





A. S. Shulgin

Clean notes: Book #8

Many found during post-Thursday clean-up.

Thursday, October 27, 1964 - the BIG day!

~~known~~ ~~attempts~~

H<sub>2</sub> → overcast 161

H<sub>2</sub> → Hd 133

5- substituents

5-MeO-DIPT 106, 109, 124, 147, 265

5-MeO DAET 264

Tryptamines:

NSS  
or  
NMRs  
↓

Peganum harmala: 48  
Tetrahydroharmane: 44

DSBT 58  
GIPT 56

$\alpha, \beta$ -TMS 5

NMT 22

MIPT 32, 125

MSBT 34

MPT 36 (DPT) 938485

DAET 38 (NAPT)

NET 54, 64 (di)

NSBT 58 (DSBT)

NIPT 60 (T)

NPT 62 (DPTXT)

BMT

66 (T, NMT, carboline)  
70-71, 1-methyl, carboline

NOC

pip

pyc

102, 104

S-meO

~~132~~ wip

T 152 wip.

MIPT great 125

Chc's:

Red-flowered T. grandiflorus 137  
Yellow-flowered T. grandiflorus 147  
Red T. grandiflorus #2 148

5-MeO-pyr-T 120

w/lyc isoginsins

6,7(CMF)<sub>2</sub> (38 amide)

134, 141

140 → dihydro, try

142 wip

143

143 → amine 162  
activity

6,7 MD0 amide 145A side 150

6,7 MD0 ~~NSBPA~~ NSC → 110

→ amide 111

DMHT-4

MIPT → Grande TMT

→ amide → DMHT 131, 132

→ amine 134, 135





2.0 g indole carbonylalkaldehyde  
 25 g EtNO<sub>2</sub> Δ → sol.  
 0.5 g NitroAc

SB spl ~ 2 min 1/2 hrs done by TLC shift

Reflux f. 20 ml boiling IPA, ▽, filter, wash  
 with dry → 1.56 g 54% 54% mp 179-80°

MLs 0.81g

xtal f. 10 ml EtNO<sub>2</sub> Δ ▽

→ dull gold xtabs

0.57 g mp 178.179° Σ 2.13 g 78% theo.

lit 182-184° mp. reference ③

repeat

1.21 g Aceto

15 g EtNO<sub>2</sub>

0.39 g NitroAc - on EtO 16:20 [120] v. dark already.  
 off (1:15)

↳ 1.58 g v. dirty red xtabs.

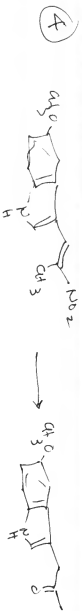
filtered on 25 ml denatured EtOH Δ to Sol. ▽ O/N

filter - wash i small amt IPA -

→ ML → sweep  
 solution

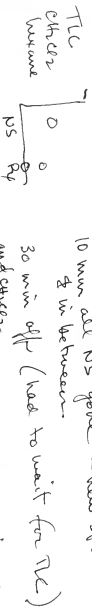
air dry. 1.24 g old gold xtabs

③



8 ml H<sub>2</sub>O<sub>2</sub> } on S.B. add  
2 ml H<sub>2</sub>O

1.7 g electrolytic iron dust, when hot, add  
6.5g nio-nis a glob at a time, over 2 minutes  
10 min add NS grease - new starts at origin  
& in between.



Into 100ml H<sub>2</sub>O - 2 flasks and ~~separated~~ indistinctly  
filter through paper - separate, 2x50 ml extract - wash  
̄ sat K<sub>2</sub>CO<sub>3</sub> - filter → 0.2g film.

KR at 0.1 μm. 130°  
140°  
150° (vac to 0.05)  
215-230° over → 0.24g oil - off white

IR c=O at 1710 cm<sup>-1</sup> weak other OK.  
stand a couple of minutes → keep brown color.

### Repeats:

30 ml H<sub>2</sub>O<sub>2</sub> } Δ SB ~ 60°. add  
5 ml H<sub>2</sub>O  
4.55g Fe electrolytic - up to ~ 70° - bubbling, add  
1.24g nitrobenzene - over 2 minutes - color discharge  
5 minutes off. - into 250 ml H<sub>2</sub>O, 50 ml CH<sub>2</sub>Cl<sub>2</sub>  
Filter through paper - wash ̄ CH<sub>2</sub>Cl<sub>2</sub>. separate - separate - dist off  
̄ 2x50 CH<sub>2</sub>Cl<sub>2</sub> - combine, flash → 1.17g amaran oil - dry at  
at the end small

Stand 3 days in cold lab → stands! wip out w/ 75° - polymer?  
8:4

TLC (0.15 μm)  
OK  
benzene

5

1, N, O-TMS



0.22g Ketone (4) in  
 2.5 mL IPA  
 add  
 + 0.4 mL 40% aq.  $\text{NH}_4\text{Cl}$   
 + 0.3 mL 25%  $\text{NaOH}$   
 stand a week.

0.3g Al strips  
 0.3g  $\text{HgCl}_2$  soln (20g/mL)  
 20 min incubate.

try again, with  $\text{NaOH}$  on the solid material 8:4

To 4.3g  $\text{H}_2\text{O}$ , the in an E-Flask - stirrer - add

20 mL  $\text{NaOH}$ , add

1.17g Ketone 8:4 - grind in methanol - nearly all in OK  
 0.5g  $\text{NaOH}$  (pH green - to yellow with <sup>about</sup> 1.1 the, next one.

into dilute  $\text{H}_2\text{SO}_4$  - wash & extract  
 1 x 20 mL water

5:00 - 7:40 - 8:05

to 150°

Evaporate to 0.76g.

distill - 0.15mm

then 0.05mm.

to 160° - then over. - clear white oil

0.65g white oil.

rinse out (ether, near, then)

→ 0.58g pale yellow solid

let evaporate

(5B)

transfer to storage vial -

0.41g (5B)

6:00pm - 7 drops.

6:15pm 5 more

2PM next day 26 drops

2PM next day 3 drops.

2PM next day 2 drops only

then 20 min

play IPA,  $\text{CO}_2$  no strips

play L-malic acid, IPA

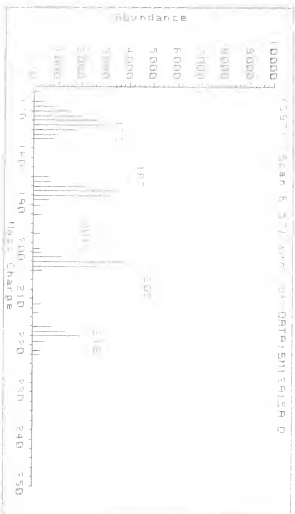
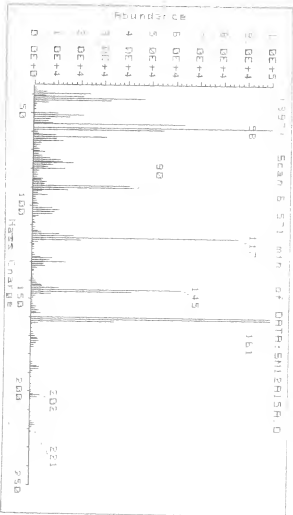
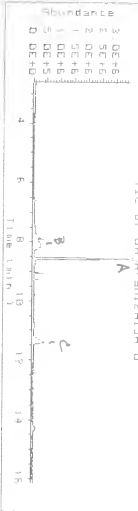
no strips

let stand

(5C)



6



MW 218

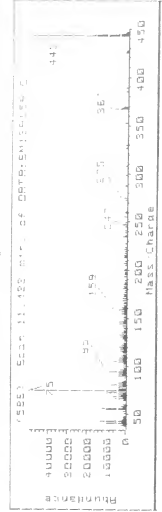
HC=CHCH<sub>3</sub>

CH<sub>3</sub>

58



4/78



SIM of 203 → no ketone.

Handwritten notes at the bottom of the page, including '4/25', '5', and '160'.



Properties of the cellulosines:

Methylone:

IR:  $\text{C}=\text{O}$  1680  $\text{cm}^{-1}$  fingerprint. 720, 748, 771, 932, 1043, 1056, 1127

(148)

mp: chunky 220 (amber) shr. 230 mp 231-235  $\bar{\text{c}}$  dec. bubble

Sample 60g  
Nova Screen C

G.C.M.S.: cracks  $\sim$  60/40 149/58  $\rightarrow$  207  
149/56  $\rightarrow$  205 dehydro

See page 10, 11 MW 243

Dieting one:  
(148)

IR:  $\text{C}=\text{O}$  1678  $\text{cm}^{-1}$  fingerprint: 754, 846, 935, 981, 1041, 1086, 1110

mp: 150 amber 200 darker 204-~~205~~ 205  $\bar{\text{c}}$  quite sharp dark

Sample 15.88  
Nova Screen D

G.C.M.S.: cracks  $\sim$  70/30 149/100  $\rightarrow$  249  
149/98  $\rightarrow$  247 dehydro

See pages 12, 13 MW 330

t-BUTYLONE

IR:  $\text{C}=\text{O}$  1651  $\text{cm}^{-1}$  fingerprint: 989, 941, 1046, 1111

mp: 200 tan shr. 240  $\bar{\text{c}}$  mp  $\bar{\text{c}}$  dec 247

Sample VIII-107  
Nova Screen A

G.C.M.S.: cracks  $\sim$  50/10 149/110  $\rightarrow$  249

149/98  $\rightarrow$  247  
See pages 14, 15 MW 285

ACRIDINE (vacuic)

Sample I-113  
Nova Screen B

Dane

fluorocalls

#2 April 14 1995 - OK -  
We will alert we re

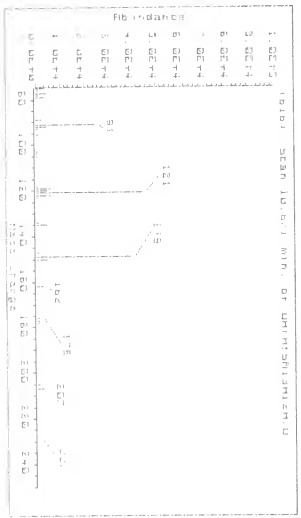
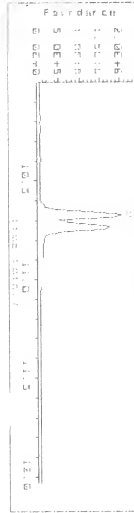
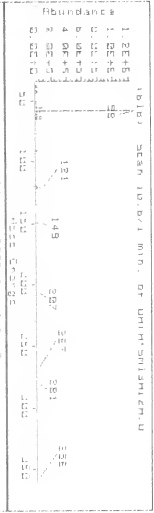
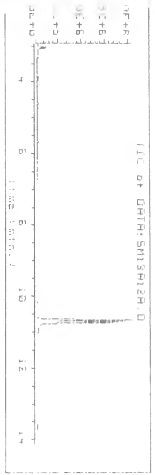
Pablo sources.

\$110 for micro analyses.

MW 259

110

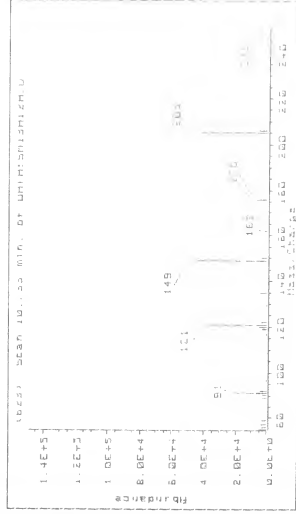
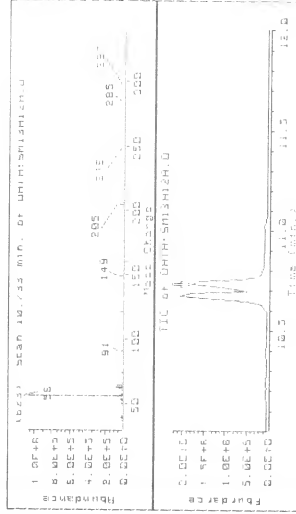
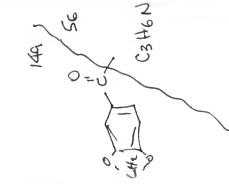
from methylene GCMS



mw 207

(11)

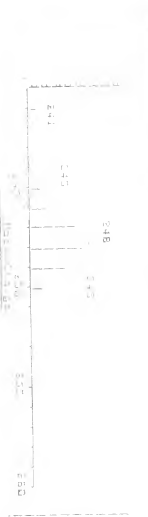
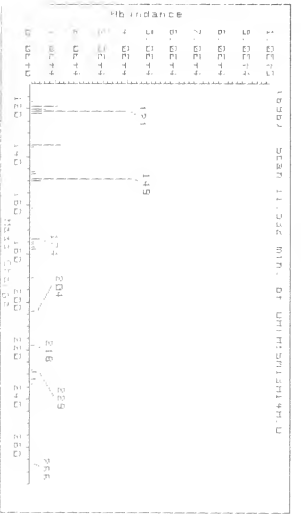
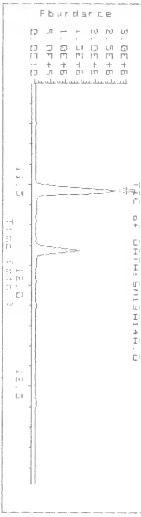
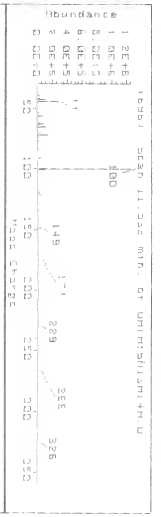
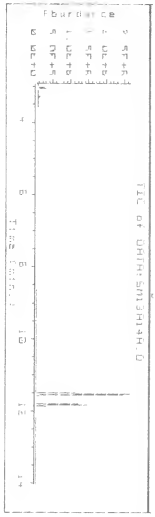
Nov. 205



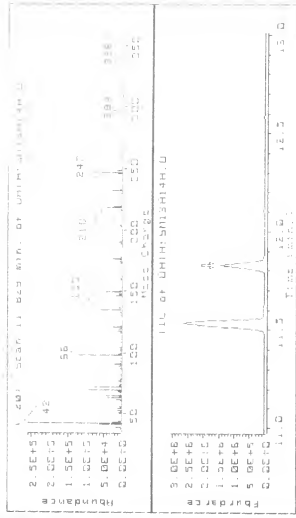
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(2)

DIETHYL LOSE . See page 9



UVA



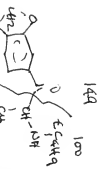
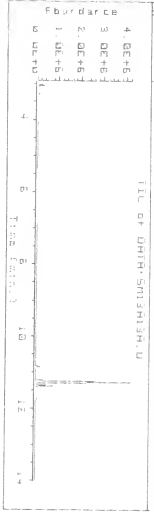
144 mw 247



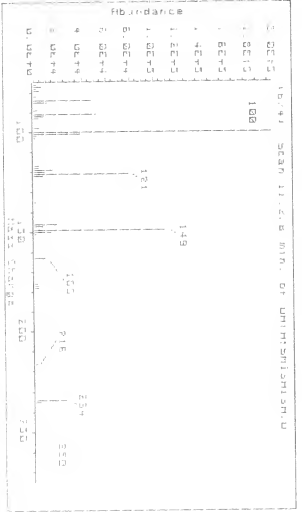
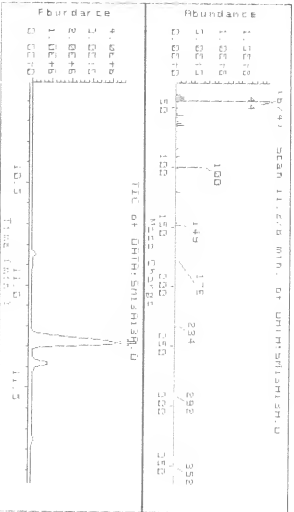
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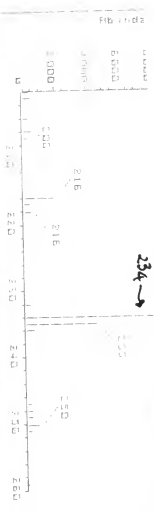
NA



MSD 249



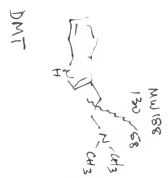
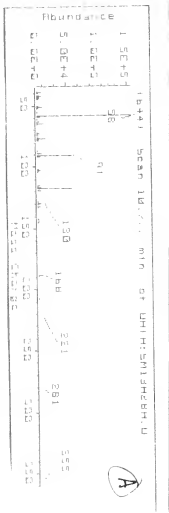
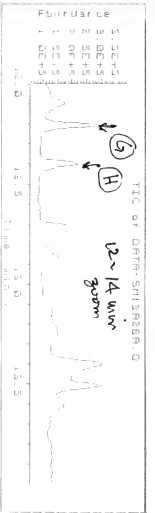
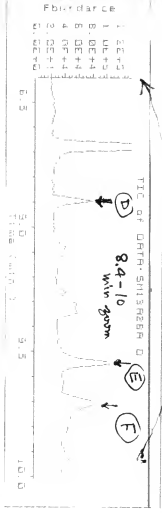
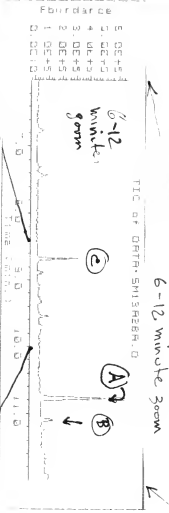
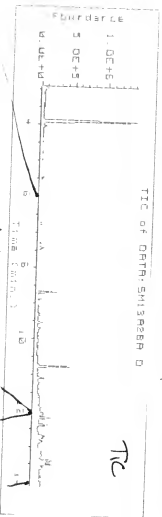
MSD 249





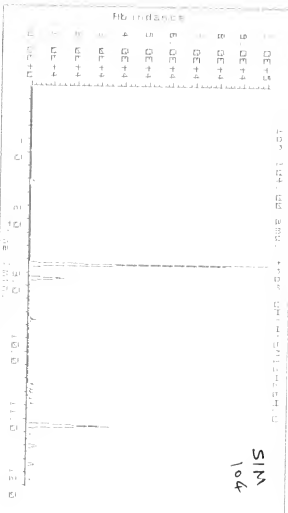
Al

AcaciaSophora, alkaloid fraction, or D. S. salt

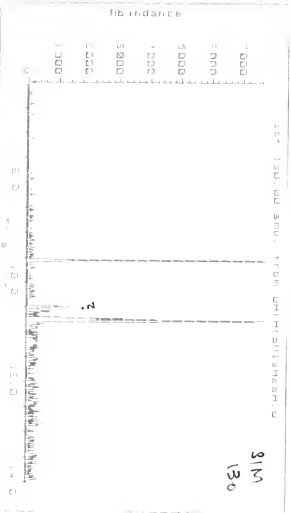




16



MWT  
DMT  
perhaps was  
often undetermined

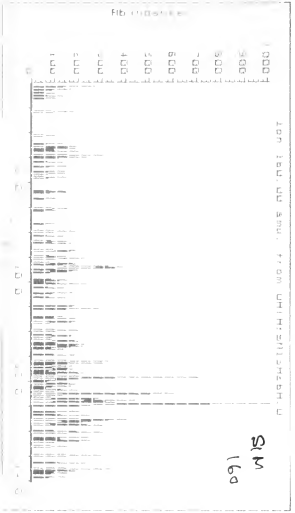


no hint of



no S-MeO-DMT  
S-Nitro-DMT

DMT  
S-MeO-DMT  
S-Nitro-DMT  
DMT



119



120  
12  
48  
180  
12  
16  
216  
192g  
crude

10 g tryptamine - absd  
10 g Butyric formate

↳ to reflux in RB - mantle - 24 hrs → dark, clear solution.

strip on PE → 19-2g viscous oil.  
into 250 ml H<sub>2</sub>O.

acidify & HCl (strip in blue-top NOT HCl) (big 6 lb bottle OK)

xrt 3 x 25 ml ether  
purpl - wash 1 x die HCl. 50 ml.  
wash 1 x 5% NaOH 50 ml

strip on PE. to hard vacuum → (1.5g black oil.  
into V.R. Vbr to 0.05 mm. distil  
220-240° over - best vacuum tubing?

11.5g crude  
1150 washing  
1350 .06 min. water  
490  
1450  
1600

Almost color. Still strip in hot.  
pink to 250° - no residue in hot.  
not xrted ON - some will oil to start.

Let stand - very hot weather. Very slow xrtals.  
GCMS shows some inverted tryptamine -  
and, strongly - hints of di-CHO ing.

90% over.  
6 (Blue)  
0.05 empty

a week later - slow growth of more xrtals

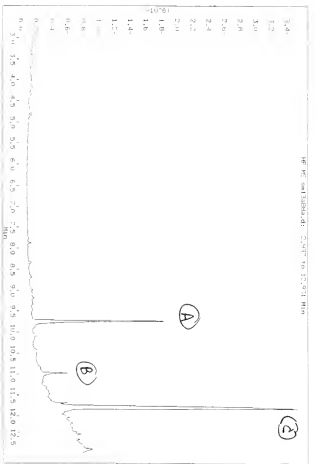
Add ~30 ml Me<sub>2</sub>SO  
reflux 0.5 - 1 hour. now a simple formate peak - BUT  
a smt at 3 minutes? strip hard → 77 g. H<sub>2</sub>O

(purple paper 20, 21)

170° spot.  
190° done } → 6.87g pale yellow oil  
0.1 mm

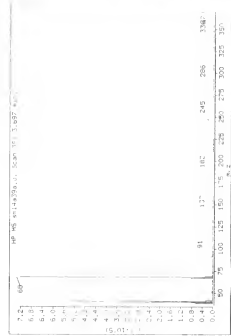
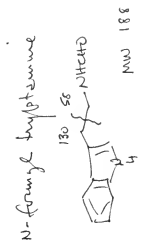
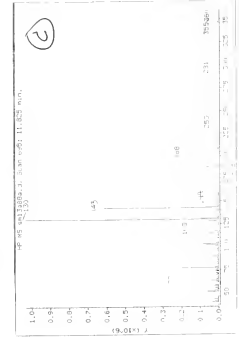
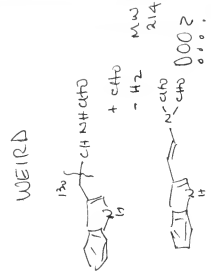
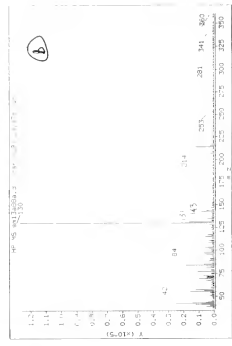
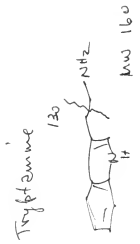
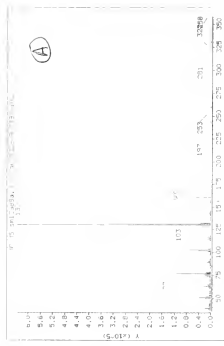
(20) Spectra of C1=CC=C(C=C1)C(=O)OCC from page 19.

Crude, showed impurities yellow oil,  
 from page 19  
 Then, pure dihydrate - no TDM layer



12/18

(21)



early!  
 ???



22



In a RB 3 neck flask - Argon - stirrer, reflux, good gas flow. Add 100 ml 1M LHM in THF. At room temperature. (60 min)

Make a solution of 1.88 g Formamide in ~ 20 ml aq. aq. aq.  $\Delta$  LHM to near reflux, add ethan solution of diphenyl - 2 fold excess of LHM at next generation.

Up to reflux in evening - good stirring, steam distillation of white surface scab - RM - still reflux (stems? not THF) and a lot of white solid.

Remove a squirt of THF suitable for ACSIS analysis  $\rightarrow$

RM - to RT. 24 hrs later - kill with

30 ml 50% aq. THF. no more figs.

Filter, wash  $\times 4 \times 25$  ml damp THF.

Check pH of filtrate - damp pH paper. DMSO

Scrap Filtrate + washes to dryness in ~~the~~ rot. evap

$\rightarrow$  1.39 g pale oil. starting to decolor to

acid of base - start of spontaneous xtal. -

Sour Seed -

Distill at RR

0.11 mm / 60° no

110° sharp

120° wide bands

135 - 145° men. white oil.

1.22 g - slant. & immediate xtal. to white solid.

4HTT NMT

1.1.1.1.1.1

Conversion to the monomer - heat up 90°

0.22 g base (no wt taken) (flaming easily burning) + 1g IPA

$\Delta$  puts it into solution. add conc. HCl -

acid drop  $\rightarrow$  green - blue color. Heat dissolution on

stirring - when acidic - color stable - no xtal. -

add a  $\frac{1}{2}$  mg of HCl  $\rightarrow$  clearing - stir - smooth  $\rightarrow$  xtal

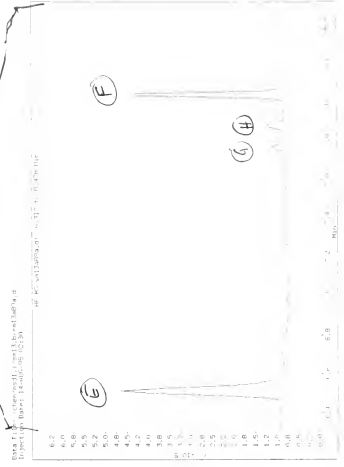
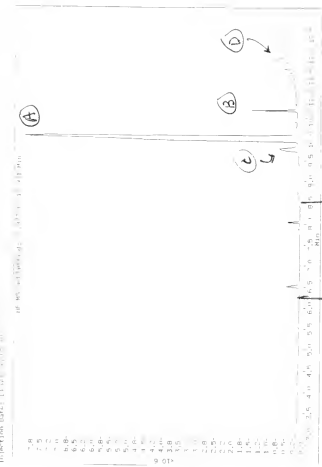
After yellow wash then other wash - dry to constant weight

IPA

4HTT 0.116 g.  $\rightarrow$  (gray Hmg?)  
 0.116 g. of blue xtal. (Hmg?)  
 MW-174.6

GEMS of course, sublog, of 22.

