75% of the potency of LSD as a psychedelic" (gogerty/dille 1957)

...Nichols in his paper "lysergamide cousins of LSD" talks about it as well.

Otto Snow page 46:

Quote

> LA-111 also called ergine (d-lysergic acid amide) is the active constituent of an Aztec entheogen called Ololuiqui. It is a feeble psychoactive which is active in humans at 1 mg. LMP-55 (lysergic acid methylpropylamide) has been reported to be
> less then 25% the activity of LSD-25. (Abramson.) LSM-775 (d-lysergic morpholide) is active between 300 to 600 ugs. (Grogerty 1957).

Quote

> LSM-775, N-Morpholinyllysergamide. There are conflicting reports; one states that 75 micrograms is an effective dose, comparable to a similar dose of LSD, and the other stated that between 350 and 700 micrograms was needed to elicit this response, and that there were fewer signs of cardiovascular stimulation and peripheral toxicity.

I've read that perhaps it is more dreamy/dxm like than LSD:

Quote

> N-Morpholinyllysergamide (LSM-775) is a derivative of ergine. It is reported to have some LSD-like effects at doses ranging from 75 to 700 micrograms. LSM-775 is said to produce dream like states similar to DXM. Its visual component is like that of LSD, but it is said to be less intense and not as pronounced as LSD.

In addition, the following paper which I have a hardcopy of but no file "David E. Nichols, "Lysergamides of Isomeric 2,4-Dimethylazetidines Map the Binding Orientation of the Diethylamide Moiety in the Potent Hallucinogenic Agent N,N-Diethyllysergamide (LSD)",Journal of Medicinal Chemistry, 2002, Vol. 45, No. 19, 4344--4349:"

list several additional analogues that are quite similar to LSD...not only that but lists their strength at all the receptor sites as compared with LSD (5ht2b, 5ht1e, 5ht1d, 5ht5a, 5ht6, 5ht7, d1, d2, d3, d4, d5, h1, A1, A2, etc.)

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Mango** on **September 24, 2013, 01:06:30 AM**

Key thing about morpholine is "There are conflicting reports".

Nichols' PyBOP paper.
http://nelix.id.au/papers/unsorted/www.murple.net/nichols/nichols-dimethylazetidine-lysergamide.pdf

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Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **September 24, 2013, 07:46:51 AM**

Yeah, doesn't sound interesting to me anyways....sounds very dreamy and short lived.

Anyhow, i've lost interest in all these LSD ramblings.

I fell in love with properly prepared ayahuasca/pharmahuasca

in other words: 70mg harmine freebase + 35mg Tetrahydroharmine + 40mg dried dmt paste

please see here if interested:

https://www.thevespiary.org/talk/index.php
/topic,3892.msg37980.html#msg37980

As you can probably tell, I have ADHD, but I don't take drugs for it, the only drugs I take are psychedelics and 2 diet dr. peppers in the morning ;D

Title: **bla**

Post by: **fishinabottle** on **October 02, 2013, 11:50:32 AM**

its just state of the at that pyBOB is a really good coupling agent for LSD, if you dont know how to use it read the regarding literature, its outlined there so often....

Whats the fuss about?
Ergotamine from seeds?
What a meme.
Read the regarding literature and you know why.

over and over and over and over and over again.....

get a real life or just start reading.
if you dont understand the articles hire somebody who does or just skip it.
an education is something you have to have before you start thinking about this.

alternativly: meth from gunblueing sounds as good as the posts on the boards on LSD lately...
just more promising.

/ORG

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **October 03, 2013, 04:34:51 AM**

Concerning DEET to diethylamine:

Yes, we are talking DEET bug spray here, 100% DEET is available at *almart and camping stores in the US, the 100% deet affords protection from bugs and trolls for up to 12 hours as opposed to the 20 to 30% variety which allows protection for several to 6 hours.