(23)



See  
 separate  
 analysis  
 of  
 (A)  
 in  
 page 26

activity

174

in file

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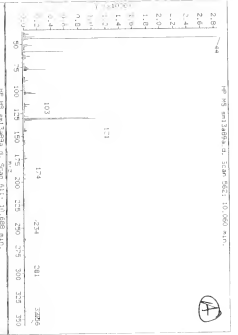
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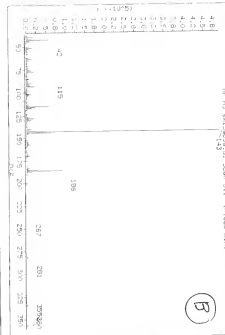
with

24

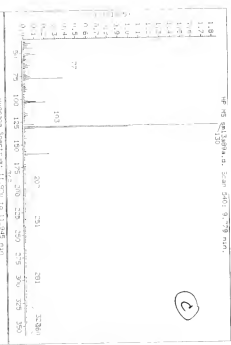
from page 22-23 under MT



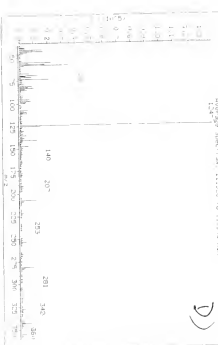
(A)



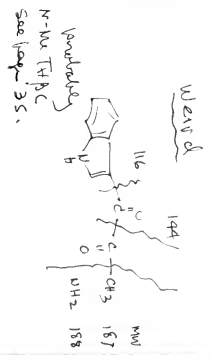
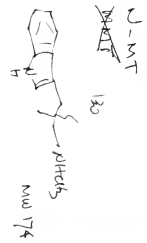
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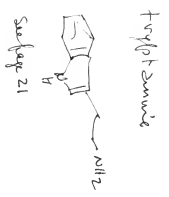
(C)



(D)



parabenz  
N-Mu THPC  
See page 35.



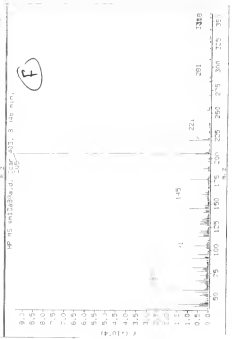
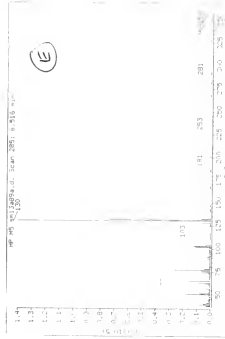
25

skatole ?

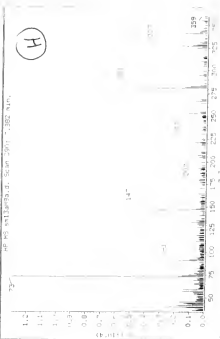
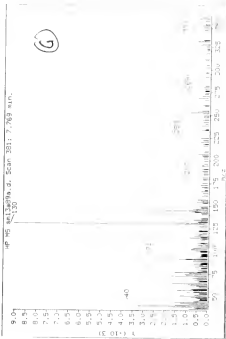


MW 131

?



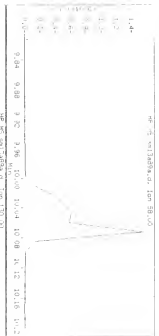
dimethyl- $\beta$ -thiobutanoic  
ethyl- $\beta$ -thiobutanoic  
MW 145



?

2c

Contains for complexity in peak A, page 23

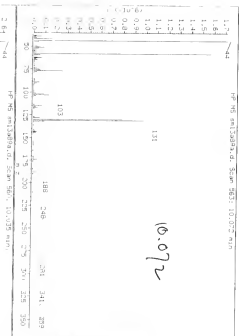


ion 58  
 $CH_2 = N^+ - CH_3$   
 INT

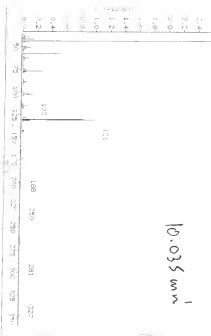


ion 130  


ion 131  

10.035 w<sup>+</sup>

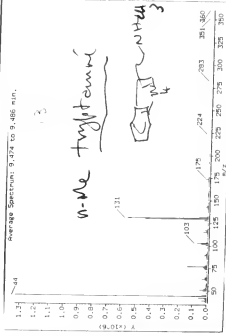
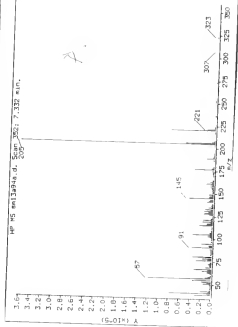
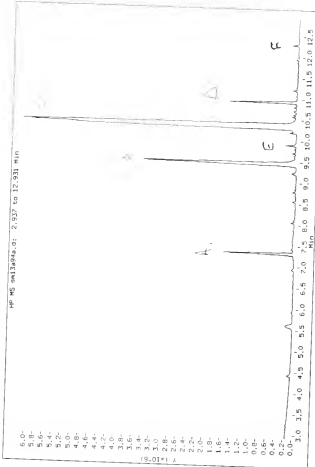


at SF.

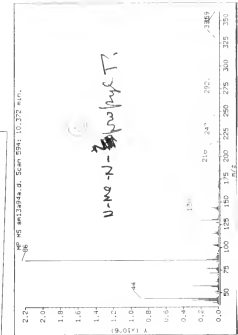
add neat hydrolysis vial to a few milligrams of  
 MMT - heat 100 °C for 1 hr → GCMS that says 4/5 hydrolyzed,  
 some trace Pr<sub>2</sub> (=  $\overset{\text{Me}}{\text{N}}\text{-Pr}$  + cracking) of (b) (c)

27

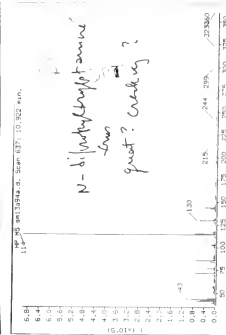
Data file: c:\msd1\7215172151\01\data\_4  
 Injection Date: 05/24/97 07:11



n-Ne tryptamine



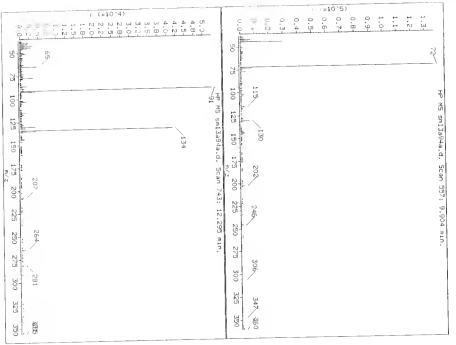
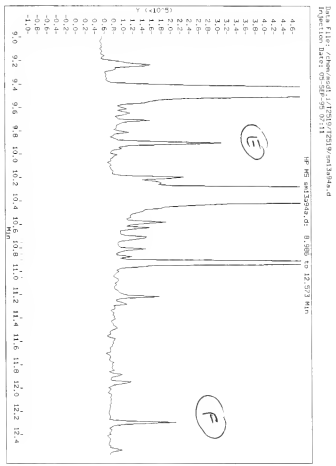
n-Ne-N-~~propyl~~propylamine



N-dihydrotryptamine

quat? cracking?

28



29

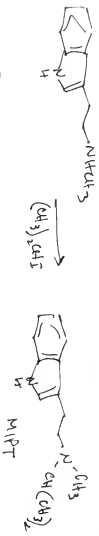


(  
20

7

32

Attempt:



0.3 g free base NMT (1.2 g batch; page 21)

disolve in 4.5 g EtH - seem to disolve RT, when goes in first - ∴ maybe good as reagent. solvent.

1.2 g verpungeliodide (4x 300) 50 10 lbs. TLC 95/5 ether/acetone → good done (with separate)

50/50 ether, 50% hexane.

1.5 g more iodide - 50 15 more lbs. done (largely). no split at origin (no great?)

Shift to reagent - substance between 50 and ether and 25 wt 5% reagent - all goes in - color mostly to ether - separate - shift base is a little white part - water is 5% so some be wet (the soln - 5mm, not all, color goes. nice water is 5% water. Peak → 0.36 g (wash oil for cleaning).

Don't get RT like ~~buttle~~ buttle behavior 150-160° → a white oil - will not crystallize

0.19 g very viscous.

100 blank  
 135 - long look many files at first  
 130  
 150 run.  
 0.05 samples  
 DR sample. 0.36 g excellent base 97% MPT 5% solvent NMT

33

11.1.2

10/10/10

10/10/10

10/10/10

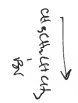
10/10/10

10/10/10

10/10/10

34

Account:



300 mg ~~MSBT~~ into

4.5 g IPA - dissolve + swirling & swirling - add

2.0 g sec. Butylamine -

replant glass w/ 1/2 conc TE  
 reflux 18 hrs w/ = essentially done.

Strip → extract oil - dissolve in 50 ml ether - add  
 25 ml 5% NaOH. Shake vigorously. Flush

→ 0.29 g crude

(Crude P) recycle

110° w/ IR. #150-160° / 0.05 mm.

all bands in IR clear.

0.27 g 65% stuff.

4.5 g IPA

0.63 g sec Bu Ar  
 2.4 g KI - into SB.

0.2-0.9 PM,  
 off IR (19 hrs w/ more)

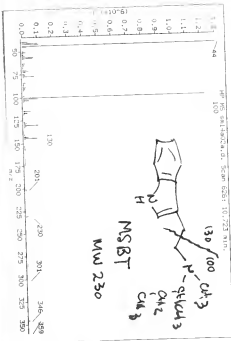
~~strip~~ Very light colored liquid - IR kid not accurate.

Thin w/ IR (MSBT) for  
 comp B 79% pure.

strip - saw band between circles & all bars

Self work - H<sub>2</sub>O - strip circles → 0.37 g pale oil - too fluid.

IR. 170-180° / 0.05 mm Hg.  
 0.12 g white oil, added water





36) repeat of SE run.



0.3 g N-methylmethylamine, into

4.5 g IPA - acid

1.3 g 1-iodopropane.

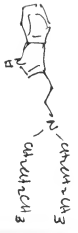
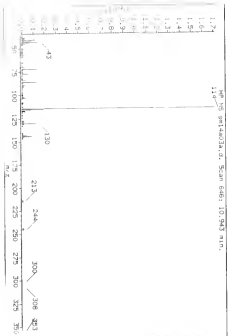
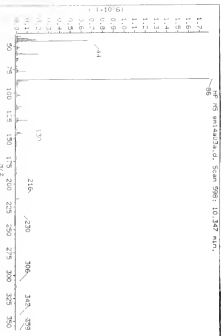
- refluxing 1 1/2 hrs.

strip - GC/MS. more slow than

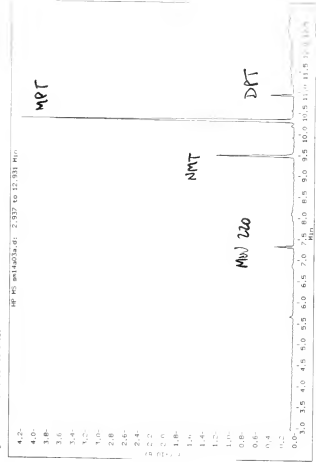
+ 4.5 ml IPA + 1.3 g 1-iodopropane - GC/MS. wait here  
5.5 ml g strip - IR - 1600-1700 / 0.05 → 0.19 g off-axis or 0.19 g

not

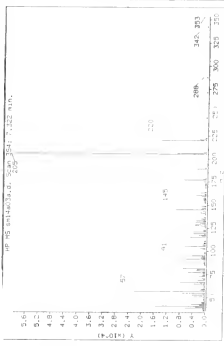
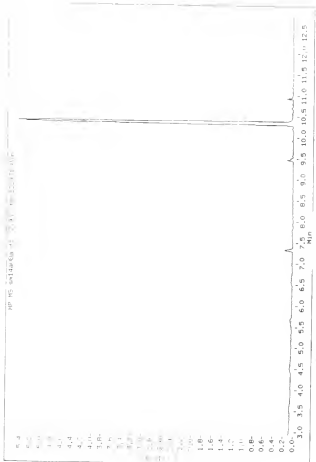
1.25 g  
0.05 ml  
199 °  
100-110 °  
0.05  
0.19 g



Date File: c:\chemdata\c014\m01403a.d  
 Injection Date: 11/09/99 10:50



Date File: c:\chemdata\c014\m01403a.d  
 Injection Date: 11/09/99 10:50



MW-220

NMT

37

1/2 hr  
 assay

12 hr  
 assay

37

1/2 hr  
 assay

12 hr  
 assay

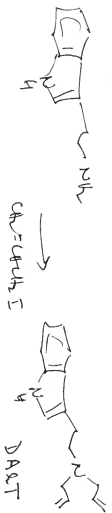
37

MW-220

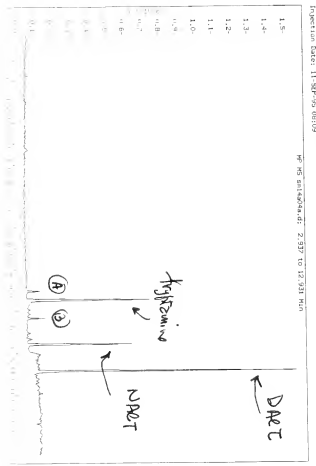
NMT



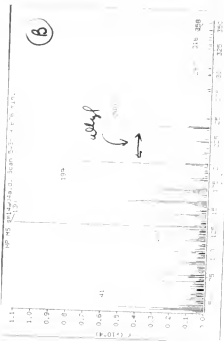
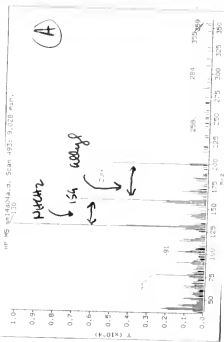
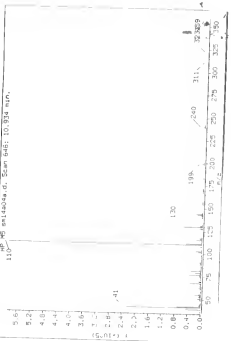
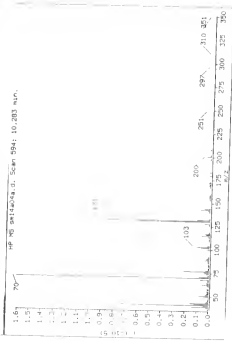
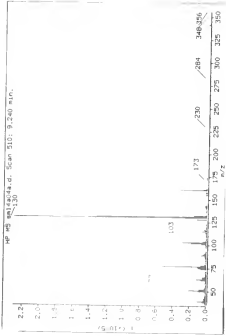
38) attempt.



1.0 g tryptamine in  
 8g IPA. add.  
 4g allyl iodide - into reflux for 40 minutes.  
 + 5mg  $\text{CuCl}_2$   
 + 8g IPA + 4g allyl iodide - on  $\text{4-30}^\circ\text{C}$   
 work up next Am - strip  $\rightarrow$  no yield !!!



39

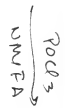






(42)

1 mg



11 Pkt 580

7.2 g POCl<sub>3</sub>  
6.7 g DMAP

Let stand RT start 1700 on clock.

by 1735 just about everything - into SB.

2.5 g imidazole - vial - gets very hot - waste - boiling.

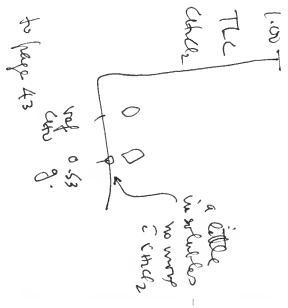
Let go ~ 5 minutes - into 150 ml HD - stir 10.

Am - decant from beads ml - + 10, maybe 15 ml

Meat - scrape, scratch → Strides.

Sifts - wash 2 small amounts Meat.

↳ 0.53 g Residual solid.



Reheat, but cold.

Page (42)

7.2 g POC<sub>3</sub>

6.7 g NMeFA.

~~2.5~~ 2.5 g Indole

still very exothermic, even at 0°C.

stir until all at 0°C, no more rx.

heavy blebe oil - add ~150 ml H<sub>2</sub>O -

stir → water wash out - red color into water.

decant, + water 2x

stir until blebe oil + almost ~~blebe~~ no red into water.

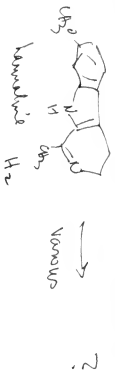
decant. Then + 10 ml MeFA - Strain oil.

(43)

Δ SB ~ 10 min → decant. ▽ to ice bath.  
stir.  
add

H4

trial:



A bunch of trial changes of norbornene, different endotherms, to challenge its end, the stability. The low hydrogen, then norbornene

- (A) H<sub>2</sub> in water, low depth the **high**.
  - (B) H<sub>2</sub> in water, low depth 25% react. **high**.
  - (C) H<sub>2</sub> in water,  $\bar{\epsilon}$  Utrawin C. **high**
  - (D) H<sub>2</sub> in water,  $\bar{\epsilon}$  Utrawin C. **high**
  - (E) H<sub>2</sub> in H<sub>2</sub>O,  $\bar{\epsilon}$  strip of gas from very **high**.
- add - submerged the by oceans
- (F) into acidified (H<sub>2</sub>O) water, + Adams catalyst, + ag. particles soln - hot - sparkless - make basic, xnt  $\bar{\epsilon}$  90/10  $\bar{\epsilon}$  Adams React. can s. extending tetrahedral lowing H<sub>2</sub>O.

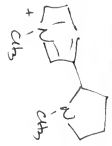
(45)

1724  
N  
Bridgman

1725  
N  
Bridgman



(46)



24.5g inactive (just over 15 mins)  
 use ~~24.5~~ 30 mg HI (to pH ~ 2)  
 at 50% strength.

into 200 ml kanthard  
 add 75 ml 50% HI (try to get pH ~ 2)  
 failed on the yellow strands have been  
 water - wet first - 2 mg. was it in  
 stringy stuff.  
 Play: other, 2 floors - no x-tubs - play - dilute  
 in water

dilute to 1 L.  $\in$  several steps - other to seed,  
 ready to fill. other  $\rightarrow$  x-tubs - strand on water -  
 a bit of a  
 thin,  $\in$   
 strong

fill for, water = 50% other/strand,  
 $\rightarrow$  light yellow strands  
 air dry 24h  $\rightarrow$  38.50g

Mix + other again  $\rightarrow$  2:1 other/strand ratio -  
 more light yellow strands  
 filter, wash  $\in$  2:1, air dry 24h

$\rightarrow$  14.0 g

Mix of Mix -  
 turning very dark  
 OUT  $\rightarrow$  52.50 80% yield  
 53%<sub>20</sub>

Nicotine from (46)

52.50 g di HI salt of nicotine - completely dry. dissolve in  
150 mL MeOH (not sol at all in (Pt.!) add  
20.34 g nicotine (convert to mono. HI) add  
34 g  $\text{CH}_3\text{I}$  (10% xs) - into SB vial at 4:45 PM.  
slight darkening in evening - let go ON -  
off soon. play a bit, scratch, etc.,  $\rightarrow$  seed.

Pour all into a beaker while still very hot - seed  
takes - all sets of xtoline solid. stand ON. break up  
and filter, wash  $\approx$  4 some portions of MeOH -  
Air-dry to constant wt. 98.24 g pale yellow  
sheds

98.24

MW's

nicotine	162
N.HI	290
N.(HI) <sub>2</sub>	418
$\text{C}_6\text{H}_5\text{I}$	142
<hr/>	
$\text{C}_6\text{H}_5\text{I}$	304
N. $\text{C}_6\text{H}_5\text{I}$	432
HI	

72.84 g  $\rightarrow$   
N.HI

(47)



(49)

1872

1872

1872



(5)

1876

52) attempt:



4-20  
4-23N



4-24-3

hydrofluoride

16.0g ~~hydrofluoride~~ (MW 160) 0.1M - add.

25 g 4-23N (MW 101) 0.25M - >100% xs. , add  
squaric acid

11.2g 4-20 (10% xs). very inf. - gets  
stony good

stir until exotherm done - into 500 ml

4N - 2 phases on 48. stir down to a thick, gummy  
sludge - show a few drops - add ~50 ml chexes - very stony  
to go into solution - then clean solution - add more chexes -  
add 100 ml 40, 40 → pH acids.

Separate - exhaust ag. = 2x50 ml water chexes?  
wash chexes phase 1x ~1/4 sat water - very slow pH follow -  
separate - stir → 12.57 g dark viscous oil - to KR3

The sludge from 0.5 - 0.4 Δ 0.2 → 0.1 m. dinkil.

185-200° / 0.11 mm/Hg. about 9/10 clear - residue  
looks increasingly sticky. Oil in vacuum can. looks  
good.

→ 9.45 very vis. oil - amber.

Flame out → 9.37. exhaust play → white clots  
green  
no in food  
stick.

mp 70-73  
mp 71-74

1330 hours.  
22:07  
9.37

mp 70-73  
mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

mp 71-74

(53)

1/10

1/10

1/10

1/10

1/10

1/10

1/10

1/10

1/10

1/10

1/10

1/10



5A

Attempt:



more ethyl groups on amine

What do I call it?

NET is N-ethyl-N-ethyl

NET is N-ethyl-N-ethyl

N-ethyl-N-ethyl

11 May 22

A solution of the hard glass acetamide  
The 30 ml dry THF, water, Ac. Heating, pale yellow  
solution (water 2.02, used 2.315 - levible glass).

A solution of LAH in THF (1M), 60 ml - brought up to  
damp's reflux, under Ar, stirring, 3 ml RB solve.

Add amide dropwise - good stirring. Hold at  
reflux 10 pm.

Stir 20. Sample at ~10 min - (let cool, let stir) here

Heat 20 min - add 50 mg of THF - stirring (total 20 mg)  
filter through paper - wash 2 x 25 ml of THF.

ML base to wet 1st paper - strip → 1.60 g pale  
cream-colored oil.

KBr, 125-135°/0.1 mm Hg → white rice crust  
x halides in the vacuum.

1.58 g  
poured for film - x halo - drop y

1.60 g  
strip

0.1 mm

90° w

125° 0.1 mm  
white,

little more by  
40° w.

Free base white solid

(Acetone benzene 2:1)

as white, w/ 80-81°

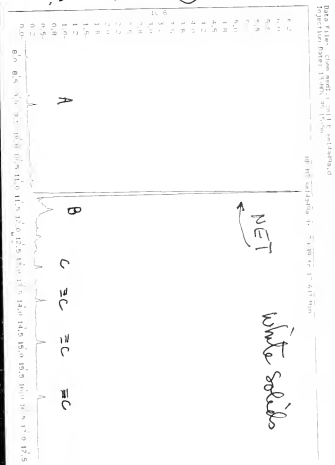
150 mg into 750 mg IPA

to sample the

9 analysis often →

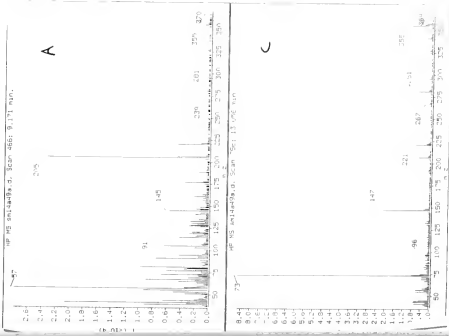
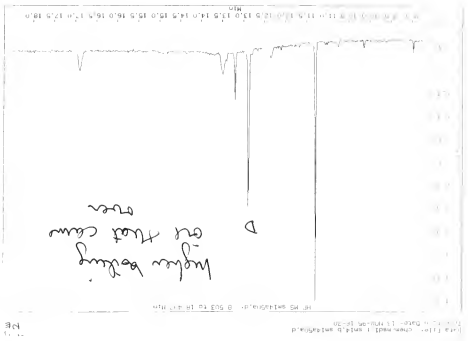
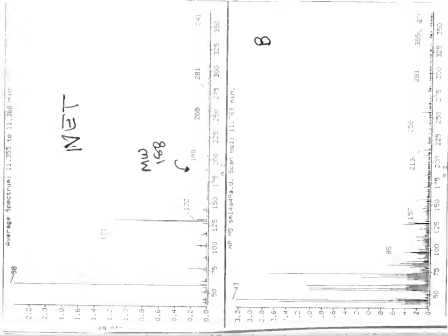
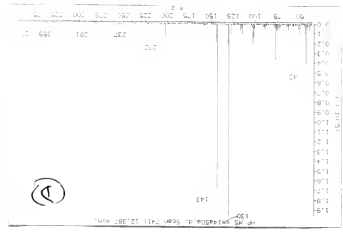
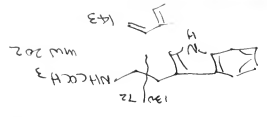
9 closely - X-ray known!

10 days (81-132°  
w/)



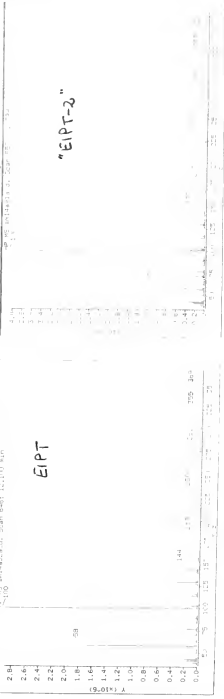
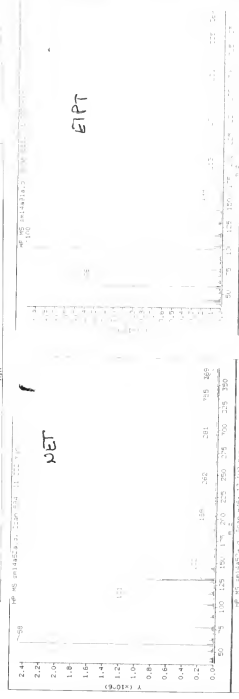
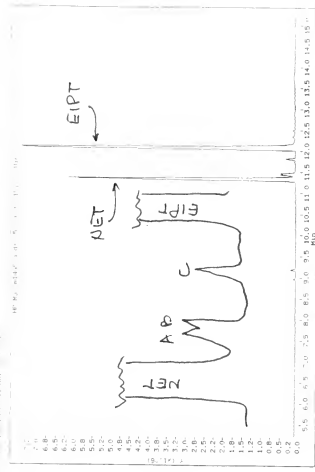
04/76

55





SUKAGIA ST



- (A) 7z 130(131) 202z  $\int \text{CH}_2\text{N}(\text{C}_2)$
- (B) 7z 130(131) 202  $\int \text{CH}_2\text{N}(\text{C}_3)$
- (C) 44 86 130 small 210  $\int \text{CH}_2\text{N}(\text{C}_4)$

(normal) displacement of alkyls is I'?

Udaly  
(100%)

Sf

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100

58

Attempt:



See Br Br



CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>  
CH<sub>3</sub>

DSBT (vapor & recoverable)

1.0 g hydrochloride, into  
 8 g IPA - warm, no, hard to give solution. slow, not low;  
 5.5 g 2-bromo butane - mix vialy SB - 18 hrs total  
 into 150 ml pro, add 150 ml water → give color &  
 clarity - rt 2x 50 ml water - wash-water: 1:10  
 flash → 1.72 g clear residue - to IR.

Starts over 120°/0.05 mm - very faint, warm, then condense.

end at 150° → 1.17 g white oil.

Residual  $\beta$  at least 1.16 g

in a waste on so - two g of 0.5 or less of white crystals

small amt,  $\pm$  IPA,  $\pm$  the same,  $\pm$  skin → fine white

fine white

mp 101°; x-rays

SA 108;

21.7m (lit)

25.5m (lit)

All into 6 ml IPA - clear soln. add cover the (21.7m (lit))

Add ether to turb (16 ml) skin → crystals. stand (1 hr -

filter, wash & ether) → 1.27 damp, 1.07 dry white solids.



(60)



3.2 g Nipfermine (20mm) into  
25g IPA add

6.8 g isopropyl iodide (20mm) - into viallet 500 PM.

off - Seife → dark oil. IR E stem off. darker red due to  
bleg e seed → no

into Na<sub>2</sub>CO<sub>3</sub> + 150 mm (pH → acid to base). Mt 2 x 50 mm

chisel - separate. ~~separate~~ → separate again - dry ↓ -  
slow, even prod. not a good extraction - little chisel - splat  
film → dark oil. - base now due to water.

2.19 g acetic oil. NIT outside developed  
drip fall at 14R. 0.05 mm polyethylene. 0.2 mm bearing.

30° 0.04 - 0.1150. want white oil now at 130° - distill 130-150°

51g total weight 1.51g - chavin not 2 trials → 1.2g - x total  
dispense in 6 ml IPA

1m-150°  
1m-150°  
1m-150°  
H<sub>2</sub>O → blue red ~~red~~ 30-34 drops.  
H<sub>2</sub>O from 1/2 mm tubing. add  
monomers 4 wt IPA - 8 to 10 wt

4 + 20 wt ether → 20 wt water -

filter - warm 2 ether (→ can't, warm). can dry.

↳ 1.55g. H<sub>2</sub>O w.p. 224-227°

Small amount  
 wt: 10-11g  
 m.p. 94-95°

(6)

11/18  
2  
4

(13)

11/18  
2  
4

11/18  
2  
4

(15)



62



11 p. 10

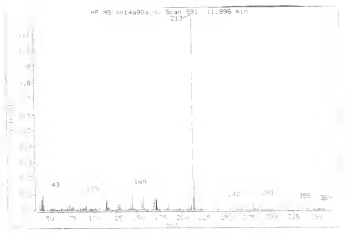
Run 3.2 g tryptamine (20min) into  
 #1 25 g IPA - add  
 6.18 g (40min) n-Pt. I. - add reflux - off 45 hrs.

distillate in CH2Cl2, add HD, add 5% NaOH  $\rightarrow$   
 immediate white cloudy - separate, washes as CH2Cl2  
 combine, wash  $\pm$  the 1 strip. Then wash on  
 all flats, funnels etc, and get the CH2Cl2, but active  
 becomes emptying. What?  
 $\rightarrow$  3.56g v. dark oil (A)

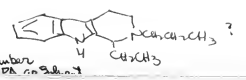
into 25 g IPA  
 #2 6.18 g n-Pt. I. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #3 25 g IPA. into "slow CH2Cl2 + 100 washes, 5% NaOH  $\rightarrow$  white  
 #4 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #5 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #6 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #7 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #8 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #9 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #10 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil

into 25 g IPA  
 #11 6.18 g n-Pt. I. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #12 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #13 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #14 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #15 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #16 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #17 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #18 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #19 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #20 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil

into 25 g IPA  
 #21 6.18 g n-Pt. I. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #22 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #23 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #24 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #25 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #26 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #27 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #28 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #29 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil  
 #30 25 g IPA. reflux 90 min 1/4 damp - off - strip  $\rightarrow$  dark oil



242 mW  
 29  
 215



remember IPA on 2nd run



64



3.2 g impolymine (2 min) into  
 25 wt% IPA - add  
 8.72 g EtBr. onto gentle reflux & stirring 3PM Friday.

reagent:  $\text{K}_2\text{CO}_3$

2.0 g impolymine

5.6 g EtBr

15 g NaOH

8.5 g aash,  $\text{K}_2\text{CO}_3$

skin RT 2:45

5 hrs - off skin, fold through

pulver -  $\text{SiO}_2$   $\rightarrow$  3 wt%

310

boronize in  $\text{ATX25}$  and check  
 diameter inside cavity

rub - convert to black skin &  
 fold through paper, clean  $\rightarrow$  0.55 black rtd -  $\text{C}_{10}\text{H}_{10}$   
 dark  
 oil.

0.16 g mm

0.005 mm

100-110°

KR. 0.005 mm 150-190°

↳ 1.15 g white oil (A)

dispense in 5 wire  $\text{Ac}_2\text{O}$  -  $\Delta$  briefly on SB. (vacuum scale 37°  
 (CCMS (B) 4 mm

acid-base (into 0.5 wt%  $\text{H}_2\text{SO}_4$  - wash 2x  $\text{CH}_2\text{Cl}_2$ )

(bore  $\bar{c}$  6  $\mu$   $\text{H}_2\text{SO}_4$ ) - xH<sub>2</sub>O - skin  $\rightarrow$  0.31 g  
 count

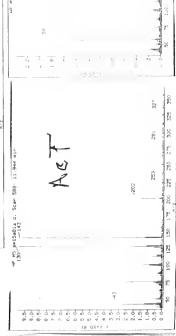
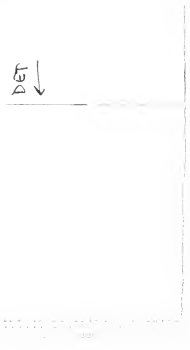
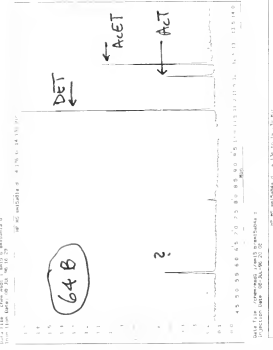
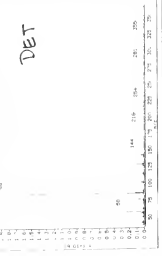
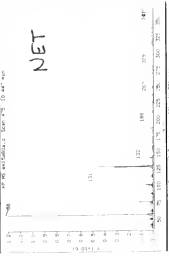
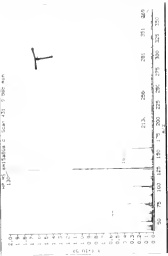
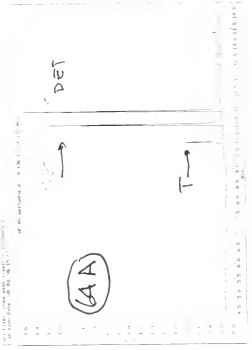
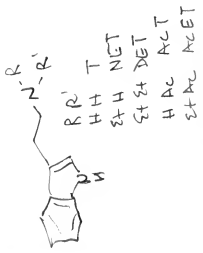
KR. 155-160° / 0.005 mm  $\rightarrow$  7.19 g oil  $\rightarrow$  white xH<sub>2</sub>O

$\Delta$   $\rightarrow$  white solids 0.175 g removed

1:64

130° AM  
 0.05  
 145-160°  
 0.05  
 19

65



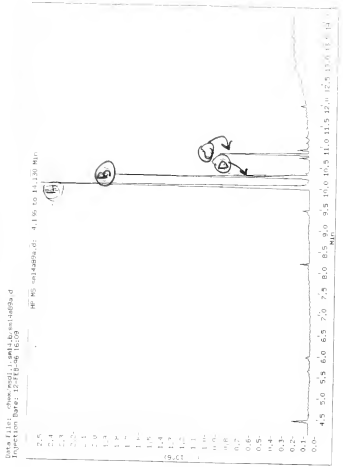
Casine  
 quat  
 100%

100%  
 100%

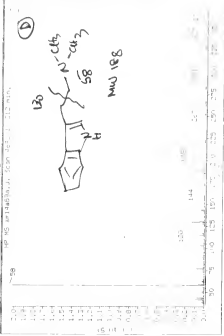
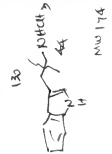
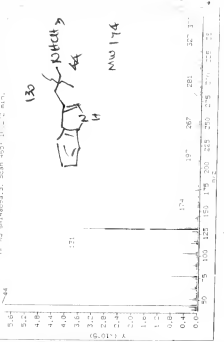
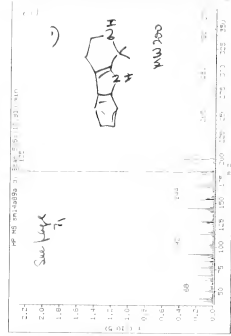
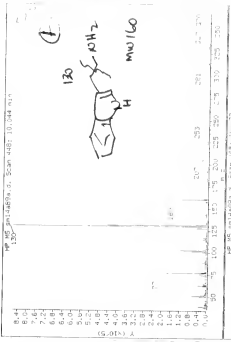
100%  
 100%



69



2.75  
 2.54  
 2.22  
 2.11  
 1.99  
 1.84  
 1.74  
 1.64  
 1.54  
 1.44  
 1.34  
 1.24  
 1.14  
 1.04  
 0.94  
 0.84  
 0.74  
 0.64  
 0.54  
 0.44  
 0.34  
 0.24  
 0.14  
 0.04  
 0.00





Solids from Hydroxamine + MeI

4.42 g wet. This has been twice extracted c 10x.  
water insoluble

into 2 x 20 ml boiling IPA - separate

Soluble

0.26 g gray solid,  
when dry

0.24 g, gray solids.

Insoluble.

Filter - Such -  
3.55g in sol.  
when dry

2.40 g white  
solids.

IR - spectra  
identical

NH 3400  $\text{cm}^{-1}$  sharp.

fingerprints  
767 large  
919 }  
953 } medium  
978 }

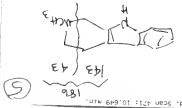
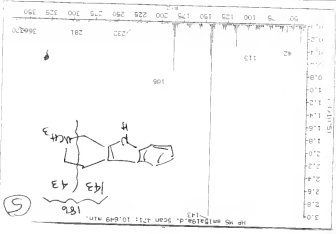
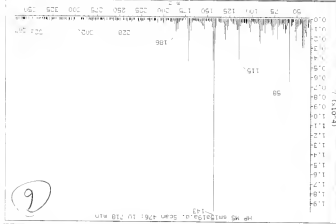
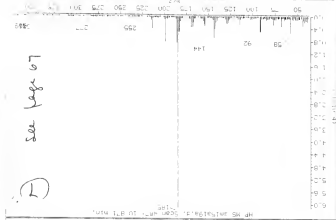
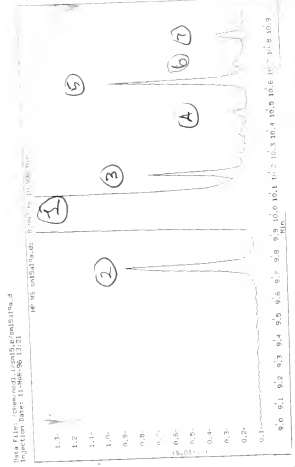
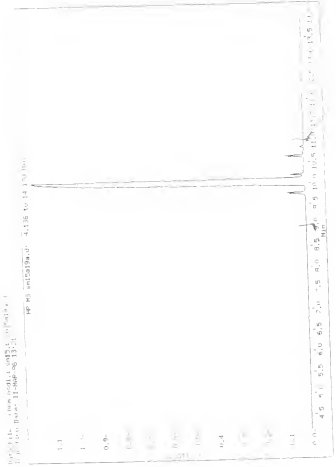
in file

Small amount of each - fused to rock  $\rightarrow$  bleached tan  
c light amine smell. ~~Filter~~ - Combine - dry  $\phi$ SiO<sub>2</sub>





(4)



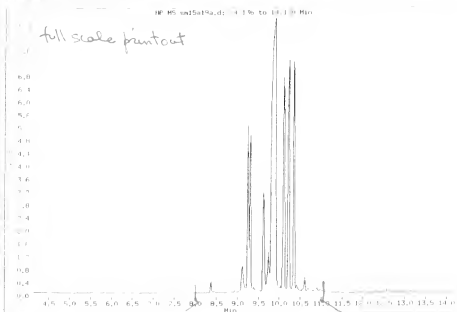
See page 07

(7)

(9)

(5)

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Injection 5.0000 11-NOV-96 14:55

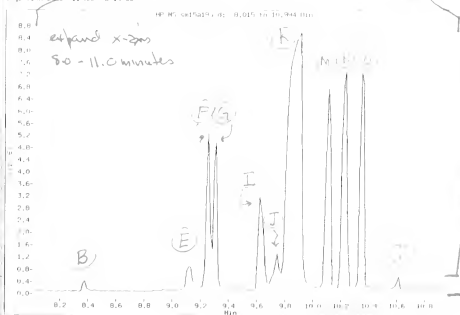


~~Pachycerous~~  
Pachycerous Kingla

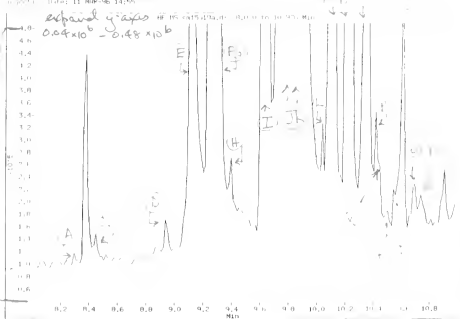
(1)

Total extract 1 part P.b. aqueous  
1 part 6N NaOH  
1 part 90/10 toluene  
butanol

Data File: ChemData1 (1) (1920) (1) (1920) and 14746.a  
Injection 5.0000 11-NOV-96 14:55



Data File: ChemData1 (1) (1920) (1) (1920) and 14746.a  
Injection 5.0000 11-NOV-96 14:55



Compounds reported in P. p.

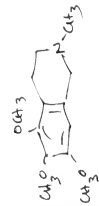
(73)



NW 193 Heliammonite

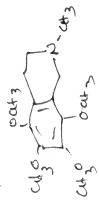


NW 193 Lendireocarine



NW 237 Tehaunine

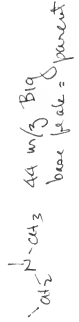
Puccin reports N-oxide NW 253



NW 267 Weberine

all by Metz, R. & McLaughlin, J.H. Planta Medica (1980) 38 180-182

note - from M-8501



44 w/g Big base peak = parent



44 w/g Small weak 30

MW TALLY (Components)

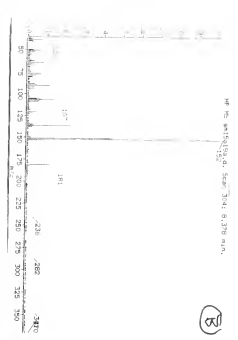
152 (156)	192	206-7	236	266
8	9	FFI (K)	MN	0
	(+44)			

page 74 } page 75 } page 76 } page 77 }

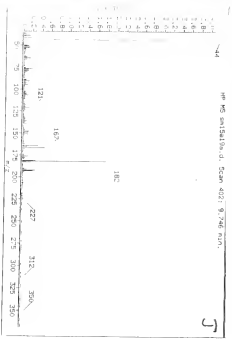
74

lower ml compounds

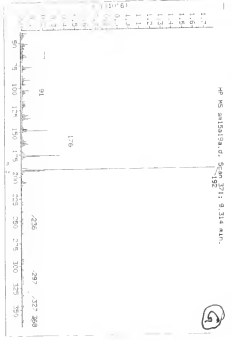
(B)



J

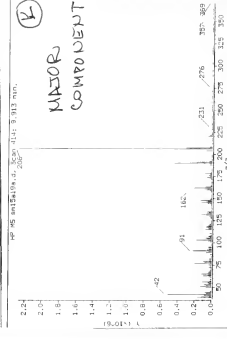
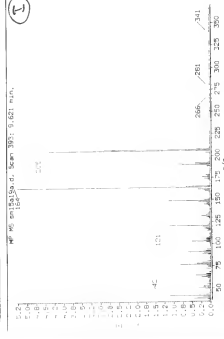
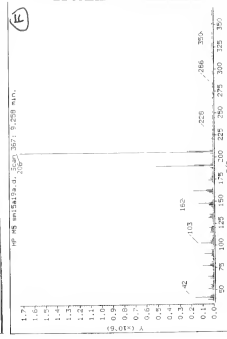
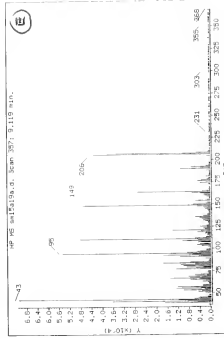


(A)



206.7 MW components

(75)



possibilities



12 isomers

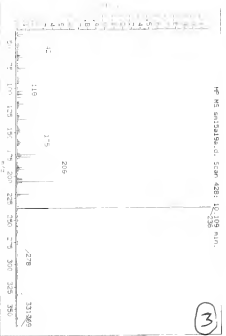


none of these have been reported in P. p.

MAJOR COMPONENT

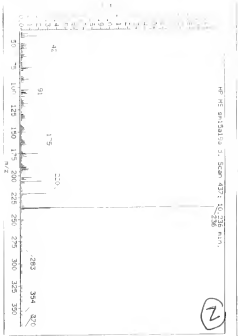
76

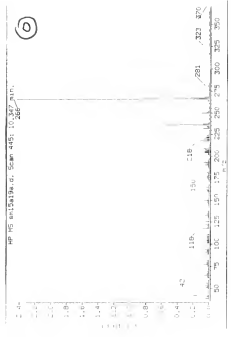
MS Spectrum of 1,2,3,4-tetrahydro-1,2-dimethyl-1H-indole



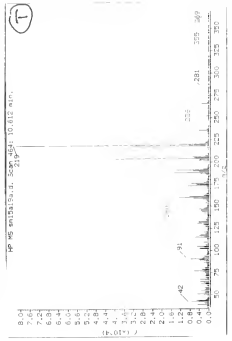
3

2





MW 266



MW 219  
236  
266?



MW 267

Wetmore is a  
valerianol component  
of P.p.



96



10g Tryptamine - into  
20ml ether - stir a long while

→ 14 ml ether  
some adsorbance T

3.6g T. add.

11.0g CH<sub>2</sub>I - stir 3 days → graining solid

precip. ether

Snake-out gunk - 15.154g.

water 10 ml  
wash 10 ml

black oil - big white  
wash 10 ml water - no solution  
wash 3 x 10 ml CH<sub>2</sub>Cl<sub>2</sub> - clear, yep

ether  
n < 1g  
oil  
shift  
out

white solids

wash 10 ml

white solids 3.40g

hydroxide  
under

orange organic.

fish → 2.63g

also 11 gunk  
from water phase -  
from washing

gust HI = 330 mm.

the white solids  
when dry 3.05 g  
dissolve in 35 ml hot water  
care → x-ray

Salt water wash a bit extra

Snake skin - dry = 3.07g

0.3 g  
0.3 g

wash with  $\text{P}^{\text{O}^2\text{F}}$  2.77g

tan gunk  
gunk

comp

black gunk  
let dry.

6.4g solids  
H<sub>2</sub>O.

ground under 2x5 ml  
CH<sub>2</sub>Cl<sub>2</sub>

-Ch3OH.

(79)

2.50g quat - dry - into 25ml MeOH - Δ SB → clear solution  
to RT - gaseous white crystallization (save a bit on plates)

Δ again - soln. add  
1.0g AgNO<sub>3</sub> (10% xs) - looks like white solid (quat, not AgI)  
Δ again on SB ~ 10 min.

Fisher - wash in boiling MeOH (another 20 ml). to  
slightly opalescent yellow brown solution, add  
1.0g PSH. opalescence disappears. strip on RE. → 2.12g  
vacuum oil. Δ in flame - much red flux (PSH?)  
but a lot of bubbling. 5 min. Δ - fine color.

wash in ether into beaker - + 50 ml ether - xht  
2 x 25 ml di ether → ag. in nice yellow color - add  
5% NaOH to pH blue - slowly!! - xht in 3 x 20 ml  
ether - cool - flask → amber oil 1.04g.  
Slight PSH smell. to KR.

Over at 145° (up) 1.5 min. - first colored,  
then white! Smoothing funny in vacuum.

1.04g  
115°  
2 min  
no  
165°  
16 min  
over  
170°

5g thymptamine → into 25g MeOH Δ see ∇ RT

5g 37% chro pH very green.

2.5g NaCO<sub>3</sub> - stibery - pale green pH → sh. green  
quite exsiccative; ∇ = running thro - add

50/50 same thro/MeOH until pH ≈ yellow. If does not  
run - still yellow! strip RE → reddish thin  
oily sludge

7.6 g theptamine (60mm) into 100 ml MeOH add

34g CH<sub>2</sub>I<sub>2</sub> (4 eq) (used 42g) - add

40ml 25% MeOH - onto 3B - ve (fume) → solids, off at 4 hrs, ▽

filter → 52.55 g slotted wet xtals.

dissolve in 100ml boiling MeOH, ▽ twice → pale cream-colour xtals

filter, wash sparingly w/ cold MeOH - air dry

→ 12.75g around xtals

12.75g into 100 ml MeOH

+ 5.0 g Ag<sub>2</sub>O (3.2 I have, 2.0 form) →

5 hr - stand a few days - filter - wash w/ MeOH

→ 2.4g MeOH  
amber fluid -

+ 5g φSH

Flesh → 8.97 viscous amber oil. Am trying to dissolve into ether to get into a KR flesh, bubbles, exothermic, and eventually 2 phases in ether! NOT slottaneous φSats ↑ but surely acid & base smthg! ? into small flask. flesh on R.F.

Total residue (2 phase, still?) to KR. 0.15 mm - over at 130° → white oil. push to 170° - terrible residue lot

140  
over.