You hydrolyze it in a mixture of 70% etoh/30% water and Naoh, inside a 250ml autoclavable container (the kind that is heat and chemical resistant), screw the lid on, shake it up every now and then, let it sit for a day or more inside a water bath in a crock pot that is set to a certain high temp, then transfer the container to a fridge to cool down, once it is completely cool, then you carefully open it, pour out the contents into a distillation setup, and pull off the 1st fraction from 131 to 133 degree F, this will be your diethylamine. You want your etoh/h20 mixture to be about 70%/30% as it will allow just enough water for the hydrolysis without allowing seperation of the chemicals to occur. If the hydrolysis completed successfully after a day, then when you open the container you will smell diethylamine strongly instead of deet.

Usually hydrolysis is a chemical process in which a molecule of water is added to a substance. Sometimes this addition causes both substance and water molecule to split into two parts. In such reactions, one fragment of the target molecule (or parent molecule) gains a hydrogen ion.Acid–base-catalysed hydrolyses are very common; one example is the hydrolysis of amides or esters. Their hydrolysis occurs when the nucleophile (a nucleus-seeking agent, e.g., water or hydroxyl ion) attacks the carbon of the carbonyl group of the ester or amide. In an aqueous base, hydroxyl ions are better nucleophiles than polar molecules such as water. In

acids, the carbonyl group becomes protonated, and this leads to a much easier nucleophilic attack. The products for both hydrolyses are compounds with carboxylic acid groups.Upon hydrolysis, an amide converts into a carboxylic acid and an amine or ammonia. The carboxylic acid has a hydroxyl group derived from a water molecule and the amine (or ammonia) gains the hydrogen ion. The hydrolysis of peptides gives amino acids

diethylamine bp = 131--133 F

ethanol = 173 F

M-toluic acid = 505 F

any leftover deet (N,N-Diethyl-M-Toluamide) bp = 550 F

DEET is shown below and diethylamine below it:

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **October 03, 2013, 07:25:28 AM**

This is what an inert gas tank looks like (N2 tank), and this is what an oxygen only regulator looks like, notice the adapter that fits between the n2 tank and the o2 regulator, this adapter (coming out of the right side of the regulator) is required to fit the two together. The meter gauge at the top right tells you what the internal pressure is inside the tank, the meter gauge to the left of it tells you the psi that you are letting out of the tank, you allow gas out of the tank by turning the handle screw clockwise slowly tell you get to the desired constant psi you want to have come out. To turn the gas off, you quickly turn the handle screw counterclockwise tell the psi out gauge reads 0.0, the meter gauge at the top right will still read the internal gas pressure no matter what position the handle screw is in, so you always know how much gas is still in the cylinder. A cylinder that goes up to about chest level will read about 15000pka or 2000 psi on the needle on the internal gas pressure gauge when the tank is completely full.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **October 04, 2013, 05:44:23 PM**

Alkaline Hydrolysis of N,N-Diethyl-meta-toluamide---->parent compound + diethylamine

To better understand why director of sound used crockpot temperatures (and in the first example, a day long heating in the crockpot) close to the boiling point of ethanol (170 F) to hydrolyze DEET to it's parent compound and diethylamine, it's helpful to look at this student lab experiment on the hydrolysis of the simplist of amides, acetamide to it's parent compound and amine.

Alkaline Hydrolysis of acetamide------->parent compound + amine

Experiment 8 (Organic Chemistry II) Carboxylic Acids Reactions and Derivatives
Quote

> Of the various acid derivatives studied here, amides are the least reactive toward nucleophilic attack, because the unshared electron pair on the nitrogen is delocalized to the carbonyl carbon through resonance.

> Thus, unlike most acyl halides or anhydrides, amides must be heated to boiling with aqueous acid or base in order to hydrolyze them. For a primary amide, the product will be ammonium ion and the acid (for acidic hydrolysis) or ammonia and carboxylate ion (for alkaline hydrolysis). The reaction mechanisms are similar to those for the hydrolysis of other acid derivatives.
>
> In this experiment, you will hydrolyze acetamide under alkaline and acidic conditions.
>
> Hydrolysis of an Amide
>
> Add 0.5g of acetamide to 5ml of 10% sodium hydroxide solution in a test tube, and gently warm the solution and heat the mixture to boiling. Note the odor of the evolved gas by gently wafting its vapors toward your nose. Test the gas by holding a piece of moist red litmus paper in the mouth of the tube. Repeat the experiment with 0.5g of acetamide and 5ml of 10% sulfuric acid. Record the result, and write a complete reaction(s).