2.18g watery fluid  
φSH smell  
→ 5.33g amber  
glass  
out



83  
PT



with NaHCO<sub>3</sub> (A) with N<sub>2</sub> (B)

116 g T (10 mm)  
into 20 ml warm IPA A, ▽ clean sulfur  
5.1 g n-propyl I (3 fold)  
+ 3.4 g NaHCO<sub>3</sub> (4 fold)  
Stir RT 36 hrs.

add 100 ml H<sub>2</sub>O → pH blue  
& bleach oil  
xrt 3 x 50 ml CH<sub>2</sub> - clean  
Substrate very clean - flash  
dichlor → 3.18 g fluid brown  
oil - white smell. (83A I) (some impurities)

Save a bit for master spec - TLC further  
add 5 g Ac<sub>2</sub>O. ▽ SB 10 min  
into 0.15 N H<sub>2</sub>SO<sub>4</sub>. (~150 ml)  
ag. ↓  
xrt 3 x 40 ml CH<sub>2</sub> → xrt 15  
Keep ag. (Some black gunk in bottom)  
+ ag. + 150 ml H<sub>2</sub>O. Salt OUT  
ag. with 3 x 40 ml CH<sub>2</sub> + comb. flash OUT

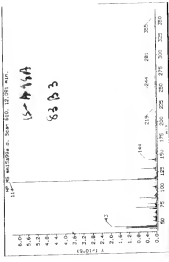
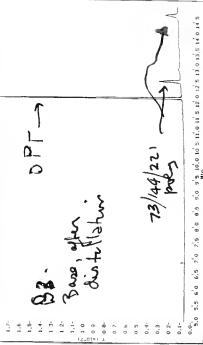
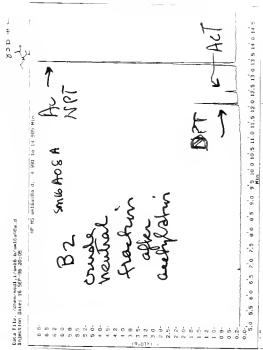
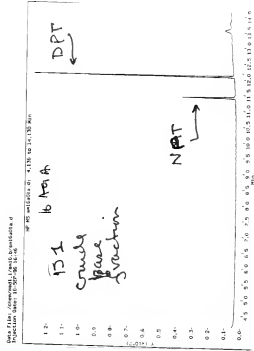
CH<sub>2</sub> + comb. flash OUT  
+ combine  
+ N<sub>2</sub> H (60) to pH blue  
xrt 3 x 25 ml CH<sub>2</sub> - flash → 0.41 g  
KR 1 ~ 150-160 / 0.15 mm  
0.37 white oil (83A-3)  
to the salt 5 x IPA, H<sub>2</sub>O, other  
mp 172-174 0.206

add 4 g Ac<sub>2</sub>O.  
A SB ~ 20 min - into CH<sub>2</sub>  
+ 150 ml 5.2 N H<sub>2</sub>SO<sub>4</sub>  
xrt ag. 3 x CH<sub>2</sub> - pool  
CH<sub>2</sub> is 15 N H<sub>2</sub>SO<sub>4</sub>  
- Swells y Ac<sub>2</sub>O wash 2 x pool  
into CH<sub>2</sub> - add  
N<sub>2</sub> H. stir -  
Sep. flash again  
→ combine aside  
fraction

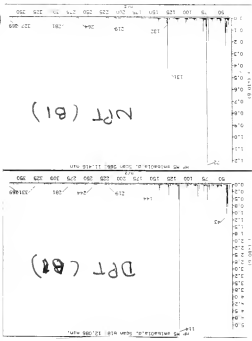
4.51 g granular white solid  
into CH<sub>2</sub> + 100 ml H<sub>2</sub>O  
ventil - 0.4 g N<sub>2</sub> H<sub>2</sub>SO<sub>4</sub>  
xrt → powder CH<sub>2</sub>  
flash → 2.21 g brown  
fluid oil.  
83 B I  
83 B 2



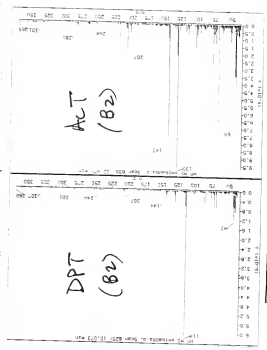
DPT (NPT)  
P 183 B



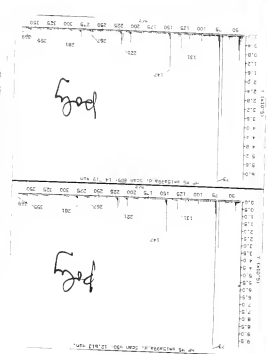
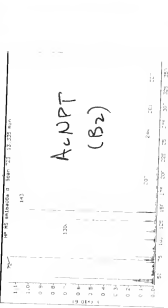
183



NPT (B1)



ACT (B2)



Good





tryptamine  $\xrightarrow{\text{PrI}}$  ?

1.6g Tryptamine base, into 20 mL hot IPA - RT, add  
 5.5 mL diisopropylamine add  
 3.0 mL (5.1g) iPrI. (30 min). stir RT.

add 1.5 mL more base Friday evening.  
 strip on Sunday evening  $\rightarrow$  3.2g medium brown oil  
 add 5 mL Ac2O. 5 min stir.  $\Delta$  to RT.  
 add 3-5 mL more NITRO.  $\rightarrow$  hot - let come to RT.  
 AM - still acid !!? - add goods NITRO - let stand 4x  
 next day - partition 0.5 N to 50% (no, stronger!).  
 wt. 3x 40 chex2

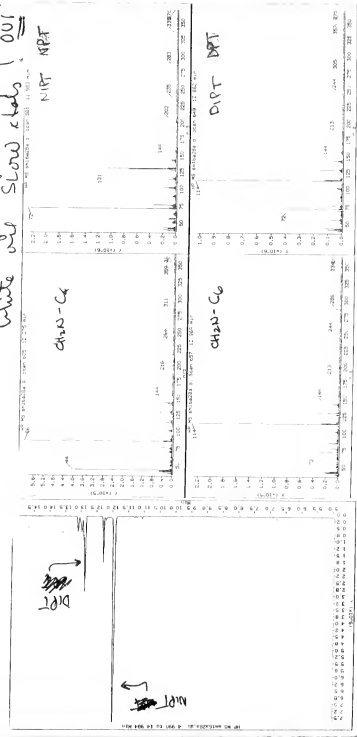
Xtract powdered chex2  $\in$  H2SO4 vs 3x chex2  
 40 mL

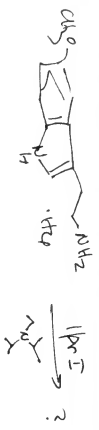
strip  
 0.20

$\swarrow$  ag. prod.  
 base  $\in$  ~~acid~~ NITRO  
 25%

xht 3x 40 mL chex2 - flash  $\rightarrow$  0.20 g ygh.

Kf: 170-185 $^{\circ}$ /0.05  $\rightarrow$  a couple of droplets of white oil slow chex2! OUT





10g amine .H<sub>2</sub>O MW 226.5 = 44.2mm  
 10 wa IPA

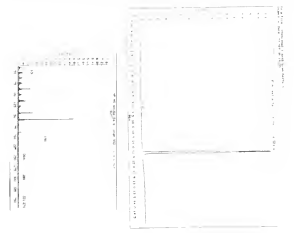
4.0ml amine MW 124 x 5 = "645" MW  
 x 4.42mm = 286g  $\Delta$  = 742 = 88ml  $\Delta$  to add  
 Et<sup>+</sup> odds

1.33ml iodide  
 IPrI MW 170 x 3 = "510" MW  
 x 4.42mm = 226g  $\Delta$  = 170 = 135ml

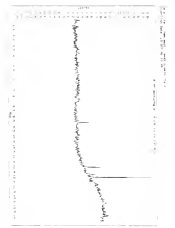
site for ~ 36 km<sup>2</sup> RT stream to dump out a white ppt.  
 filter solids 0.77g dry, beautiful - only ML<sub>2</sub> to

Ad 0.77  
 NL good  
 Swudge

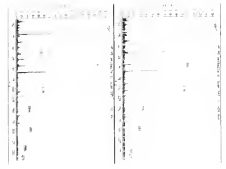
MW 5.460T  
 w/p 160 + 30 = 190  
 w/d 160 + 72 = 232  
 w/d 160 + 114 = 274



white solids (MW 101.5)



Swudge



Repeat  
p87

tryptamine  $\text{H}^+$  ?

1.6g T base into 20 mL IPA on sub. add  
7.5 mL  $\text{H}^+$  add

3.0 mL  $\text{H}^+$  on sub 7 PM Tuesday e-flux. off noon Friday

strip quite hard  $\rightarrow$  ~ 7.8g dark mass - ①

don't believe the weight difference in ~ 40 mL

ch<sub>2</sub> - wash 2x 100, spot a bit of ch<sub>2</sub> -

strip - add 200 mL ~~water~~ add 5 mL

H<sub>2</sub>O -  $\Delta$  56°/5 min. to RT - Add 5 mL H<sub>2</sub>O

(not base after warming at 55° while -

add group more ammonia - base ~~...~~)

0.5 N H<sub>2</sub>SO<sub>4</sub> to 200 mL, acid.

extract 3 x 40 mL ch<sub>2</sub>  $\rightarrow$

ag. wash 2 x 40 mL ch<sub>2</sub>  $\rightarrow$

base  $\bar{c}$  6 N NaOH  $\rightarrow$  cloudy.

strip 3 x 40 mL ch<sub>2</sub> - flesh - into

50 mL 14/20  $\bar{c}$  ch<sub>2</sub> - flesh  $\rightarrow$  0.96g brown

oil.

~~...~~ To K.R.:

0.6 mm 160°-175°

0.85 g off-white oil  
pretty much  
x-tals

0.6 mm

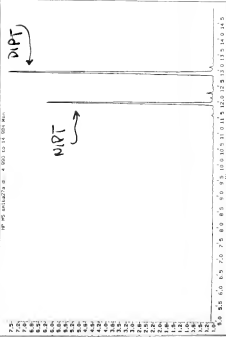
(40° wa

100° x/2

175° done

100° x/2

175° done



what add 3x 40  
ch<sub>2</sub> - to extract energy for  
into ch<sub>2</sub> - then strip  $\bar{c}$   
do H<sub>2</sub>SO<sub>4</sub> BUMP - leave 20%

digest = 0.91g 1?

grind up under 10 mL RT hexane  
filter

0.31 white  
x-tals

89 D

+ pale yellow ML  
- let evaporate

89 E

TO Alice

89

90

push 65  
again



IPTE  
?

0.77g white shade f. p 88 - into tho (SRT) + 600 water  $\rightarrow$  pH  $>$  8  
 xht  $\hat{c}$  3 x 40 we chills flash, add add p. 88  
 soundly?

0.83g fine lat. temp. (Snow Kites farming)  
 try again, without ce

10 wt IRK  
 4 we  $\sim$  1/2

1.33 wt iPRTE use 1.5 we into SB 36 hrs  
 strip to residue (save list for TLE, MS) (A)

+ 5 we Arzo - gets quite hot, did not put in SB. 10 min -  
 5 we Core Water. Not Nephthurens, + lots of water -  
 water that - to home - xht 4 x 50 we chills pond  
 Not forked other shacks

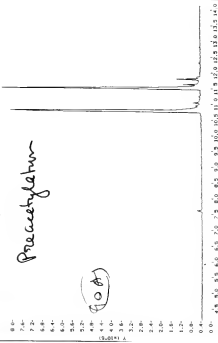
1  $\hat{c}$  200 we. 5/10 these work  $\hat{c}$  2 x 40 w  
 chills  
 1 again  $\hat{c}$  200 we. 5/10 these work  $\hat{c}$  2 x 40

pond, home  $\hat{c}$  6  $\hat{c}$  100 (Sunday. we) xht  $\hat{c}$  3 x 50 w  
 chills  
 2nd  $\rightarrow$  80g cracks  
 KR 175-100 0.10 mm  $\rightarrow$  0.52 g off-white and (B)

17.10.16  
 11.11.16  
 11.11.16

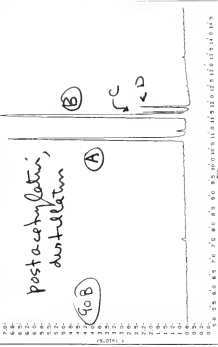
DATE: 11/10/87 TIME: 10:00 AM

NAME: [unclear]

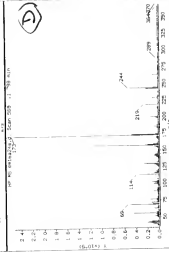
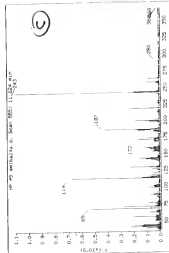
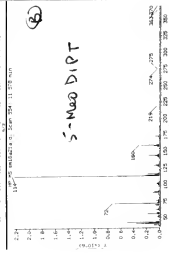
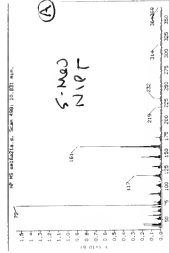


DATE: 11/10/87 TIME: 10:00 AM

NAME: [unclear]



911



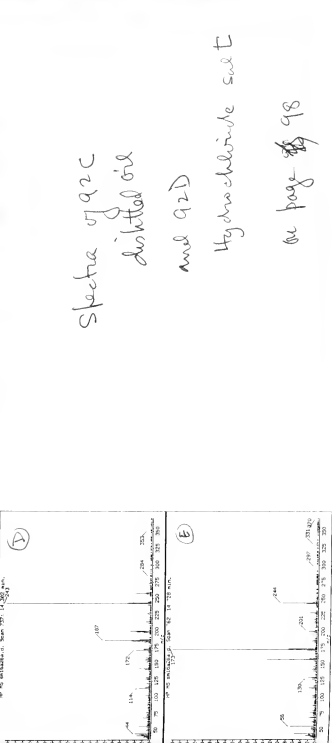
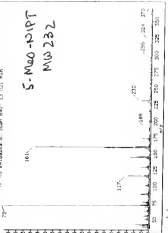
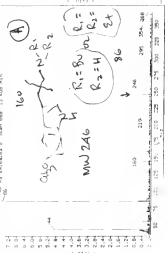
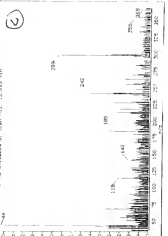
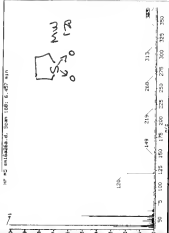
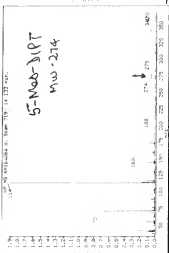
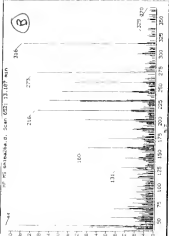
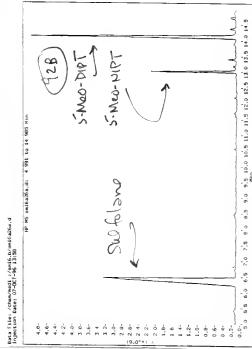
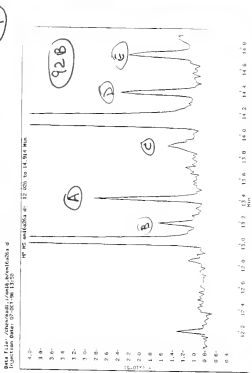
15/11

1.06  
1.74  
2.02  
2.26  
2.46

10.5



93



Spectra of 92C  
distilled oil  
and 92D  
Hydrochloride salt  
on page 98

94

95

96

97

98

99

100

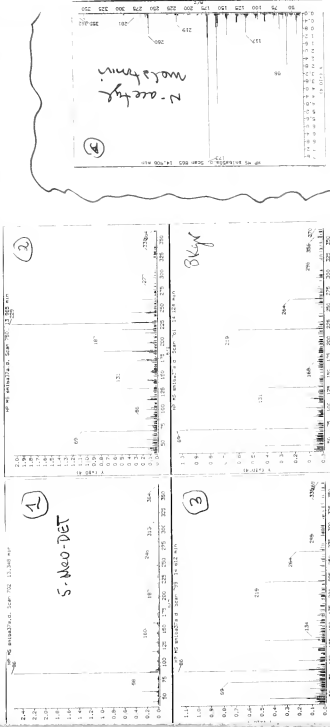
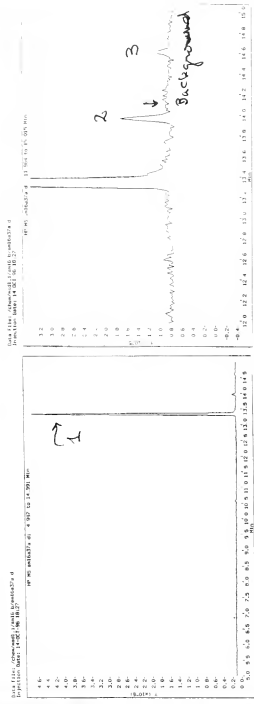
101

102

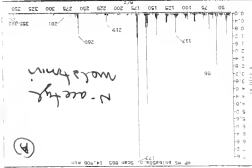
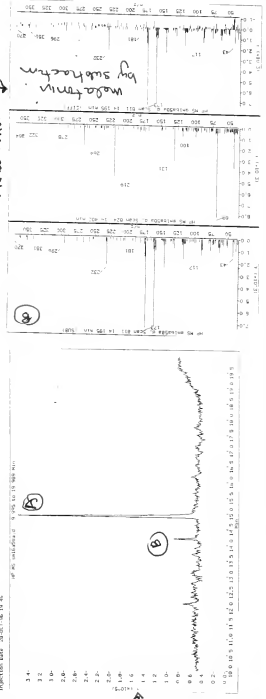




(5)

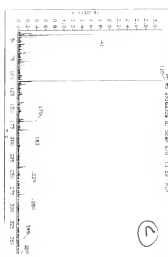
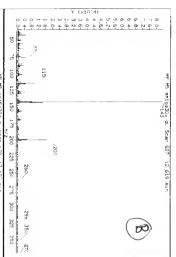
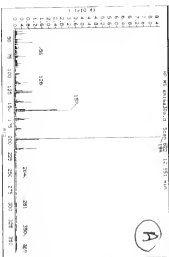
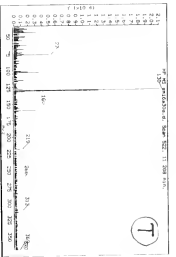
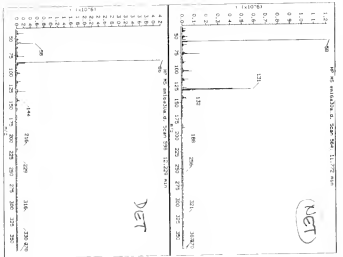
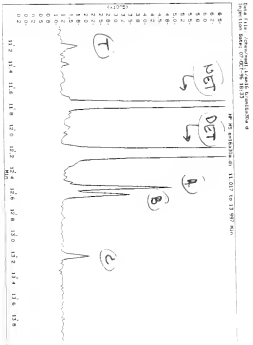
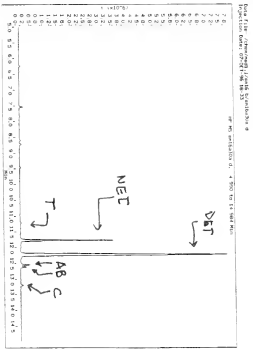


← 5-epi-melatonin  
 → melatonin



96

page 86 - proacetophatin

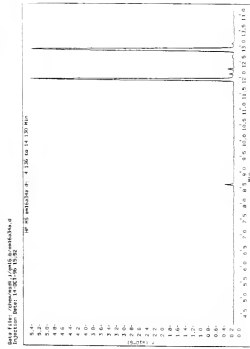
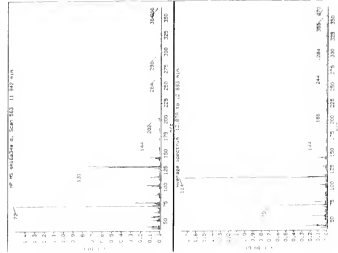






Work-up of page 89 - distilled, Acidified DIPT

(99)



page 89  
 LR's out  
 → xtabs  
 10.9g

ground  
 powder  
 10 wt Hexane  
 ↓  
 0.34g white  
 xtabs  
 (89D)

decent  
 ↓  
 Mts  
 pale yellow  
 solid  
 evap → xtabs  
 0.13g  
 (89E)

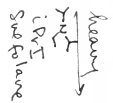
Small amt of <sup>4th</sup> (H) possibly!  
 Small amt of neat-100 sol.  
 pent-100  
 neat / hexane

wash  
 sin out  
 hexane  
 0.02g  
 (89F)

ML to  
 remaining  
 ml (a little  
 less)  
 0.12g  
 (89G)

ML  
 ↓  
 ML

APD



10 ml - was for ring conditions

1.6g triethylamine  
10g washed sulfonane (with all dimers)

6.5g diisobutyl (ethylamine)

8.5g polybutyl ether  
into SB. 2 phases - shake occasionally

12 hrs later - reaction still 2 phases - off into 100 ml -  
pH in blue, xH<sub>2</sub> 3x 30 ml hexane, (and water = 100 ml  
water - crash on R.E.)

Knowledge of fluid for  
GCMS (stable in vials)  
good spectrum - integrate  
by peak weight = 95.6%  
small amt NIPRT -  
trace sulfonane, water not

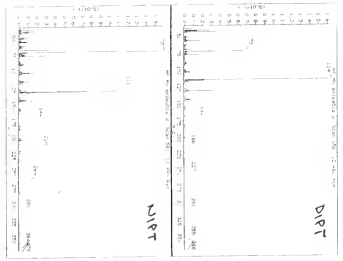
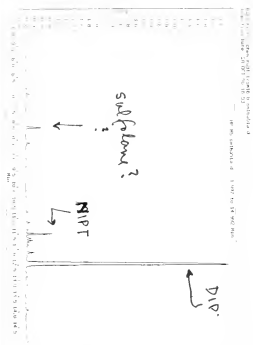
1.37g  
mp 72-74°

Washing with hexane  
what's stable MS compare  
MS - free of sulf., NIPRT only!

2.67g deep brown in fluid oil  
OH → stable under ill -  
recount  
1.034  
contaminant dmy.  
1.37g 100-70°

mp 72-74°  
0.05m  
off white  
69-71°

1.37g material - 0.50g into 2.5ml hot IPA, 9 drops H<sub>2</sub>O, 25ml  
ether → 0.50g bottle w/ 192.5-193°



1.37g  
mp 72-74°  
0.05m  
off white  
69-71°



0.44 g awfully good crude N-St. indolemin  
 from page 94

grind under a few ml MeOH → almost white solids - Ref/OL  
 a few milligrams from MeOH → white x tabs.

340mg.

same for mp  
 sent for IR  
 trace for TLC

0.30 g amide - 6 ml 5% NaOH - into SB - 2 hrs - still  
 white solids - H<sub>2</sub>O evaporating - + 3 ml 25% NaOH -  
 10 min hr. Still solids - 10 change - add ~ 2 ml  
 IPA, lower - 2 hrs - going into sol. a couple of grams  
 remain, keep at 100°.

overnight - into 100 ml H<sub>2</sub>O (is basic  
 of course) x tab 3 x 40 ml CH<sub>2</sub>Cl<sub>2</sub> - prod.  
 x tab CH<sub>2</sub>Cl<sub>2</sub> 3 x 50 ml 1 N H<sub>2</sub>SO<sub>4</sub>

← 0.2g  
 CH<sub>2</sub>Cl<sub>2</sub>

flask

→ 0.24 g residue

film goes to solids  
 amide?

when dry 0.19 g  
 almost white solids

IR carbonyl:  
 1608 cm<sup>-1</sup>

Fingerprint

780, 829, 928, 1131, 1068

1108, 1152, 1199.

wash 2 CH<sub>2</sub>Cl<sub>2</sub>

OH<sup>-</sup> 25% NaOH

x tab 3 x 50 ml CH<sub>2</sub>Cl<sub>2</sub>

flask

→ 0.05 g







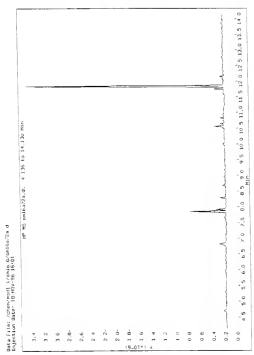
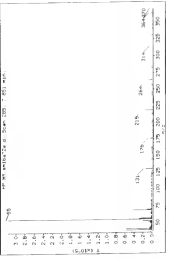
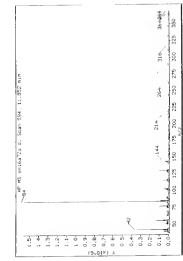
1.44 g I  
 2.6 g sulf  
 RT.

1.6g Tryptamine base with 2g sulfolane Δ about to dissolve  
 3.6g I  
 2.6g sulf

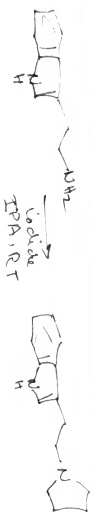
Stir RT - 15 hr work up - there is a 2-phase character to it all - into 100 ml H<sub>2</sub>O (to basic) and x/4 = 30 ml hexane. (There are globs not soluble in either phase). Strip hexane → 0.25 g sticky white

into citric for distil - strip down hard  
 ↳ 0.08 g - distil → trace product

36.3 = 100% 64  
 2.6 7% 130  
 1.1 3% 214



104



1.6g Naphthamine base into 20 mL IPA & T  
 3.6g Ethylacetate, E  
 2.2g  $\text{NH}_4^+$  stir RT a few days. solvent evaporated  
 stand a couple of weeks.