Acetamide:

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **October 06, 2013, 07:12:50 PM**

The reason 70% ethanol and 30% water was used by director in the base hydrolysis is that DEET is mostly insoluble in water. The solubility of DEET in water is only 2-3mg at 68F (20C)

unknown (2009) wrote:
Quote

> Did an acid hydrolysis instead of a base hydrolysis:
>
> Added 125 mls of 100% DEET to a 30% dilute sulfuric acid solution.
>
> Once the PH was at 3, refluxed it for 8 hours at around 160 degree F (70C or so).
>
> After the reflux, added distilled water to the solution and after seeing that it did not form two layers, knew the hydrolysis was done.
>
> Then added KOH until the mixture was at ph=7 (any where from 7 to 10ph should do).
>
> then distilled the solution with ice cold condenser into an ice/salt chilled receiver round bottom flask.
>
> The diethylamine came over at around 57C (55--59C) (131-138F) It has a very similar boiling point as that of acetone, and when a tiny bit was left out, it quickly evaporated into thin air...it has to be stored in a dark vial, stoppered tightly and kept cool & dry. It has an irritating ammonia smell, heavier than air and very volatile...best to work with it under a fume hood, if not wear a face mask around it.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **myCH3** on **November 22, 2013, 05:47:58 PM**

Just to add a little something that you left out of your ramblings (they are awesome by the way thank you). It seems there is a convenient method two step through the ester that forms the isomers that are wanted. https://www.erowid.org/archive/rhodium/chemistry/lysergic.amides.html Once the ester is in hand it seems that it just has to be heated in a closed system with the amine.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **December 10, 2013, 05:39:47 PM**

Excellent link there on the ester w/amine, thanks myCH3, keep up the good work

and thanks for the kind words....p.s. once you have diethylamine and your peptide coupler, the rest is really not all that hard...perseverence is the key.

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **Thiophenechemist** on **December 10, 2013, 10:21:05 PM**

Hello all,

My RANDOM suggestions are as follows- take note that these methods are not as EXTREME as they may initially seem and that the stability of the LSA SALT is one of the major problems. For this I propose VARIOUS ideas.

IDEA 1:
1a. defat and follow a typical procedure for oil extraction of total alkaloids
OR
1b. defat and soxhlet extract the mass with DCM/1-2mol eq triflic acid/toslic acid

2. total crop of alkaloids is left to react in a solution of dcm and 1-2 mol eq. triflic acid
3. the triflic acid will cleave all NH-R bonds of ergine, ergometrine, lsa alpha OH etc... leaving LSA in HIGH YIELD
4. the obtained dcm solution now has LSA-TRIFLATE or TOSYLATE
5. the solution is washed with water to remove impurities- the LSA-TRIFLATE, or LSA-TOSYLATE salt has a high chance to be SOLUBLE in the dcm still, if the LSA TRIFLATE is in the water then basify with bicarb etc
6. basify the washed dcm LSA-triflate soln w bicarb
7. column for LSA TRIFLATE OR LSA TOSYLATE fraction

IDEA 2:
To an aqueous soln of the alkaloids(extracted by whatever whichever lets assume methanolic KOH),
1. a calculated molar equiv of TOSYL CHLORIDE is added, in a base (not pyridine, if you can use methanolic KOH be my guest)
2. the LSA- SULFONAMIDE will be the only to precip- as it can be the only one to form the adduct(i.e tosylates hate forming TERTIARY amines initially)
3. the LSA TOSYL SULFONAMIDE is crystalline and stable and easily recryst'd or column'd.
4. the TOSYLATE can be cleaved by hcl, sulfuric etc HOWEVER NOT WHAT WE WANT TO DO.