104A

(1) 2g. Ethylacetate  
 from fraction

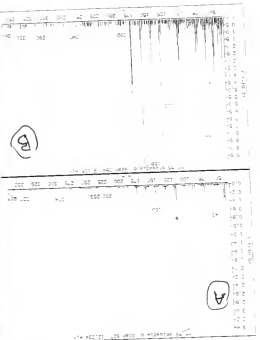
hexane  $\rightarrow$  2.1g

(2) 2g. Ethylacetate  
 from fraction  
 $\rightarrow$  1.47g. ground under  
 Rock. mill  
 under 1/8" mesh  
 130 mesh

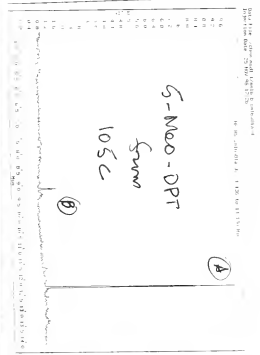
I forgot I used starter from

3.2g T 30 mL IPA - not all in  
 9.5g water  
 9.9g ICA I stir RT  
 8 PM

104B

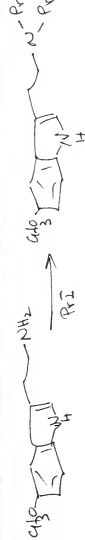


104



5-Meo-DPT  
 sum  
 10.5c

(105)



1.02 g 5-MeO-T into ~50 ml H<sub>2</sub>O, 5% base → below pH-  
 xHCl + 3x25 ml CHCl<sub>3</sub> → flask → sudden xHCl 0.87g  
 0.85 g 5-MeO-T base in 10 ml IPA. Δ to dissolve - acid;  
 2.8 ml ~ 10% (3:1)

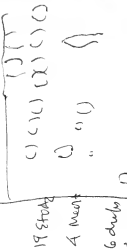
1.5 ml Cl<sub>2</sub>CHCH<sub>2</sub> I very dark.  
 emb SB for 4 1/2 hrs. (500 rpm)  
 in T, worse & dis- no yeast  
 Strip on Rotary → sponge -  
 fair amt. Inocul 5 ml.

add ~ 50 ml CHCl<sub>3</sub> - abs solvent  
 clear red - brown solution;  
 add 1 g Ac<sub>2</sub>O - no Rx. brk in  
 SB for 5 minutes - str.  
 add 2 ml ag. H<sub>2</sub>O - no Rx. str.  
 add 60 ml 1.0 N H<sub>2</sub>SO<sub>4</sub> - stamp acid -  
 60 ml + some + 50 ml H<sub>2</sub>O x hrs

CHCl<sub>3</sub> red brown  
 17% GCMS - 1 peak - 5 min. DBP  
 no other peaks  
 (105C)

TLC pure dust! (as substrate)  
 Partition between H<sub>2</sub>O & MeOH  
 hexanes 4x25 ml  
 flask → 0.56 g pale yellow oil (D)  
 to small 14/20 = 0.54 g

(all color left in MeOH)  
 KR  
 new vacuum tubing  
 170-160°/0.04.  
 beautiful white oil.  
 0.49 g  
 into 2.5 ml IPA  
 7 too little & too much (damp H<sub>2</sub>O) of just xHCl  
 + nDS w/ ether. Filter - ether - wash air - dry  
 → 0.54 g white. stable IR fine mp. 193-194°



19 str.  
 4 MeOH  
 6 double time  
 10%  
 T MeOH 4x25 ml  
 over  
 A B  
 C D

even less ag.  
 wash to w/ CHCl<sub>3</sub>  
 flask → 0.51  
 still in  
 MeOH

KR 120 ml  
 0103 150 ml  
 10 to 220°  
 pot  
 have in  
 the vent  
 looks  
 funny!  
 (105B)

over  
 no oil!  
 (105A)  
 no peak  
 product!  
 bad  
 GCMS data,  
 product!

56  
 100  
 160 ml  
 0.4  
 170  
 190

106 (attempt):  
 MW NPT 302  
 RT 170  
 RT 238



0.50 g NPT into 5 ml solvent - Δ SB to benzene -  
 add 100 μl to - forget it was the hydrochloride -

benzene -

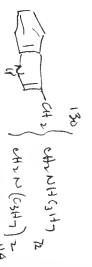
add 0.8 g 6N NaOH - then

1.3 g  $\text{K}_2\text{CO}_3$  (x2)

3.4 g (should be 1.7) but some RTI was  
 RTI got ~~down~~ cleaned up & the  $\text{K}_2\text{CO}_3$

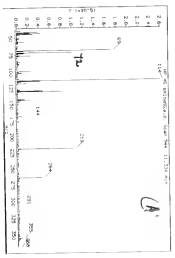
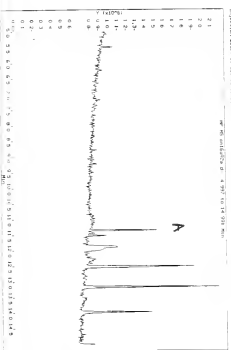
Outo SB worn - off 5:30 PM - ship → bleach vial -  
 add 100 μl to - second run - it should have been  
 absent - add 50 μl NaOH as well - now it is kinda  
 in solution. that 3x 30 we became - clear

Peak at 106A  
 0.14  
 0.10  
 0.10



(MS - it is more  
 sensitivity of MS can find  
 PPT. 106

130° large no weight. OUT -  
 0.05 min  
 150-160 (ghost waste)  
 0.05 min Hg  
 0.01 g - one booklet - saw Smudger  
 for CMS  
 PPT. 106



AQ2A

separate



see 66

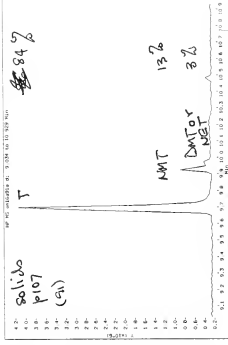
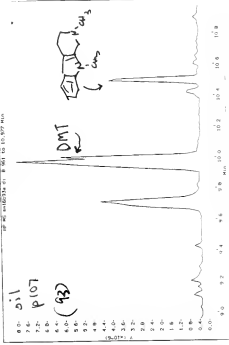
4.34 suspended in ~10 ml ether

SOLIDS which 1.72g  
TLC largely T.

Oil 1.52g  
TLC T, MeT  
other stuff. 1.82

py.  
1.52g  
TLC T, MeT  
other stuff. 1.82  
(average  
density  
x 1.465)

→ dissolve in 10 ml IPA. conc. it to red ppt paper. scratch  
stubs (washed  
with hex)



0.45g.  
white solids  
+ no  
w/ ether  
turbid.

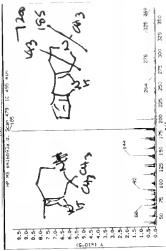
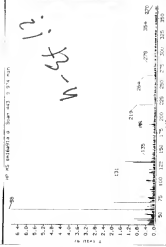
scratch - finally  
xtals.

0.19 g almost  
white solids

lots of ether → oil  
deccant firm

IPA eluting  
decenting  
oil. residue  
(signs of fine  
xtals on  
stirring)

NO  
Peak  
-15%



3



NMT

Solids 1.72  
Oil 0.6  
1.82

TLC T, MeT  
other stuff. 1.82

(average  
density  
x 1.465)

stubs (washed  
with hex)

0.45g.  
white solids  
+ no  
w/ ether  
turbid.

scratch - finally  
xtals.

0.19 g almost  
white solids

lots of ether → oil  
deccant firm

IPA eluting  
decenting  
oil. residue  
(signs of fine  
xtals on  
stirring)

NO  
Peak  
-15%

108

Synthesis materials from p 92



rotated fraction: skip hard on R.E. → 4.60 g liquid  
liquid - to KR,

by 100° (0.05mm) big white fraction

4.00 g (Substance?)  
Sats to glass - yes!

pink by 170° (0.05mm) small, section liquid fraction

~~0.23 g~~  
(Substance?)

0.23 g

ethylene benzene

same data for CENS (108 k)  
wants for reagents.

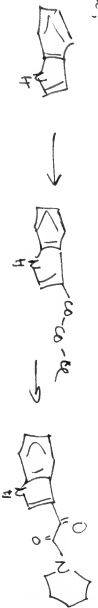
Substance  
no higher numbers  
want MS to react with  
methyl reagent with

not - out





(110) ethylene;



reagents  
PIP-T

1.0 g indene in 15 ml TBME (solvent) - to this, add  
(over 30 min, warm at 0°?)

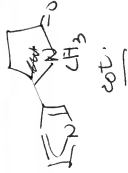
1.1 g oxalyl chloride in 15 ml TBME.

stir another 15 min, filter solids, wash  
briefly with cold TBME. - add, stirring,  
about at a time, to

2.0 ml pyridine neat. Add x5 2.0 ml to  
saturate - filter. air dry.

1.98 g air dry  
mp 175-178°  
reprecipitate from hot neat, filter, wash lightly,  
air dry → 1.01 g white xbrs. mp 182-183°

(111)



Cotinine quat

0.25 g  $\underline{\text{C}}$  PU  $\underline{\text{E}}$  41 add

10 ml CH<sub>2</sub>Cl<sub>2</sub> - add

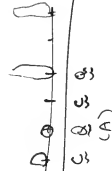
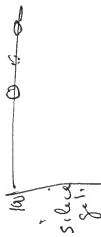
0.50 g CH<sub>3</sub>I - stir & swirl - overnight - Am - flash -  
 heavy v.l., waxy. CH<sub>2</sub>Cl<sub>2</sub> over CH<sub>3</sub>I → slight red ex heat -  
 still dark brown (A)

half to  
vial

half still  
in flask

into 1/2 ml thru  
 xrt 2 x Et<sub>2</sub>O  
 flask eq.

↳ dark  
 only spots.



(A)

Quat.

CH<sub>2</sub>Cl<sub>2</sub> already MeOH  
 1/1

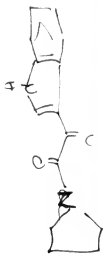
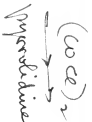
MeOH + 10% NH<sub>4</sub>OH

no different.

Quat. increasingly

border to silica

(112)



A solution of 1.0 g imide base in 15 ml TBME is stirred well. There is added, dropwise, a solution of 1.1 g ceceose (theo (1.17 g) over 20 minutes (solid appears at 1/2 addition point)) stir an addn 10 ~~min~~ min. Filter  $\rightarrow$  yellow xrbds - work " sparingly  $\bar{c}$  RT TBME - add as a solid, a bit more at a time to stir 2-1 g pyridine reat. Reboil,  $\rightarrow$  white solid & color in soln - when RT, add 80 ml  $\bar{c}$  H<sub>2</sub>O. Stir pile filter, wash  $\bar{c}$  H<sub>2</sub>O  $\rightarrow$  1.62 g wet off - white xrbds. yellow

at RT quite amorphous 1.2 g - try 10

Soln for rehd - the insoluble part 1/4 TBME toluene, strike at. CH<sub>3</sub>CO Perfect.

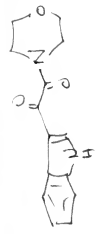
Amplure add in 30 ml boiling CH<sub>3</sub>CO - slow to

Amplure  $\bar{c}$  RT white xrbds - color in ml! (wet)

Slightly off white, fine xrbds, filter, wash lighter  $\bar{c}$  CH<sub>3</sub>CO - an dry white xrbds dry weight

0.87 g  
 w/p 219-220

113



(cocgl) → morpholine



≡ to page 112 down to "new" →

here, added to 2.0 ml morpholine - stirred - exothermic - but stays large white. let cool. then add 80 ml 1N HCl. filter - wash 2 H<sub>2</sub>O - remove, grind w/ the chunks under fresh water filter → white solids. 1.55g wet

let partly ~~over~~ air-dry. 0.95 when completely dry. pale pink.

recrystallize from 4 ml MeOH at the boil - filter - wash extremely sparingly with MeOH - air dry to 0.75  
 to that weight - white 0.75g - crude mp 183-186  
 of MeOH → mp 187-188°

0.75  
of MeOH



MET

Tryptamine, N-ethyl-N-methyl  
Indole, 3-[2-(ethylmethylamino)ethyl]  
N-Ethyl-N-methyltryptamine  
3-[2-(ethylmethylamino)ethyl]indole

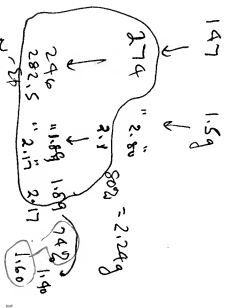
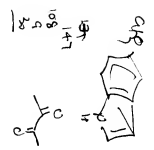
See under MPT

**SYNTHESIS:** Under an inert atmosphere, a solution of 7.0 g N-formyltryptamine (see preparation under the recipe for N-methyltryptamine, NMT) in 50 mL anhydrous THF was added to a well stirred mixture of 7.0 g LAH in 100 mL THF. This was brought to reflux temperature, and held there for 3 h. There was then added 11 mL EtOAc at a drop-wise rate, and the mixture held at reflux for an additional 2 h.

added  
solvent

The reaction mixture was cooled, and diluted with 70 mL Et<sub>2</sub>O. The excess hydride was destroyed by the addition of 7 mL H<sub>2</sub>O, followed by 14 mL of 15% aqueous NaOH, 10 g Na<sub>2</sub>SO<sub>4</sub> and 10 g MgSO<sub>4</sub>. After an additional 15 min stirring, the mixture was filtered, the inorganic solids washed with Et<sub>2</sub>O, and the combined filtrate and washes were stripped of solvent under vacuum. The residual oil was dissolved in a small amount of anhydrous Et<sub>2</sub>O.

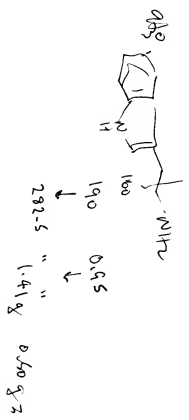
5.4 = 2.14



1.60  
 $\frac{96}{246}$   
 43 g 145  
 $\frac{36.5}{282.5}$   
 2.5 mL 8 mL 282.5  
 (RT)

246 0.45  
 282.5 51.6  
 " "

50g



Fig



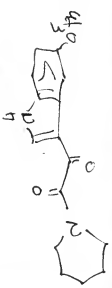
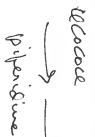
145



146

Fig





100°C

A solution of 125g 5-mevandole in 15 ml TBME

was stirred, and a solution of 1.1g oxalyldiacetide in 15 ml TBME added over 10 min. Female - red crystals start to appear after 10 min, filter. - wash with a little TBME - combine "low" female solids - add a bit at a time to 2 ml piperidine - stirred - exo Norm & fumes of solvent.

End of adding N1 we move piperidine to get everything wet & add female gone. to RT + 80 and 100 the - 100 - stain for quite a while - dig not quite - get wet with No water off by keeping adding - ground under went ~ 2 ml? solids - filter

aground, filter  
 → 0.01g pinkish dust

0.82g vacuum  
 mp 153-158 → 0.65g totally dry  
 next lot, 2g next

15) continued - filter → wet  
 medium colored solids - dry in SF6 → 1.13g

was vacuum powder  
 sample for mp 115.8 and. mp 180-200 (195 and.)

Recrystallizing from 15 ml boiling MeOH (absent)

from a wet bit of (washed side) clear hot - ∇ RT → white  
 filter → white solids, x two - another ML

washable - wash  
 acetone - low dry  
 residue. mp 210-214  
 → air dry. pale cream : 75 wet - MeOH.  
 .65 dry -  
 (reformed)



Cococyl  
→  
pyridolene



Identical to  
→ "base"

deep tomato color  
deeper than 114  
30 min +  
15 min

"base"  
make a mix 2.1g pyridolene & 2.1 ml  $\text{H}_2\text{O}$  - stir well - add deep tomato solids a bit at a time pasty again - add 10 ml water first - then pour 1 ml H<sub>2</sub>O - some solids - much gum. - filter all out (solids, gum) ground under 3 ml MeOH - filter → creamy solids - pour up particulate - no clean crystals. safe for up. into 2  $\text{H}_2\text{O}$  (2 1/2 ml) MeOH - Δ SB → solids - to it → white solids. filter - .36g product.

.59  
meat

.58  
meat

brownish product 0.11g. xtd a bit from MeOH - not good  
base  
gum sets up solid 0.15g  
ground brown to  
olive green solid  
ugh.  
15A.

.48  
not  
noted.

OUT ALL.

Repeat - drop imitations same scale 125 inside (5ml TOME) 1.1g  
oxalyl chloride (15 ml) - dropping over 20 min - 10 min stir - increase  
not dark tomato solids. filter. A brief air dry - add dab by dab to  
2.0 ml neat by inclining. This time washed all the color into the  
the base meat - all finally decolorizes to cream color - and  
it flats up to a softer solid (15 min y rub) - keep home -  
generous - good portions - add 20 ml 1:1 H<sub>2</sub>O → nice  
solids - rub the chunks in a spatula until all fine

(116)



acetone  
for purification



1110259

147  
147  
25  
25  
14  
14  
12  
12  
159

Usual combo:

1.25 g 5:Pho under in 15 ml TBME, stir, add  
1.1 g acetone in 15 ml TBME over 20 min - solids  
at about 5 min. Really over thin pencil, and get a  
normal tomato ketchup - not dark tomato. Stir over 1 hr  
10 min - filter, wash  $\bar{C}$  TBME, an dry (carefully) & add  
back into 2.0 ml neat methylene. Take time making  
if all homogeneous (15 min) until all tomato is  
dispersed in the oil, pencil - becomes dark red/dark  
brown. Then add 85 ml 1N HCl  $\rightarrow$  solids - ground  
up the chunks - no gum! - filter, wash  $\bar{C}$  the -  
onto SB at 100  $\rightarrow$  dry powder - wash red - 1.35 g  
A small amt. - out of neat OK. contly.

ALL 1.35 g aside in 10 ml MeOH  $\Delta$  SB  $\rightarrow$  clean  
blend red solution -  $\nabla \rightarrow$  xkals. wash sparingly  $\bar{C}$   
MeOH  $\rightarrow$  white - blend red ML

$\rightarrow$  get acetone

splish -  
0.85 g - very weak prod.  
things of pink color mp 193-194  $^{\circ}$



S-MeO-pyr-T

25 ml 1 M LAH in THF (cloudy) in a RB (300 ml) under Argon - Δ to reflux & stirring - add dropwise over 20 min.

0.65 g S-MeO-pyr-oxide in 25 ml dry THF (boiled & out under Ar to distillate, mostly staying in. EtOH being 4% each drop. Hold at reflux 45 min. Δ to RT - Kill c

50% aq. THF - reasonably large Pt<sub>2</sub>O<sub>3</sub> - vacuum filter through filter - wash THF. oxide goes to a infusible

7 ml gel.  
50%  
little breaks up under water

ml - to residue on rotary → grossly amt of almost colorless, fluid oil. (presumably OK smell). Then, white solid sludge, which it vigorously decomposed (!?) with the evolution of some gas & the development of a dark to black area. still fluid, but now deeply colored - into CHCl<sub>3</sub>, x 3 x NHP

110 mg.

bind up 100%  
1 drop  
1 drop  
2 ml  
or 4 ml

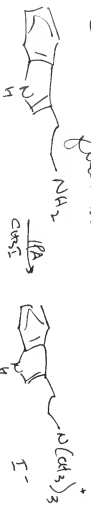
Carica  
with bicarb.  
flask → deep brown oil  
w/ argon, TH - no product?  
distill anyway.

bag.  
OH - cloudy  
x HCl ether  
flesh  
→ 100 mg  
flam.  
almost glass  
GEMS  
ferrule

no NHP

(118)

ethylene IPA as great solvent.



based on recommendations in schedule IPA work page 69.

3 g T - into ~~30~~ 30 ml IPA (Δ to dissolve, stamp in

at RT. add (stirring) 10 g HCl. - no ext. than in. 4 PM  
Thun day. First wash in 10 min. no over. Remove - 1 hr -  
heavy of solids (silver stops - too thick)

mp in IPA. - filter → cream colored solids - 2.72 g  
as in 175° wash in IPA

dry 1.81 g

xtal to 203-4°

wash with IPA 192-4

xtal clear 210-11

into T. lical

Reflux 115 - Surfer

119

96 ← 213  
dry: wash  
cell in P. salt - H<sub>2</sub>O

15 MTBE  
15 MTBE

Reflux 115 - me  
1.25 ml water  
1.05 cecococo (washed, fresh)  
2.0 ml pyridoline

differences, fresh cecococo, from Actos.

addn - 20 min → light tomato - stir 15 min -  
filter - wash (by suspension) in TBME (RT).  
Air dry several min. to rather loose state.

3.42  
wet  
3 ml acetone

1.42  
↓  
97 air dry.

Add (scatter, not bunch-wise) to 2.0 ml  
pyridoline. The few dark spots melted in  
a spatula → dark cream viscous oil. Add  
some 10 H<sub>2</sub>O → off-white solids - grind w/  
chunks, filter, wash & H<sub>2</sub>O → 3.42 g wet solid

grind under 3 ml acetone to almost white paste - it seems  
to form as I work with it. Filter - wash & very small  
amt. acetone. → 1.42 wet. ON air dry 0.97 g

0.94 g  
dry  
100%  
100%

0.94 g into 12 ml boiling ethanol - clear & to RT  
let stand ~ 1 hr. filter, wash sparingly & dried → 0.72 g wet

0.69 g dry  
mp 212-213°

0.528 wet  
p/20



See 109



(121)

2.07g S-NEOT

.15 ml Solflane Δ SB → sol ▽ R<sub>2</sub>

5.5 g  $\rightarrow$  mix

7.2g iPrI (good, used 7.9g, saving)

SB 6 hrs. darkens quite a bit - looks almost in one phase. strip on PE

28.5 thick, dark amber

+ 75 ml hex → 1 phase.

XHT 2 x 40 ml 20-60 pct ether - back wash @ 40 ml (hr)

There is a red oil interface

had into this -

XHT @ 3 x 40 ml,

wash 2 x 50 ml hex

flesh (red oil)

10.8g thin oil

product largely Solflane

(121A)

pct ether sharp head

1.47g pale

amber oil - amine smell.

d. 04 mm

120 blush.

145 over steam heat, FB

160 done

# 1.33 g

6 ml IPA

the 22 no 25 yrs #1

starts at 20 already starting.

add 4 ml ether to air fraction.

wash @ 3/2 IPA/ether

air dry 1.34 g

check IR - 109 # D



(122)

Oxals & Carbs.

5-Med-DMT.

Green base - very hot for human consumption  
→ water (causant) crystals

used was →

0.025g in 1.25 ml IPA 1 drop HCl - either to turbid (20 drops)  
no crystals could be gotten

Maybe second run of 100%? E. Flakes on lab bench in warmth in two,  
underwater. - lots of solids - red - and so wet liquid, flaked,  
strong smell of amine strip → 2.3 g red in oil.

Scrape out solids → 18.1g

3.47g

grind under H<sub>2</sub>O, crystals - self crystals

Second crystals lost  
wash & dry 1200

Flash crystals, → 3.47g

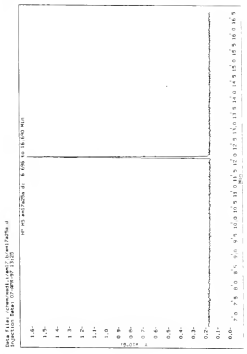
Fluorid &  
+ gum. - work & check  
(not set)

Transfer to 4/20 RB -  
Flash-RB to see what will happen.