5. The LSA TOSYL SULFONAMIDE is subjected to exhaustive ETHYLATION with 2, 2.5, or 3 eq of ETHYL CL, and a weak base i.e any carbonate salt
6. THE FORMED LYSERGIC ACID DIETHYL TOSYL SULFONAMIDE CHLORIDE SALT (+ charge on nitrogen) is recrystallized via etoh
7. THE FORMED LYSERGIC ACID DIETHYL TOSYL SULFONAMIDE CHLORIDE SALT is cleaved of the sulfonamide via TRIFLIC, maybe tartaric acid, or sulfuric acid,

http://www.ff.ul.pt/FCT/PTDC/QUI-QUI/105104/2008/Ref20.pdf

there are many ways to cleave sulfonamides i.e base, acid(tfoh reccomended), HYDRIDE

The cleavage and safety of the LSD in solution after hydrolysis of the amide is

CRUCIAL, there are methods however, that utilize bases acids, or even hydrides that will promote a healthy medium for the LSD, I would be curious of creation of stabler salts than tartaric i.e TRIFLATE OR tosylate salt is an excellent idea very stable.

To Summarize:
1. LSA- SULFONAMIDE ADDUCT
2. EXHAUSTIVE ETHYLATION
3. CLEAVAGE OF ADDUCT in a FRIENDLY medium for the PRODUCT

Acid catalyzed orgo is a tricky field, but VERY USEFUL

Title: **Re: seeds to LSD, comprehensive tek & ramblings**
Post by: **tregar** on **December 16, 2013, 09:49:18 AM**

Thanks Thiophenechemist, excellent knowledge there, much appreciated.

Been doing some research on peptide couplers, and it appears they work by turning the 300mg of lysergic acid into an "ester". This ester is formed when the 640mg of (for example pybop) coupler is added to the lysergic acid monohydrate in 100ml of Ch2CL2 (dichloromethane). Once this has begun to spin for a short time (10--15 minutes), then 300mg of diethylamine is added, which is allowed to spin for an additional 60 minutes....with most reactions completing in less than an hour, although it can be allowed to spin for up to 3 hours when using less efficient couplers.

There are a variety of peptide couplers that activate carboxyl groups, this is just one quote for example (shown from millipore): "hatu is a highly efficient coupling reagent for solid and solution phase peptide synthesis. In comparative studies hatu has been found to give better coupling yields with less enantiomerization that hbtu, tbtu or pybop® [1-3]. It is particularly effective at coupling to N-alkyl amines where other coupling reagents give poor yields and is the preferred reagent for loading resins bearing secondary amino groups."

From wikipedia on peptide synthesis:
Quote

> Activating groups
>
> For coupling the peptides the carboxyl group is usually activated. This is important for speeding up the reaction. There are two main types of activating groups: carbodiimides and triazolols. However the use of pentafluorophenyl esters (FDPP,[12] PFPOH[13]) and BOP-Cl[14] are useful for cyclising peptides.
>
> Carbodiimides
>
> These activating agents were first developed. Most common are dicyclohexylcarbodiimide (DCC) and diisopropylcarbodiimide (DIC). Reaction with a carboxylic acid yields a highly reactive O-acylisourea. During artificial protein synthesis (such as Fmoc solid-state synthesizers), the C-terminus is often used as the attachment site on which the amino acid monomers are added. To enhance the electrophilicity of carboxylate group, the negatively charged oxygen must first be "activated" into a better leaving group. DCC is used for this purpose. The negatively charged oxygen will act as a nucleophile, attacking the central carbon in DCC. DCC is temporarily attached to the former carboxylate group (which is now an ester group), making nucleophilic attack by an amino group (on the attaching amino acid) to the former C-terminus (carbonyl group) more efficient. The problem with carbodiimides is that they are too reactive and that they can therefore cause racemization of the amino acid.
>
> Triazoles
>
> To solve the problem of racemization, triazoles were introduced. The most important ones are

> 1-hydroxy-benzotriazole (HOBt) and 1-hydroxy-7-aza-benzotriazole (HOAt). Others have been developed. These substances can react with the O-acylurea to form an active ester which is less reactive and less in danger of racemization. HOAt is especially favourable because of a neighbouring group effect.[15] Recently, HOBt has been removed from many chemical vendor catalogues; although almost always found as a hydrate, HOBt may be explosive when allowed to fully dehydrate and shipment by air or sea is heavily restricted. Alternatives to HOBt and HOAt have been introduced. One of the most promising and inexpensive[citation needed] is ethyl 2-cyano-2-(hydroxyimino)acetate (trade name Oxyma Pure), which is not explosive and has a reactivity of that in between HOBt and HOAt.
>
> Uronium based peptide coupling reagents
>
> Newer developments omit the carbodiimides totally. The active ester is introduced as a uronium or phosphonium salt of a non-nucleophilic anion (tetrafluoroborate or hexafluorophosphate): HBTU, HATU, HCTU, TBTU, PyBOP. Two uronium types of the coupling additive of Oxyma Pure is also available as COMU or TOTU reagent.

The reaction is then quenched by adding 100ml of 7.5M concentrated NH4OH (ammonium hydroxide). The DCM layer is seperated out and the aqueous phase extracted x 3 times with 30ml dcm, the organc solvent layers are combined and evaporated under high vacuum down to a residue. This residue is dissolved in 40ml of cold NaHCO3 (saturated sodium bicarbonte water), and extracted x 3 times with 20ml of dcm, the organic layers are all combined and washed with water, saturated salt water, and then dried over mgSO4 (magnesium sulfate), the solvent is then filtered and then the solvent is evaporated off under vacuum to leave behind a crude LSD residue. To the 1 equivalent of the base is added 1-equivalent of d-tartaric acid along in a little methanol to salt out. The neutral tartrate of the diethylamide of d-lysergic acid crystallises in needles united in bundles. The salt is very readily soluble in water or alcohol.

Easy method to diethylamine: go down to *ally world, look for the 99% deet bug spray, add the stuff to 30% sulfuric acid made by diluting otc 98% sufluric acid plumbing product to distilled water-->reflux it for 8 hours, then let it cool, then distill the solution-->the first thing to come over will be the diethylamine at 131-132 degree F, capture it in a chilled receiver...as the stuff is like acetone--it evaporates super fast if left uncapped in the freebase form. Store cold in light proof container...for more info look for the post by "madhatter" some many years ago from sciencemadness...this is the acid-based-hydrolysis of deet...easier than the other method posted by director of sound and the multiple failed attempts in the "other" (base-hydrolysis) 15 page thread at sciencemadness. This is the link that works: http://www.sciencemadness.org/talk/viewthread.php?tid=12822

AgentMadhatter:
Quote

> Ok. After thinking the thread was done by confirming all I needed was an amber bottle with a proper seal...I didn't look back until today. If you guys honestly don't believe this was my procedure.
>
> I got a lot of bottles of 30ml of 98% DEET and 2% other (Suspected Alcohol since it allowed water and the DEET to mix)
>
> I threw in 500mls of the DEET with Dilute Sulfuric acid at a 28% concentration. Once the PH was at 3, I refluxed for 8 hours at 70C, using water to prevent any evaporation.
>
> After adding distilled water and seeing that it did not form two layers, I knew the hydrolysis was complete. At which Point I added KOH until the mixture was at 7 pH.
>
> Distilled off the DEA through my condenser into a cooled container of HXl in EtOH. Forming DEA.HCl.
>
> I have the liquid sitting in an amber glass. Trying to figure out if letting it evaporate under a vacuum will cause any DEA vapors or anything.
>
> That was my procedure.
> ---

> I'm positive I have it, because I took off the acid and made it freebase and watched my product disappear into the air.
> ---
> Like I said...I'm not chemistry major. If I get product, thats good enough for me. I don't know how to calculate percent yield compared to theoretical yield, I do know the BP was around 60 C but not over it. 55-60C.
>
> But whats the best way to get the DEA HCl crystals out of the ethanol now? Just evaporation? Or will the DEA evaporate?

entropy51 said:

Quote

> Fantastic! So it was an acid hydrolysis rather than base. Good to know.

Panziandi said:

Quote

> Personally, Agent MadHatter, I would have made the reaction mixture far more basic than pH7! I would have added KOH until I got pH10 or something, using saturated aqueous KOH.

You can distill diethylamine after an acid hydrolysis of DEET, but only of course, if you basify before distilling. The point is, that it works, and he was the first to achieve success and post it to a forum.