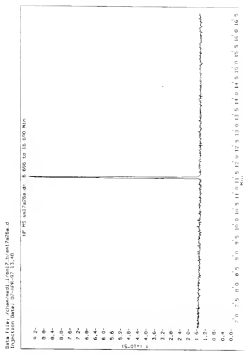
50-70 lots of white. Dry in oven.  
0.04 mm. stop at 400 change flash.

ov - 135° wa

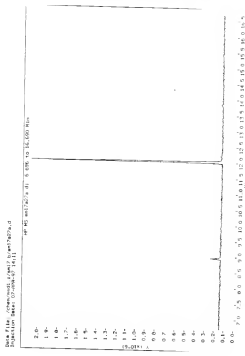
by 170° Some orange  
stuff over -  
not in dec.  
junk!  
OUT



5-MeO-DMT



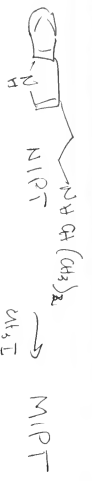
SN-17 H26-A Song base VII-17 NIBT



SN-17 A27A VII-31

weak peak  
NIBT

124

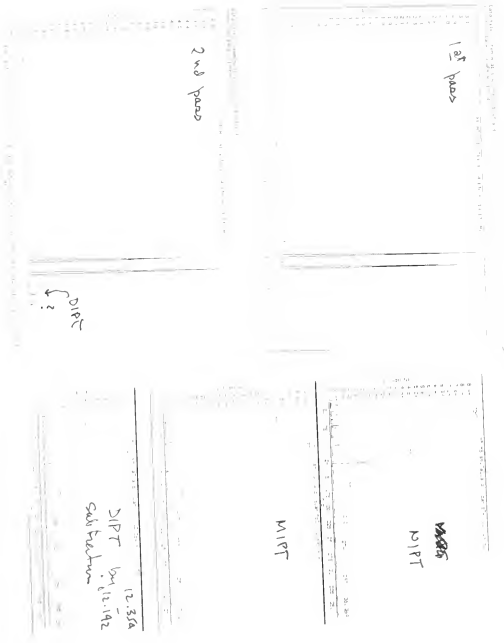


0.41 g (2 mM) N1PT. The  $\omega$  is 50 mL  $\text{hex}$ , 50 mL  $\text{NHT}$  -  
 XHT +  $\text{CH}_2\text{I}_2$  (5 x 40 mL)  $\text{N}$   
 Peak  $\rightarrow$  0.45 g oil. (thio.?)

with 25 ~~more~~ mL IPA. -  $\Delta$  50 3 hrs

+ 0.35 g  $\text{CH}_3\text{I}$  (25% x 5) 0.25 =  $\rightarrow$  dry!  
 TLC same N1PT - 20%  $\text{NHT}$  - with 10 mL IPA - +  
 0.14 g  $\text{CH}_3\text{I}$  (25% x 5  $\rightarrow$  75% x 5) -  $\Delta$  50 - cat strands

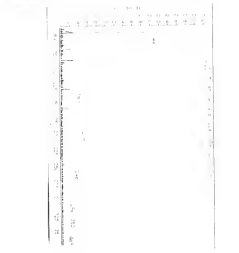
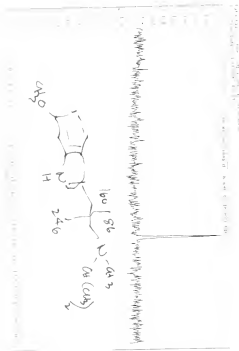
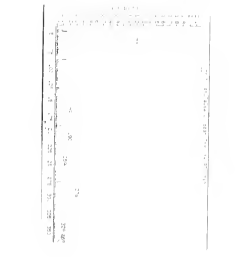
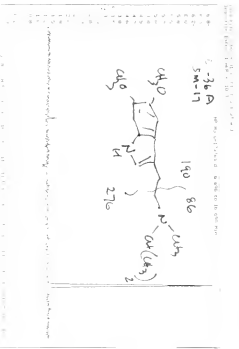
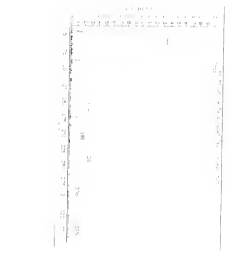
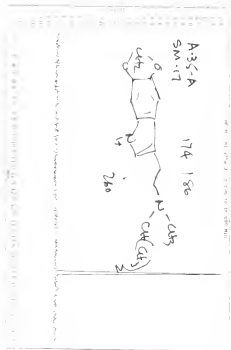
oil, 25 mL IPA 0.35 g  $\text{CH}_3\text{I}$



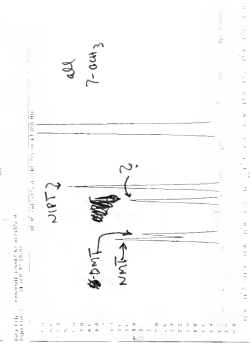
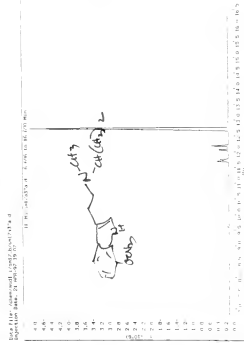


(26)

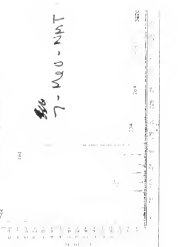
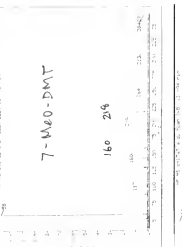
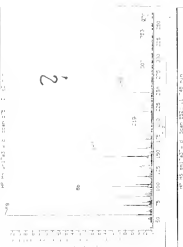
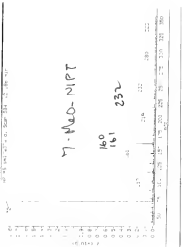
Spectra from here of these



7-NBO-MIPT spectra

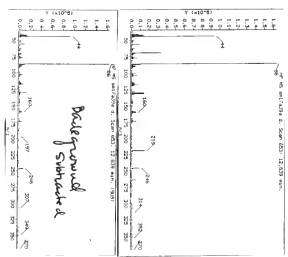
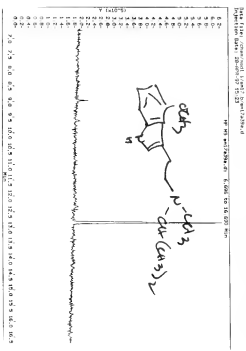
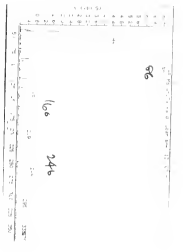
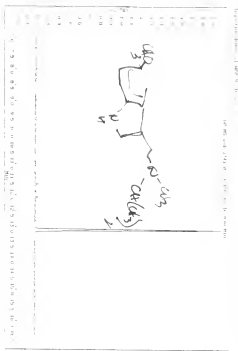


127



126

from four and five.

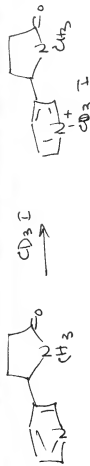


129





131



0.85 g Cofinine - info  
1.5 mL MeOH



To 20 g ag. in H<sub>2</sub>O (sat soln - kalgaram stuff) add  
 20 ml 50% base → 40 ml 25% H<sub>2</sub>O (Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub>O) (+ salt)  
 ▽ in ice bath. add  
 30 ml H<sub>2</sub>O add  
 17.2 g add 37% H<sub>2</sub>O  
 add all of this, to

23.4 g indole ethyls - skin (stick) 40 mm wick -  
 skin 0% - in 100 mm bump into

40 g Kern in 300 ml H<sub>2</sub>O.  
 → yellow foam - GAINS of ~~some~~ gum  
 mostly indole + glamine  
 a bit skatole in the 1 - wauwick -  
 a lot later in wauwick (1813) -

stand a day → fine white water ← same seed → retreat get  
 decant ag → leave white water  
 'leary yellow solidifying oil (our seed) - wts 100 wt  
 ethyls - separate, xkt ag. = ethyls  
 prod. xkt 1x200  
 1x100 ml 10% H<sub>2</sub>O<sub>4</sub>

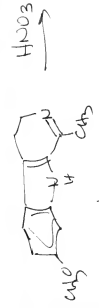
wake more = 25% base → pH ↑ 10, solids!  
 xkt 3x ~~100~~ 75 ml ethyls part fresh  
 wash - ethyls (still cloudy) ethyl yellow  
 of indoles  
 cloudy! -

skilled some getting 400 away from prod ethyls's  
 wash white solids looking at "normal ykt"  
 ykt a lot of ethyls washed  
 all the 50% base

yield ~ 11 g.  
 part of from before - bearing white wip 131-132  
 return water  
 went several start discolour from oak funnel

(Nights 10 min, 24 ethyls (fine) 112-114) (old 5. MODM 58-61°)

133



1HCl. 2H<sub>2</sub>O = 286.5

- 0.5g barium chloride. 1HCl in
- 8ml abs EtOH
- 8ml conc. HCl
- 0.25 ml & 1.42 conc. H<sub>2</sub>O<sub>3</sub> in
- 7ml abs EtOH



1HCl. 1H<sub>2</sub>O = 266.5

Done 2:12

to this, add

1 S.B. - almost hot - bubble bubble bubble!  
 30 seconds - off -  $\nabla$   $\bar{c}$  add water  $\rightarrow$  x'tals stand  
 filter - wash a bit of EtOH (sometimes would have  
 been better)  
 air-dry OP  $\rightarrow$  0.31 g pale yellow x'tals. (67.2%)  
 IR  $\equiv$  absolutely  $\bar{c}$  identical reference spectrum.

Save a small amount  
133A mp 267°

rest - to free base for GC clean deriv  
 verify IR,  
 dissolve in 3.4 ml RT H<sub>2</sub>O. 3.1 would have  
 been OK - add ~ 6-8 drops conc NH<sub>4</sub>OH  $\rightarrow$   
 globs of almost white solids  
 ground, stir, breakup, filter,

$\rightarrow$  let air dry, no 100° dry  
 small sample - without purification  
 for IR  
 for GCMS

0.22g  
 off-white  
 crystals  
 mp 255° dec?  
 133B

134

11 to 100 mg  
400



Ag H<sub>2</sub>O<sub>2</sub> was 250 = 4m

into 25 wt % solvent clean solution

plate mangle,

add (under nitrogen)

100 mg PtO<sub>2</sub> - 5hr

add

400 mg PtO<sub>2</sub> in 400 ml (3.6 ml) a few drops of a trimethyl

acetate 100 ml as needed to keep Pt acidic (a lot more than 4 ml unless you what)

20 minutes, then it would be needed.

Pt likes almost all solvents (PtO<sub>2</sub> in water)

Filter through paper → pale yellow filtrate - wash.

1hr - water bath @ 100°C → Pt white slurry.

Wt @ 3x50 ml ether

1% white solids in ether

2,3 wt % 50.

Flask on P.E. → 0.58 g white solids.

Sample wt

Flask residue @ 10 wt % Pt.

Starting Pt.

12.5 wt 8.12 drops H<sub>2</sub>O

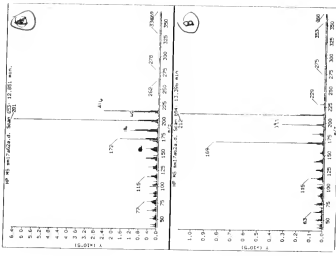
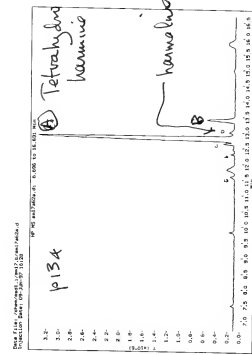
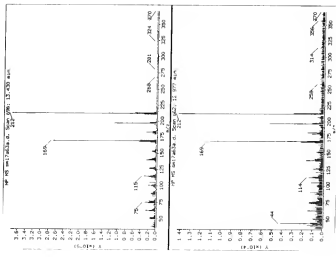
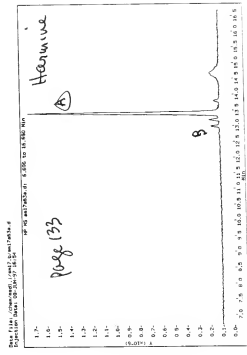
0.60 g.

Start RT 1 day

Filter, wash @ 100

0.58 g greenish solids  
15% yield  
134.8  
mp 132-134°

mp 187-9  
190-200  
134.8  
0.05 g  
recovery  
100%  
mp  
SR





#1

forming pyridine acetic acid, with low MTBE. not sat, Δ heat sat, ▽ heat sat. add

0.4 wt. oxydichloride - stir - some br? - Δ 58 - no deposit. Add 0.8 wt Brant. - Δ briefly sat.

add 25 wt sat Na2CO3 + darts of sat Na2CO3 water! pH → green on bottom (40 mols).

XHT 1 x 20 wt ch2z flask → 0.46 g white cr. pump in → still 0.46 g after small - no Brant.

#2

as above - no deposit - change solid diameter. add 0.8 Brant - worked over, less water.

15 culture of thingy from red-blossomed  
*T. grandiflorus*.

66g of shredded plant (weigh one here, 1 1/2" dia, 6" length)  
66 ml 25% NaOH - homo generous in 2 days - stir & wash  
xht 2 x 66 ml cells

cells:  
greenst  
xht 2 x 66 ml 10% HCl

cells

flask

0.38 g  
wily flake

137A

0.29 ml  
standing

GWT

flask

agaveas  
pale green

OH<sup>-</sup> 2 25% NaOH

when pH base,  
green → pale yellow?  
chlorophyll (green  
before washing)

↓ to RT.

extract 2x  
~ 25 ml cells

flask

137B-

alcohol's  
function.

0 weight

GCMS

See extract  
in lab-103

page 24

137C - same a bit for GCMS  
next (A1 mg) - find & reproduce

137

agaveas  
yellowish

neutralize

2 HCl (20%)

Juggle 2

5% NaOH

will  
want,

+ 60 ml

Sol

NaHCO<sub>3</sub>

(pale green  
pH flake)

pale yellow  
soln.

Wittmann

fraction

0.03 g

filmy  
slide

137C

agaveas

MS

MS

MS

MS

MS

MS

MS

MS

MS

MS

MS





(139)

from (138)

Final product  $\rightarrow$  1.14g Amber oil.  
distill. 0.2 mg/45 hrs

150  
160  
170

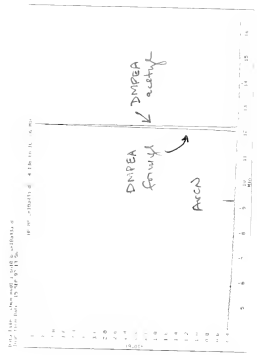
150  $\rightarrow$  170  $\rightarrow$  0.1 mm

160  $\rightarrow$  170  $\rightarrow$  0.90 oil white  
xtals.

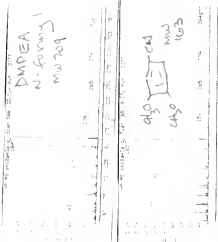
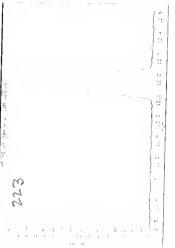
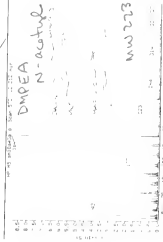
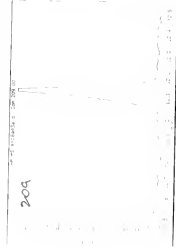
This is the amide  


Remore 0.64 | 1140  
Remains 0.74 mg

(139)



See page (41)  
Hexane used.  
cyclohexane used.  
meant to eat.



4.251 min

(140)



Dist g white dim killed solid (p 139). add 1.5 g  $ROCl_3$  (may be old - screen-cap cracked)

$\Delta$  90 1 hr  $\rightarrow$  deep red - brown  
add 20 ml the stand oil  
add more water. (shaking !!)

extract chloro

2 x 25 ml

water wash  
2 5% NaOH  
xmt 2 x 25 ml  
chloro

Chloro  
went to the brown color

0.11 g black oil  
summit  
(140A) out

repeat page 144  
flask

0.49 g  
pale off -  
white solid -  
warm!

(140B)

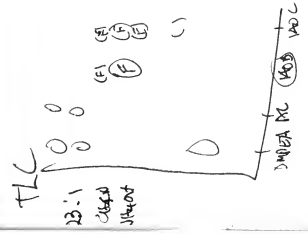
Save 100 mg  
the rest  
reduce.

into dilute  $H_2O$  0.39 g

+ 5 x 5 test tubes

strong yellow  $\rightarrow$  pale yellow  
out - xmt - clear -

$\rightarrow$  0.35 g @ 130-140 / 0.5 microns  
140C.



DMPEA MC (14) (15) (16) DMPEA → acetamide p 138

Repeat - weigh out & wash dead.

3.6 g amine (+ carbamate) ~ 20 ml

6 g pyridine

2.5 g base

→ onto SiO<sub>2</sub> ~ 10 minutes. V + 100 ml tbo -

make strongly basic ~ 25% NaOH - x 1.5 + 25 ml CH<sub>2</sub>Cl<sub>2</sub>

color into ether (amber) - wash 2 x tbo (boil-H<sub>2</sub>O)

slight → 3.44 g amber oil

→ 2.46 g white oil 170°-190° / 0.2 mm/Hg x tbo

all to page 144.

0.15 mm/14g

150° 0.2 mm

170° white oil

180° characteristic (oil)

190° still considerable pot off

See CMS

page 139

an. side - characteristic thin amide 0.13 g from p 139

distill in 0.13 g boiling stop. let cool → insignificant x tbo.

filter (p. plate) - wash repeats ~ 5 tbo.

mp 97-98°

Van 18.

See CMS.

142



144 A



Page 144



144 B

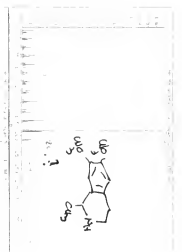
MW 191

MW 205

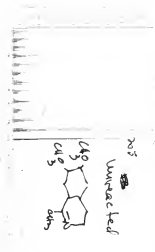
Page 144 A  
 Page 144 B



205 ion  
abstraction

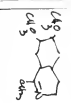


NO  
str  
ratio



205  
unreacted

NO  
str  
ratio



Trial



143

from page 144

part of 0.84 g white solid, fused x tabs.

0.40 g. of distilled "acet" =  $\sim 2$  mm.

1.0 ml 38% (40%)  $\text{CH}_3\text{OH}$  =  $\sim 13$  mm

0.5 g  $\text{NaOH}$  =  $\sim 13$  mm.

AcOH = 60 .05 ml =

6 g = 100 mm

.6 g = 10 mm

.12 g = 2 mm = 2 drops +

into 20 ml  $\text{H}_2\text{O}$

+ 4 drops  $\text{H}_2\text{O}$

+ 1 ml 38%  $\text{H}_2\text{O}$

+ 0.6 g  $\text{NaOH}$  - a dash at a time - leaning.

all in. base +  $\sim 30$  drops  $\text{H}_2\text{O}$   $\rightarrow$  pH 7

stir  $\sim 30$  minutes - add

5%  $\text{NaOH}$   $\rightarrow$  pH blue. x 4 x 2 x 25 ml  $\text{CH}_2\text{Cl}_2$

Slush. 0.44 g white oil.

< 0.14 g

0.054 g 105-120°

#143

#143

0.36 g white oil

+

78% 1 ml

20% 2 ml

20% 2 ml

traces  $\text{N}_2$  - diethylamine

traces  $\text{H}_2\text{O}$  219

turning slowly yellow on standing - no  $\text{CO}_2$ .

deep brown in 4 minutes.

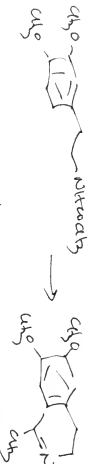
into simt. visl

same 143

Spectra 162

(144) extract of page 140

See  
sketch  
p. 142



2 g of amide (2141) (all volatile) added  
 10 g  $\text{PdCl}_2$  - onto sp. 1 hr. filtered with water and  
 spun (in hot) & lost a hole of it into the  
 lab bench

1.16.  
 weak acidic  $\text{O}^-$   $\text{CH}_2$ ,  $\text{OH}^-$  95%  $\text{NaOH}$  -  $\text{CH}_2^-$   
 $2 \times 50 \text{ ml } \text{CH}_2$  - flask  $\rightarrow$  1.16 g residue that is  
 a fine tan solid.

save a small (149) for cens. (144A)

vert 1.08 g  $\tau$  into water 100 to  $\sim 2$  g  $\text{HCl}$  -  $\text{NaBH}_4$   
 on small volume - old white no residue to keep  
 acid  $\text{S} \sim 7.5$  - low yields at 8.6 g/hr. - this

should be another experiment - accept as such - work  
 $\bar{c}$  2.25 ml  $\text{HCl}$ .

add  $\text{NaBH}_4$  spm by stream - note as reacted to keep  
 acidic.

after a gram in two of  $\text{NaBH}_4$  - oxygen sl. acidic.

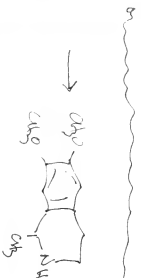


the water phase  
 under wash



2 colorless  
 xh t.  
 $\text{CH}_2$

yellow  $\text{CH}_2$   
 0.06 g out -  
 run in all the carbon column



$\xrightarrow{\text{O}^-}$   
 0.15 gm  
 130 - 145  $\rightarrow$  white oil  
 0.9 g oil - almost  
 0.9 g in flask xh t.  
 0.84 mg out  $\bar{c}$  breaking  
 reagent, down.

144B

to page 143



MW 200 = 5 mM

1.0g MDREA. 44 ancient reference sample Box 1A15!

#11 to 141 into 50 ml H<sub>2</sub>O (brownish soln) + 5% NaOH → blue xht 3 x 25 ml CH<sub>2</sub>Cl<sub>2</sub> flesh → 0.82g tan oil

1.5g pyridine

0.6g Ar2O Δ SB ~ 10 min → RT acid v. 40 ml H<sub>2</sub>O + a couple of ml 25% base. xht = CH<sub>2</sub>Cl<sub>2</sub>

\* oil changes  
same as 141

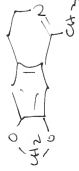
wash pooled chcls, 2 x 25 ml 10% Et<sub>2</sub>O  
flesh → off. white solids 0.54g.

GENS One fresh - good structure  
see page 150

neutralize a bit  
from Stoffr. oleostearic

Volat - see 209, 215

POCl<sub>3</sub>



0.52 (about) xhtaling amid above = 1.5g POCl<sub>3</sub>

onto SB 45 ~~min~~ minutes → 7 to RT. Looks

like stels coming out. Is strongly acidic -

wash 2 x 25 ml CH<sub>2</sub>Cl<sub>2</sub> - CH<sub>2</sub>Cl<sub>2</sub> is 25% NaOH - very

cloudy - xht 2 x 25 ml

CH<sub>2</sub>Cl<sub>2</sub> - flesh →

white → quite a

bit of double emulsions

(amide?) 0.17g

under STPE gum.

to acetone - then

solids. OUT

0.70

0.61g

GENS Part on P150

trace of 78-15mm

trace of aromatic Ia.

See volat page 216

145



NITRO CH<sub>3</sub>



1410



0.55 g mDove into 20 ml H<sub>2</sub>O + 10 ml dmf  
 until into solution

an original weight of 3H<sub>2</sub> was added, with preservative  
 depth of H<sub>2</sub> added to main from ~ 100 ml to pH  
 10. n 2 hr. dilute to 50 ml, 0.1% = 2.2 x 10<sup>21</sup>  
~~2 x 10<sup>21</sup>~~ ml water - shift → 0.45 g deep carbon  
 oil - to K.R. 0.04 mm / 145° → 150° white oil.  
 solvent no ppt. 0.40 g absolutely  
 white!

on the cards.

uncovered old flow sheet  
 to 20 ml - 8.4 T

14.2 g H<sub>2</sub>O  
 13 g H<sub>2</sub>

All top flow 2 in 805  
 white with everywhere  
 with 50 ml H<sub>2</sub>O; water going  
 home, not clear, flash,  
 + 9 g H<sub>2</sub> + 5 g pyridine  
 1500 20 min + distill H<sub>2</sub>O;  
 2500 same - clear - xvc  
 2 1/2 hr - ~~to~~ to dry acid  
 2 1/2 hr with xvc - flash  
 → 1.19 g K<sub>2</sub>O · 0.05 mm  
 130° - 145° - white oil  
 0.40 g - xvc.  
 overall wt. 0.33 g.

(147)

isolation from yellow-blossomed *T. grandiflorus*.

140 g green ground-up centers - add  
140 ml 25% NaOH - stir w/ mortar. (2 days - virtually  
all w/o solution - some except some centers) add = vol the  
extract 50 ml ether - leave to centrifuge to separate  
phases -

ether → eg. →

dark yellow - flesh -  
weight nil.

was small vol (2 ml) ether

GCMS - on 1/2 of it. (147A)

other half.

+ a couple of drops pyridine  
and a couple of drops H<sub>2</sub>O

GCMS (147B)

no DMT, gramine ~~etc~~  
obvious.

148) Do another red-browned T. grandiflora. Red #2

Run one for factor, for overall slides.

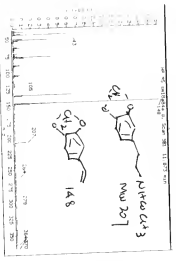
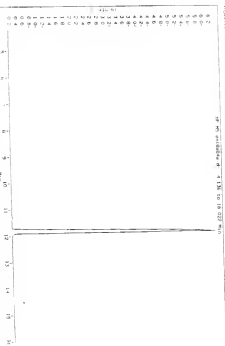
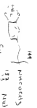
one banana-skip casher 244g - alives so pairing, try  
dum-ice gene we want do it! Try on all spines with the red  
cliffers

4.2g spines }  
+ shoot + dist } → 22g brown group. add 400 me # 1.5 N H<sub>2</sub>O  
into steam bath ~ 3 Pm.

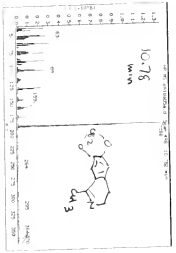
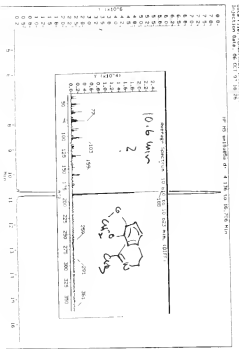
Red Do yet another (I think it is red) = wild and (149)  
#3 One volume by large ~ 8 cm dia, ~ 25 cm long cactus  
472g, worked up to slimy pulp - add = volume (470ml)  
2.5% citric acid solution.

150

DATA FILE: C:\MSDCHEM\145B.DAT  
DATE: 01/11/83  
TIME: 11:30:00  
INSTR: FTIR  
PROC: 145B.DAT

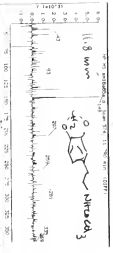
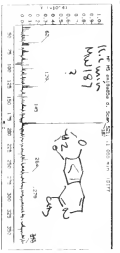
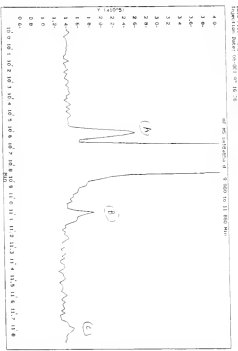
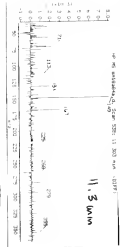
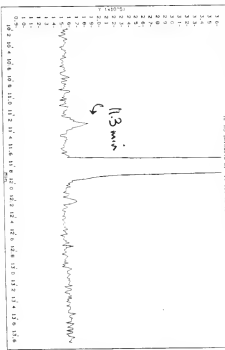


1458A  
1458B



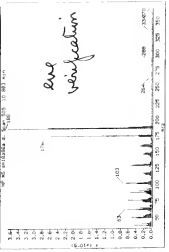
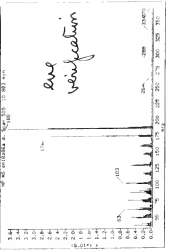
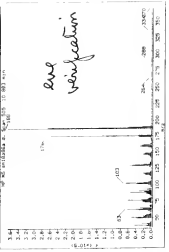
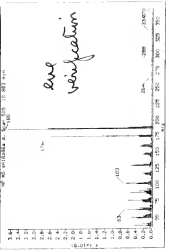
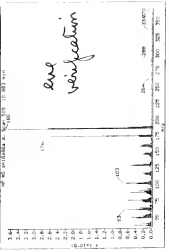
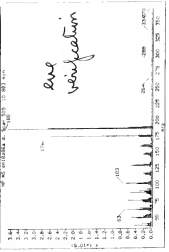
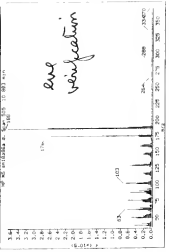
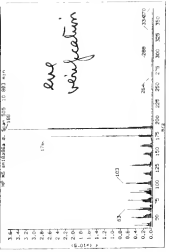
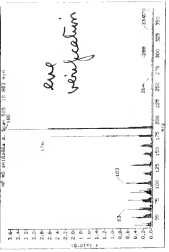
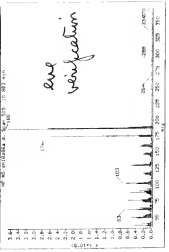
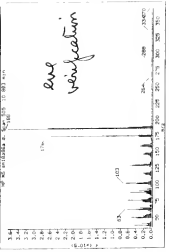
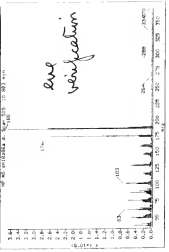
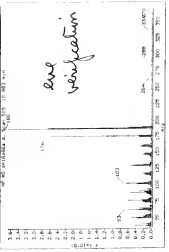
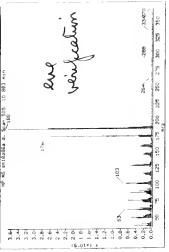
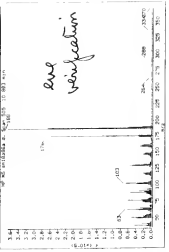
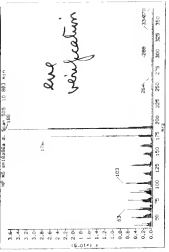
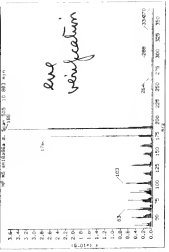
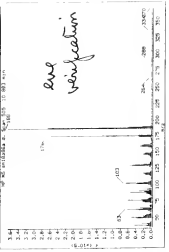
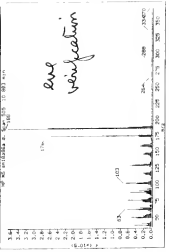
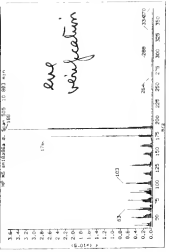
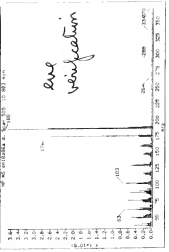
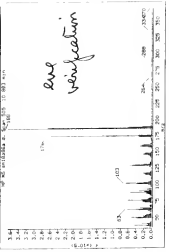
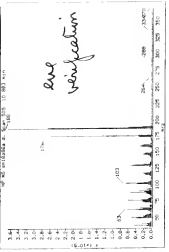
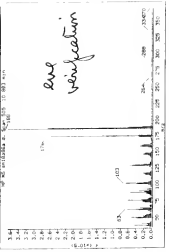
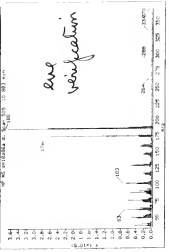
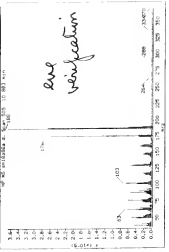
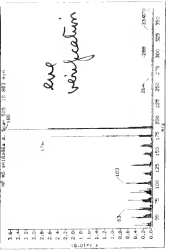
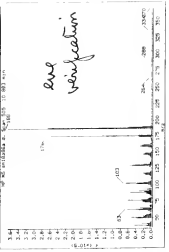
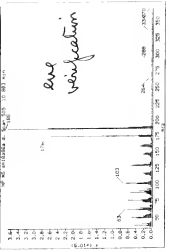
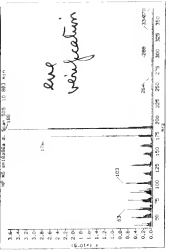
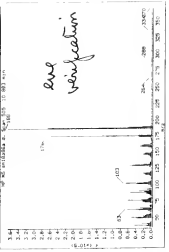
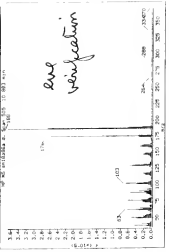
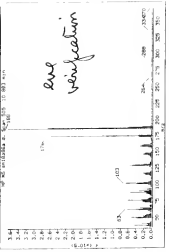
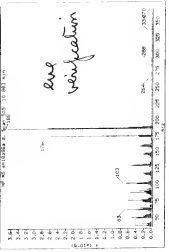
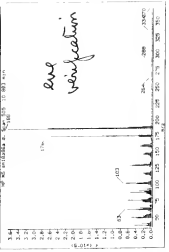
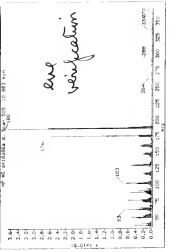
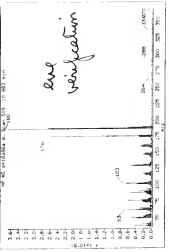
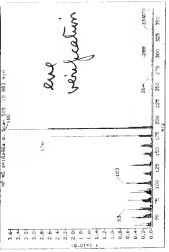
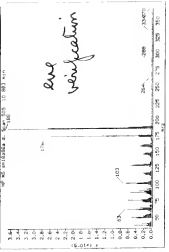
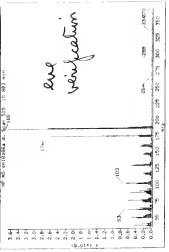
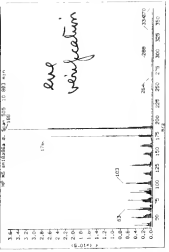
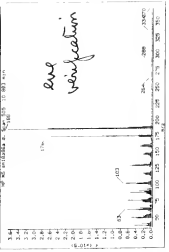
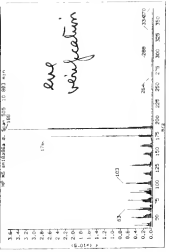
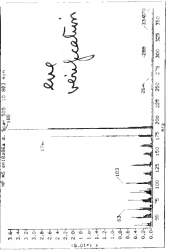
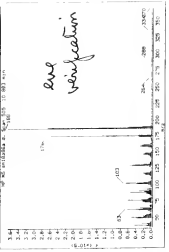
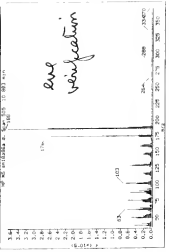
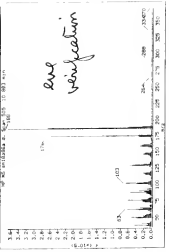
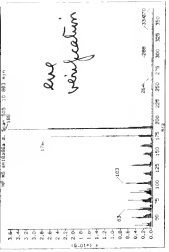
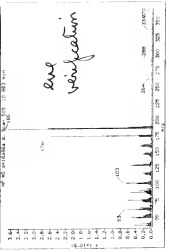
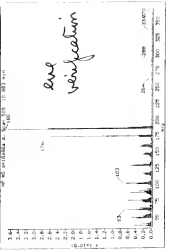
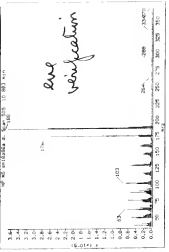
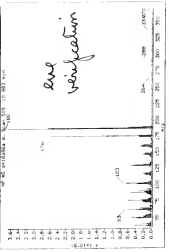
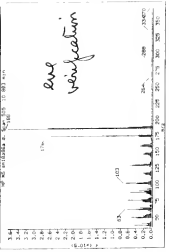
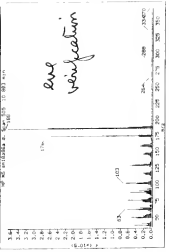
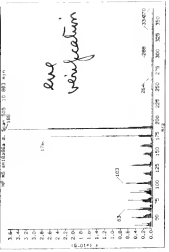
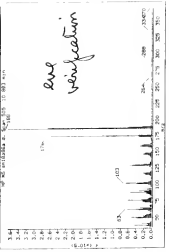
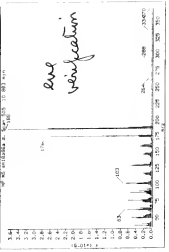
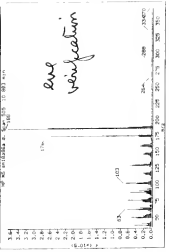
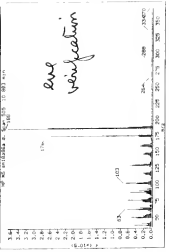
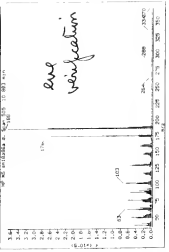
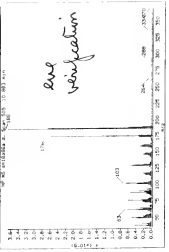
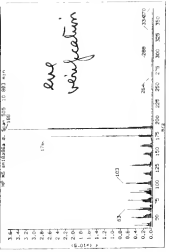
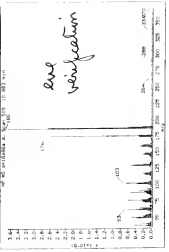
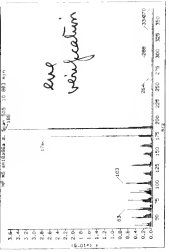
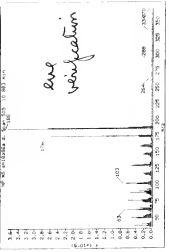
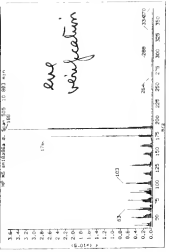
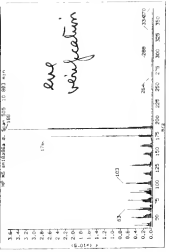
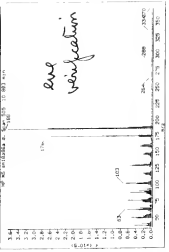
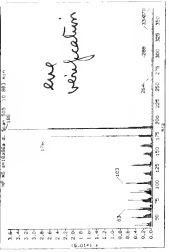
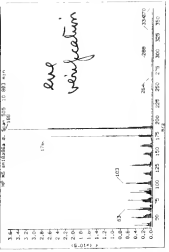
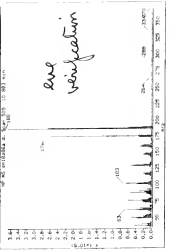
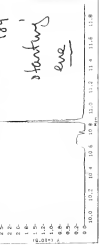
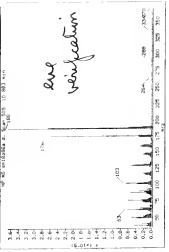
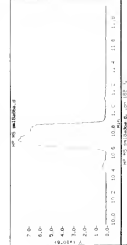
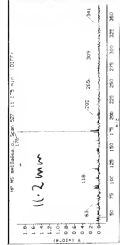
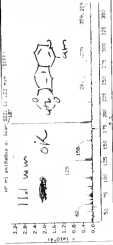
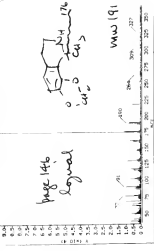
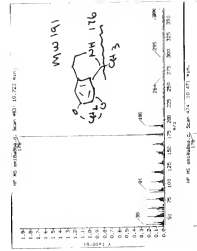
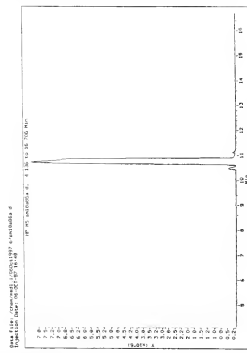
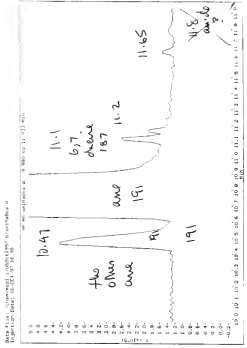
from 145A

DATA FILE: C:\MSDCHEM\145A.DAT  
DATE: 01/11/83  
TIME: 11:30:00  
INSTR: FTIR  
PROC: 145A.DAT



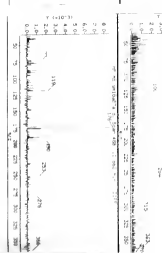
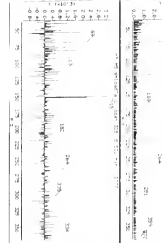
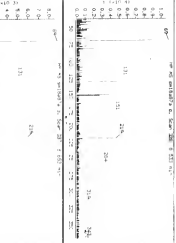
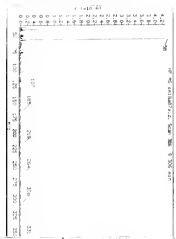
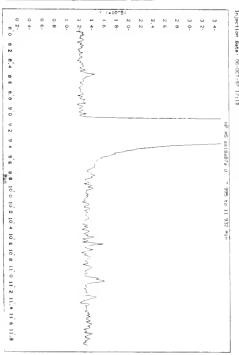
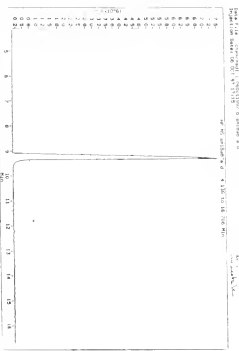
From 146 6,7 MDO Meth H4

151



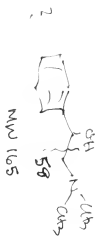
(152)

PH 9 fraction from Rad #1 page 137



waxy lactid  
page 137.

spikes



Try!



Initially - 5g 5-HT-170 to fuse base, alkylate, o-styrylate - there you are!

5.16g separation. H<sub>2</sub>O into ~ 50 ml 170 soluble - make basic - sat NaHCO<sub>3</sub> - stir ~ 3 x 50 ml EtOAc -> nothing! pH back to neutral - etOAc - nothing!

Add a bunch of NaOH - extract in etOAc -> something. + a glob of brown gum in sep funnel. - wash in etOAc.

Add 10 ml MeOH - soluble! - flask -> 0.4 g fluffy film

4g Sulfurane - largely dissolves in Δ.

1.3g diisobutyl-ethylamine

1.6g isopropyl iodide. (last of it, vendor)

Δ sep in occasional swirling. 3 hrs.

Sample removed - Ac2O (B) -> separate for GCMS

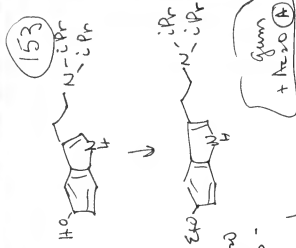
Post + ~~2.7g~~ 2.6% NaOH

~~2.7g~~ 5g EtBr - stir ON.

5g a few minutes in sep. - stand on.

both sample (A) and sample (B) pretty much unrecoverable

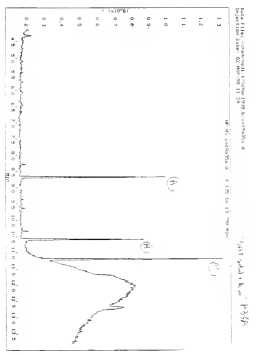
See next page



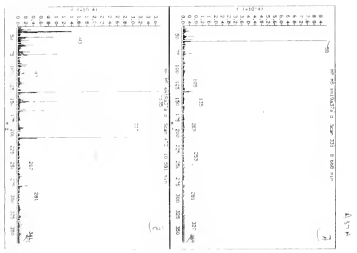
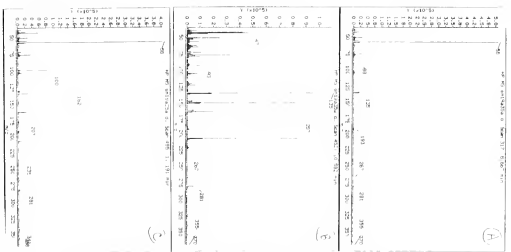
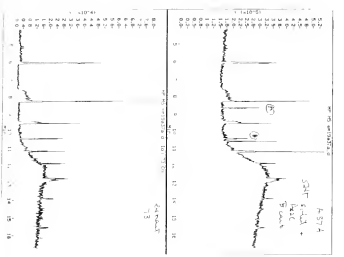


154

Sample A ('sensitiv' A Arzo)  
page 153



Sample B (after PCT)  
page 153



Outyept:



155

10 mM. - under nitrogen

2.12g serotonin. HCl in ~10ml H<sub>2</sub>O, add 25% NaOH

(0.4g) 1.7ml neutral water stir anywhere

+ 1.7ml pretty much goes back into solution  
+ 0.5ml more, for good luck

2.0g (1.0ml) (30% xs) EtI.

stir RT ~ 2 hrs under nitrogen - not much  
discoloration

xht ch<sub>2</sub>cs<sub>2</sub> (color stays in aq.) (open to  
air - rapid darkening).

flush xht → almost nothing

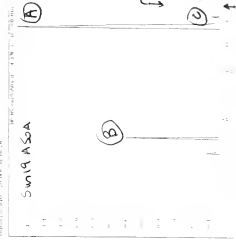
run in 90/10 anyway  
major product -

Sm19 a 50 a



Sm19 A50A

(B)



(A)

5-Eto-T 11.67 min

5-Eto-NET 12.11 min

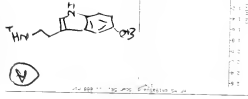
5-Eto-DET found by  
sample run 86  
12.56 min

5-Eto-DET

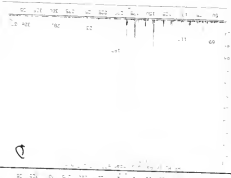
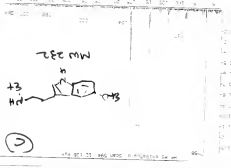
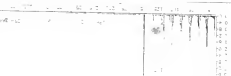
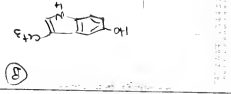
(C)



(D)



(E)



156) again - try aush!



2.12 g zero-toning H<sub>2</sub>O - water 20 ml aush. DMF under Xe goes totally into solution !!!  
 10 mM. — add 20 mM. <sup>prod</sup> at 129

4.0 ml di-n-butyl-ethylamine  
 looks a bit turbid at end of addition - then clear  
 = 3.504 mL  
 dm. 0.742

160m

30m

2.0g (1mL) STI - stir.

Sample out at 0.5 hr. against. + 5x 100 clean  
 + drop of D<sub>2</sub>O (doubly!) xrt 90/10 Sun 19 AS1A.  
 covered by triup, MS = major peak 105  
 trace of 5-thi-DET?

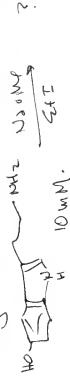
wrote up - slightly warm from stirrer. Into 100 ml H<sub>2</sub>O (in al. vial) - extract quickly as is with 3x 40 ml ether (Sun color light xrt) - part-extract 2x 50 ml dit H<sub>2</sub>O, water eq. waste & ammonium hydroxide - xrt 2x 50 ml ether part - flash → check off quickly - ~~some~~ much residue (if resist?) off more slowly → final residue of ~ 1.9 - no number! so is OLS aCMS in 90/10 Sun 19 AS2A. H<sub>2</sub>O/L One heavy peak - no spectrum - search for 58 & 66 → nothing.



R=H (141) R=H (58)  
 R=st (174) R=st (86)

∴ M<sub>2</sub>'s to look for  
 H H 204  
 2+H { 232  
 H 24  
 2+ 24 260

Again - try MeOH a/c.



2.12 g serotonin HCl - into MeOH 10 ml sat !!

1.1 g NEMe (20 mM) then 1.08 - in MeOH 10 ml sat with exotherm

under argon - add MeOH soln to serotonin & swirling - white solid out which do not redissolve (NEMe?). Strip well on rotary.

Residue - brittle - heavy oily stuff - some solids around the perimeter. Not nice. Add 0.81 ml (1.56g) EtI - in 10 ml EtOAc

Can't see scraping  $\rightarrow$  goes sublimably (surface) white x tabs (NEMe?) scrape, scratch - more and more x tabs. Dramatized. Let stand under xenon for a couple of hours. White solid !.

Swofend in 100 ml MeOH (clean) (pH 11.0) acidify &

dil (1M) HCl  $\rightarrow$  red. wash  $\approx$  2 x 50 ml EtOAc - base

25% MeOH - xHCl 2 x 50 EtOAc - some little bit of the brown color does go into the diether. - prod - flash  $\rightarrow$  small amt - viscous oil.

GCMS page 158

n = a/c  
5-ETD-T  
5-ETD-NET  
5-ETD-DIET

heat i acetic anhydride  
kill NEMe - xHCl 2 x 25  
xHCl  $\approx$  dil HCl 2 x 25

MeOH -  
CHCl<sub>3</sub> wash  
 $\approx$  100 - flash  
 $\rightarrow$  80 mg  
MeOH

aq. water base  
3x HCl 2 x 25 ml EtOAc  
flash  $\rightarrow$  80 mg

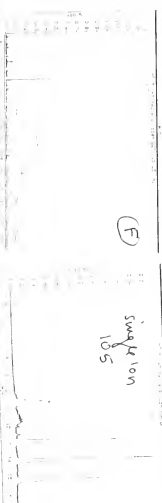
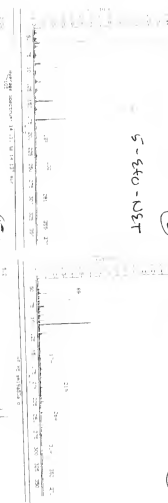
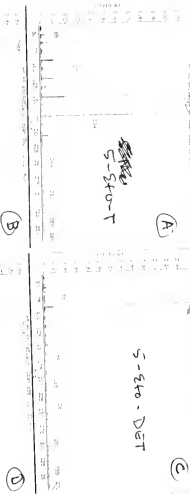
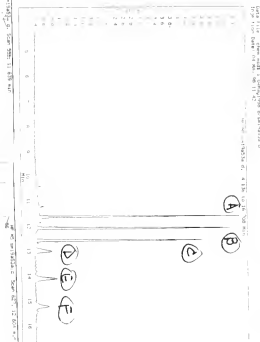
test amine fraction (10% HCl other  
no tabs)

spectra 159

spectra  
page 160

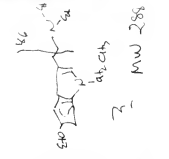
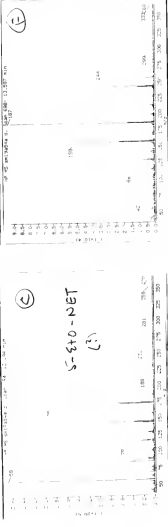
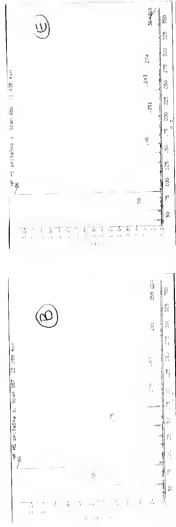
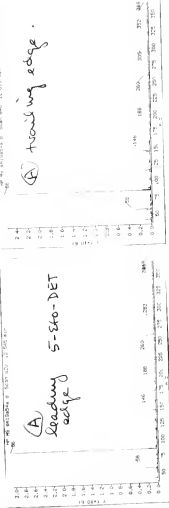
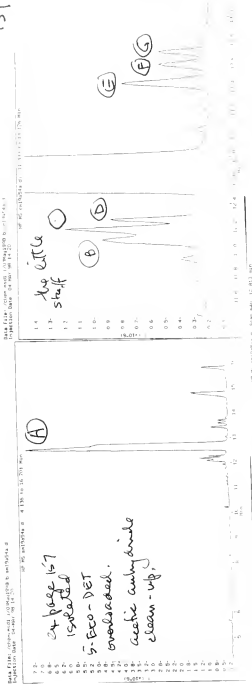
158

Spectra from 157 S-HT, NaOH, 2TI



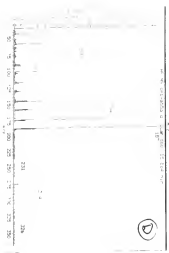
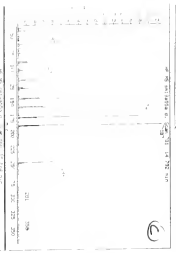
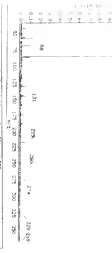
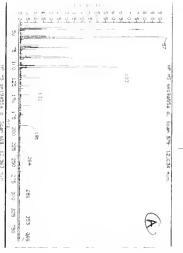
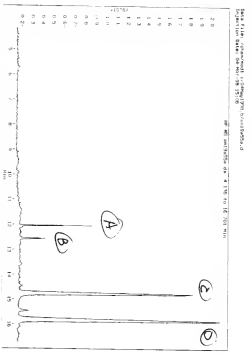
Swaption  
105

159

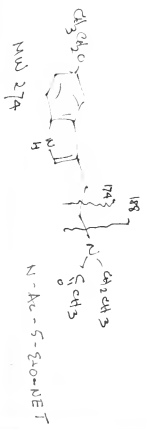
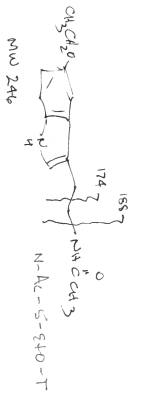


150

acetylacetal neutrals - see page 157



5-ethyl-DET



Small scale experiment

161



CH<sub>3</sub>I



NaBH<sub>4</sub>

a few mg H<sub>2</sub>O → into water → clear yellow soln.  
add a few drops N<sub>2</sub>SO<sub>4</sub> → white cloudy - xht i  
~2 ml CH<sub>2</sub>Cl<sub>2</sub> - evap → fine beautiful white xhts.

SM19 A63A Sample TLC(A)

dissolve in ~1 ml CH<sub>2</sub>I - never really dissolves but  
gets immediately yellow - should have added hindered  
base? let stand ~3 hrs. CH<sub>2</sub>I - evaporate sample TLC(B)

dissolve in H<sub>2</sub>O OK, + dil H<sub>2</sub>O OK → yellow soln -  
add two small spatulas of NaBH<sub>4</sub> → colorless

but ~~was~~ no white cloudy. xht i ether - evaporate  
→ smoky that on spatula scraping → yellow solids!

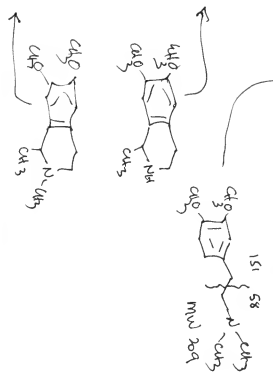
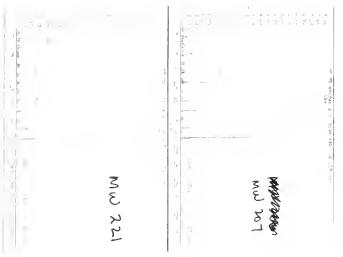
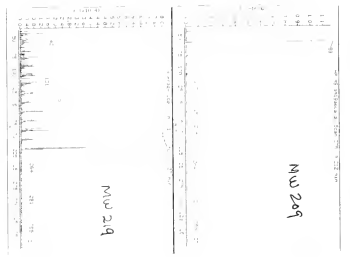
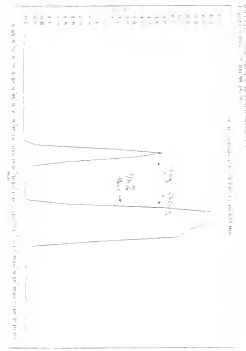
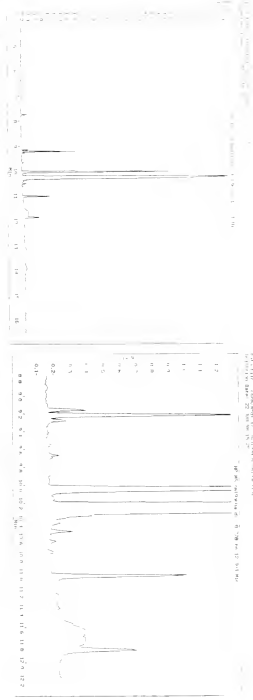
see page 164 for barrier/barreling resolution

Sample TLC  
(C)  
SM19  
A64A



162

from page 143 incomplete DMMM



161

Push the rest of the way.  
DMMH  $\rightarrow$  DMMM.



never did push it - used the great intermediates and that went very smoothly (see page 20's)  
reworked 143 9 months later - deep brown oil essentially  $\equiv$  spectrum

(163)

164 Let's straighten up the Sigma Humaline/lanamine where the red sigma humaline the lot 123N (123N 3717 affixes) to the lanamine by GCMS page 161

Ref. lanamine

Humaline the TCI lot A501 I break seal. 10 wgt into  
~1 wt (to seal) + 5% NaOH - dices  
empty → solid. (A) 5M1A A65A A73

Humaline base, p133b - humawade, by synthesis of humaline  
Humaline the, Radio - lot 229 into 1 wt <sup>(B)</sup> ~~the~~ the - + 5% NaOH  
→ slowly - shot 2 white - equal →  
a white film (C) A67A

Ref. Humaline  
Humaline (base?) Sigma lot 28C-0433 - into dices -  
equal → pale yellow film. (D) A68A

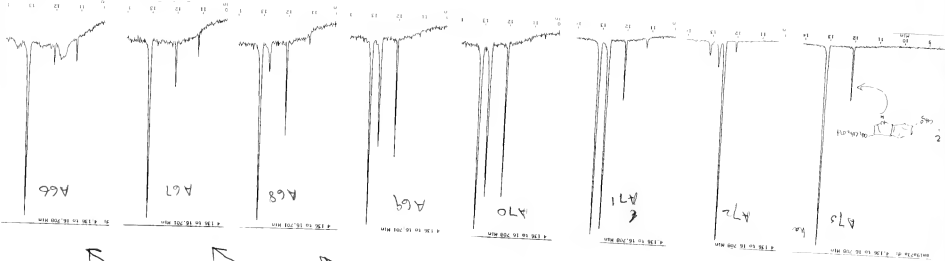
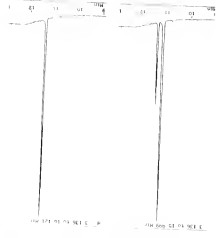
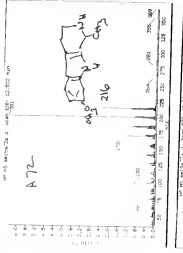
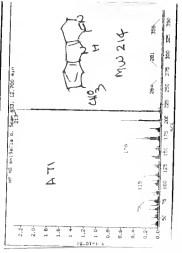
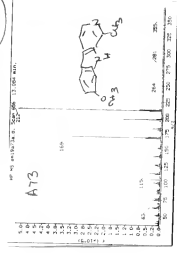
Humaline (base?) Aldrich, lot # 01321AV I break the  
seal: into dices, (set) and equal →  
yellow (pale) film solid. (E) A69A

Humaline (base?) Aldrich lot # 11226PX into dices -  
equal → film of solid. (F) A70A  
Humaline the Sigma lot # 123H 3717 - this is the  
SPECT sample that precipitated this  
white thing. See in lot the base:  
5% NaOH (G) A71A

Ref. Tetrahydrolanamine  
Lafco lanamine (base?) Radio lot # TL549 P1-70  
into dices - sample solid film. (H) A72A

A-73A - yellow of A65A -  
A-74A - yellow w/ film lanamine

165



same paper samples clean column

code 127  
another literature  
code 239

Mass  
Spectrum

166

170

170

170

170

170

170

170

170

170

170

170

170

170

170

170

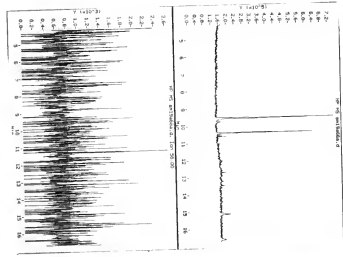
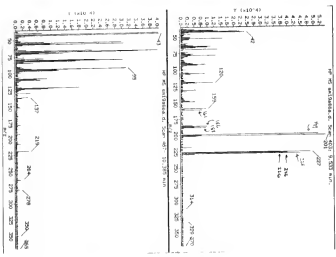
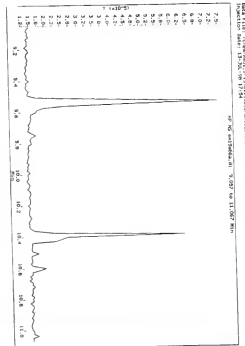
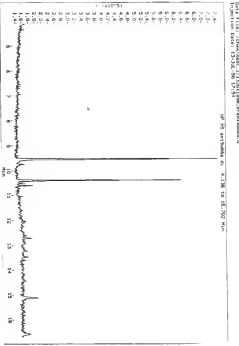
170

160

Extract.

1189 aerial part

S and HO - thymophy swatch + count in Nant-  
xht c 1/2 was ch. d. - plus → identified green  
solution - released - Peak - int w/4 was 90/10



58 161  
180 188  
199 201

226  
227  
228  
229

157

157/41

1189

1189

(167)



|| p 121

2.46 g TCI amine - dissolve in  
 18 g warm (50) Sucofolane; add  
 6.5 g  $\text{H}_2\text{O}$  and  
 9.4 g iprI TCI

$\Delta$  in SB bath. - Stirred RT in 5 days - 1  $\phi$ ! why bother  
 to strip in R.E. 2

add 150 ml water  $\rightarrow$  2  $\phi$  (heavy oil, red)

Kft  $\bar{c}$  35-60 per other 3 x 50 ml  
 heavy strip  $\rightarrow$  1.16 g deep amber oil. slight brown

-182.47

-39.77

transfer  $\bar{c}$  ether to KR flask 0.92!

no smell!  
 (then ugly)

KR!.

0.2 mm

95°/40 mm cloudy.

flame - flash into  
 sunbath. -

up Temp.  $<$  155/40 mm. white oil. trivial black ppt.

0.57g.

0.57? more? balance crafting

into 2.6 ml IPA

+ some ether  $\rightarrow$  red = 23 drops!! stabs

+ another ml IPA (still stabs)

+ diethyl ether 2.0 ml fine stabs

Soliter - wash  $\bar{c}$  10 ml 3/2 IPA/ether  $\rightarrow$  white stabs

0.82 dry?

ml out.

(106)



11 p 135

9.0 g fresh - clean amine from TCI add  
 15 ml pyridine add  
 6 ml H<sub>2</sub>O - quite hot - let cool to RT over 1 hr  
 + 150 ml H<sub>2</sub>O - some H<sub>2</sub>O  $\rightarrow$  used (a few ml)  
 xHCl  $\approx 3 \times 75$  ml HCl  
 prod - wash 1 time  $\approx$  50 ml 50% NaOH. Refry.

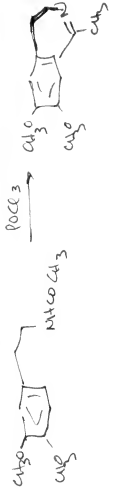
K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> every culture - know for  $\leftarrow$  I don't know the balance  
 to K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> pot  $\approx$  10.58 g solids.

0.15 min RT W.R.  
 200 gr. 100° washing.  
 150 gr. 140° washing.  
 150 gr. 150° up water.  
 165° over - white beautiful.  
 175° 1/2 over. cool  $\approx$  10 min  
 180° 10% a/c.  $\rightarrow$   $\approx$  10 min  
 190° done. vacuum clear

Gurukul Evapo  
 mp (p. 141) 97-98° at 500k  
 MS (p. 134) 164 (lit), 150 (lit) (49%)  
 140 (23%) 225 7%  
 present

9.73 in vacuum - don't burst  
 P. 63 mp's cold  
 preserved.  
 pale yellow solids -  
 9.50 clean almost white.  
 to right  
 weight  
 0.50 g same  
 9.00 g to p. 104

1691



11 p 144

9.0g distilled amide - add  
 52g fresh, clean POCl<sub>3</sub> - onto SB 4:40 PM.  
 at first starts to dissolve - first  
 heating → some bubbling - hope vent to floor better  
 → 40 min it - covered wet towel - no fumes in the lab.

Off in 1-hr. add 2 great lantins to 300 ml H<sub>2</sub>O.  
 extremely exothermic. all  
 stand (outside until RT) in finally -



xrtt  $\bar{c}$  3x60 ml CH<sub>2</sub>Cl<sub>2</sub>  
 water basin  
 $\bar{c}$  25% NaOH  
 RT cool xrtt  $\bar{c}$  3x60 ml CH<sub>2</sub>Cl<sub>2</sub>  
 back wash  $\bar{c}$  1x50 ml H<sub>2</sub>O - flask →

0.14g  
 brown  
 remainder -  
 out

200  
 work - active  
 → fine white solid  
 tho sol. colors  
 when had some H<sub>2</sub>O  
 1691A

7.27g vacuum - 3x60 ml CH<sub>2</sub>Cl<sub>2</sub> sub IPA. 1691B

Kugelrohr des 100v 100°  
 110° smalling  
 130° almost all

main vacuum 6.85g  
 scrub out, including some foam  
 2nd receiver 6.98g  
 Sublimed, largely black smy  
 lignite. 1691C

Sublimed stuff mp 102-106°  
 1691D → Kugelrohr des vacuum up 104-106°  
 → 200 mg in 1 ml IPA - 5 drops  
 H<sub>2</sub>O + 1 ml ether → extract 206-7 dec  
 1691E

SM to ANGA (sublimed) 1 (white)  
 ANGA (white) 2 (white)  
 MS for others



(170)



9.0 g fresh steam DMPER. add 20 g  $\text{H}_2\text{O}$  in  $\text{CH}_3\text{COCH}_3$  into steam bath 5:45 Off in 1 hr. Add

n 200 wt thw - initially steam, then slowly.

Conc. there  $\rightarrow$  red - xht  $\bar{c}$  3x50 wa chiller - part - all color (anion) goes into the steam. Above  $\bar{c}$  50 in  $\bar{c}$



5% H<sub>2</sub>O - almost no color remaining.



heavy brown oil - 10.95 g - kind of some acid



In to K<sub>2</sub>CO<sub>3</sub> part -  $\bar{c}$  some bicarbon. into the pump. Pump for 2 hrs  $\rightarrow$  100 yr.

130° 250 yr  
 140° 500 yr Keeping vacuum thru heat up

170° 500 yr start  
 190° 400 yr done - almost vacuum oil - that was subproduct.

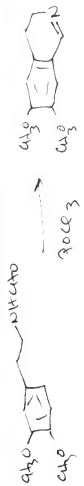
GCAN'S  
 164 (100%)  
 151 (68%)  
 149 (19%)  
 209 (15%)  
 165 (10%)

8.70 in main flask

Save as reference n 0.5g mass for thru next  $\bar{c}$  chills for POCl<sub>3</sub> p111

9.0g cell  
 In yield  
 8.43 (a little stuff used in the 2nd K<sub>2</sub>CO<sub>3</sub> flask.

GCAN'S n 96% conversion  
 n 5% DMPER  
 202 purg 173



8.5 g formamide (44.6% to remove amide from KR voluins - strip n R.E. - add

42 g POCC3 → swirl - evap. & pretty strong building - outside! - when quiet (and still quite warm) onto S.B. 7 PM off - 1 hr. Stand ON - into ~ 300 ml H<sub>2</sub>O - controllable -

forget to wash c CHCl<sub>3</sub> - beac i 25% N<sub>2</sub>O<sub>4</sub> let cool. - what is 3x75 ml CHCl<sub>3</sub> - prod - wash 1 time = 50 ml H<sub>2</sub>O -

Scam → 6.95 g amber oil - swidge in on x bed dish in lab. AM - some xstals (solid), not can D think) + some minor xstals in flask - (not wash - nice: - same bath.) 6.95 g on precipitable balance. Transfer to KR pot -

40 μ in hand setup  
 140 μ. setup  
 Δ → n 70°  
 150 μ.

200 μ 130° over  
 white  
 vacatified,  
 140° x 400 ftft  
 150° over  
 say 13.5-145/200 μ.

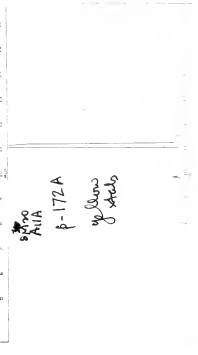
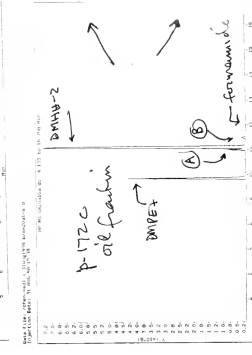
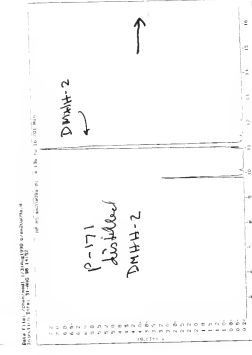
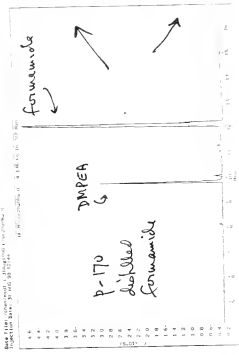
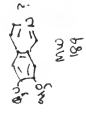
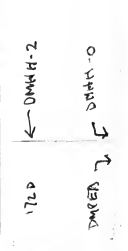
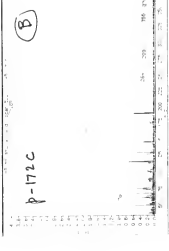
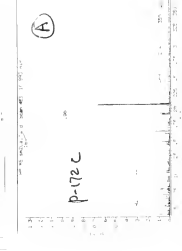
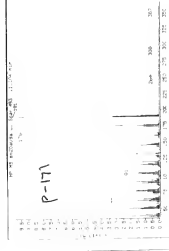
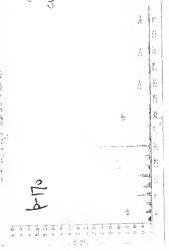
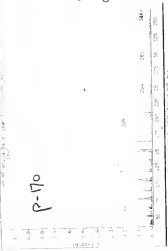
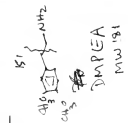
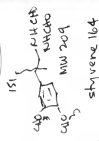
6.64 g ← .05 + 6.59 g white oil c  
 ↓ some in 2nd receiver.

ACMS  
 solvent entrols  
 DMTHT-2  
 some DMPEA  
 see page 173 for  
 spectra  
 50 mg.  
 try seed, 171A  
 without H<sub>2</sub>O. ACMS  
 a little bit - into IPA  
 + H<sub>2</sub>O → yellow xstals.  
 mono.

convert all to H<sub>2</sub>O  
 strange - on to page 172

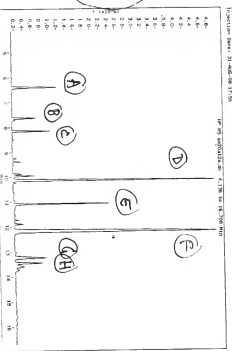


(173)

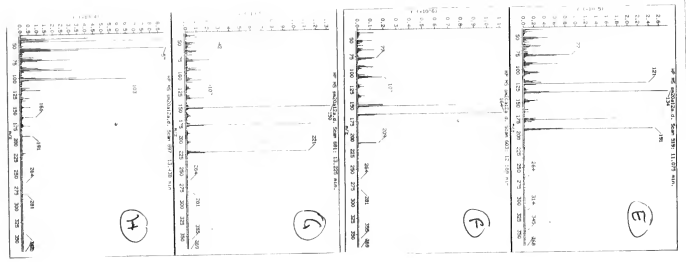
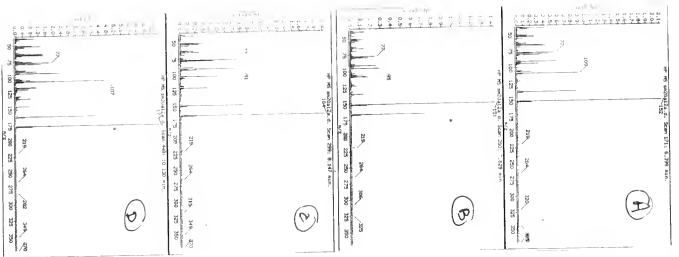


174

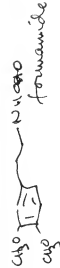
from  
172B  
sample



partial of  
acid fraction  
→



(175)

(F) is definitely  Nicotinic formamide

(D) is maybe  a zwitterion, a neutral

172 B looks more like 100 mg - deep yellow liquid oil.  
page lets see if I can get D out of it.

Make extremely strongly basic (boiled as the anion?)  
that  $i$ -thick<sub>2</sub> (all color goes into org - too bad!)  
take aqueous (25% base) + HCl until ppt ved.  
let stand - ~~2~~ KM - some color was x tabs -  
white Naze! OUT

---

176



DMHH-4

2.55 g DMHH-2 (172-D - presumably DMPEA free) into

some H<sub>2</sub>O containing maybe 1 ml HCl. Stir - add  
~ 2 g NaOH (1/2 in - re-run warm - water - and -  
3/4 in add 1/2 ml more H<sub>2</sub>O - add rest - color  
vanish readily faster!

add some more 100 ml H<sub>2</sub>O, H<sub>2</sub>O to pH blue (5%<sub>2</sub>, then  
25%). cloudy. xrt 3 x 50 ml ether (same emulsion)  
wash 5 x 25 ml H<sub>2</sub>O. Dry -> 2.31 g very pale oil  
to KR.

150  $\mu$ .  
200  $\mu$  110° w

135-140 (with 135) 150  $\mu$ . white oil  
135-145° @ 150  $\mu$  1.80 g. - add sets  
to white solid.

at 150° - sl. cloudy - now - still  
in pot. stir mixer out but re-KR. at ~ 40  $\mu$ .  
one boiling drop came over at ~ 150° - good drop  
not taken - not worth the gens - out, - wait out

Try verted DMTH-4.

vacuum too  
10% toluene in try. no.

176-X  
KACAMS

0.5 g into 18A 5 ml H<sub>2</sub>O - almost all in.  
I more in still not all. -> - no

stirring - a few 1000 emul. -  
40 ml H<sub>2</sub>O 500 wash - short. xrt 5  
immediately - filter - 18A with.  
oil dry -> 0.45 g after xrt  
white xrt 176-B mp 25+ 25°

Keep trace  
run 18A  
run mp.  
176-A  
mp 115-127

spectra  
page 173-170

(177)



1.18 g (way more?) DMHM-4  
 MW 143

his - calc. -  
 calc.

1.6 mL H<sub>2</sub>O in 30 mL MeOH

(6.2 mM)  
 (~30 mM)

add 0.5 mL CH<sub>2</sub>O 3%

(~6 mM)

add 1.5 g NaBH<sub>4</sub> exoMercuri  
 freeze  
 dry

(~30 mM)

— supplement —

(I miscalculated  
 checked MW's)

+ 3.2 g H<sub>2</sub>O<sub>2</sub>

+ 1.0 mL CH<sub>2</sub>O

another 12 ~ mM

+ 1.5 g NaBH<sub>4</sub>

another 30 mM

— supplement —

30 mL more MeOH

3 mL CH<sub>2</sub>O

4 mL H<sub>2</sub>O<sub>2</sub>

2 g NaBH<sub>4</sub> good exoMercuri. (BH<sub>4</sub><sup>-</sup> in sol. wet)

Let come to RT. + 300 mL H<sub>2</sub>O, NaOH 25% to pH blue  
 x 3 x 50 mL CH<sub>2</sub>O - flash → 1.53 clear, almost  
 colorless oil. to KR pot. - distill.

over 110° -

at 80° - first real hard vacuum grease in manifolds!

up to 140 (bp 115-25) and still a bit of fused  
 colorless in pot. - mostly over. white oil.

Wrong, wrong, wrong! IR shows it to  
 be a Sulf, but one that distills!



0.94g  
 ← (177A)

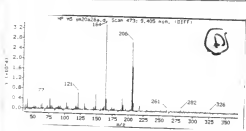
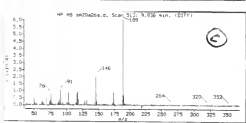
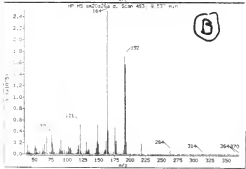
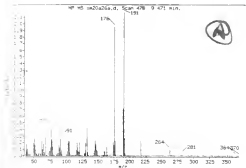
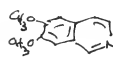
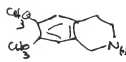
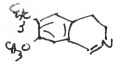
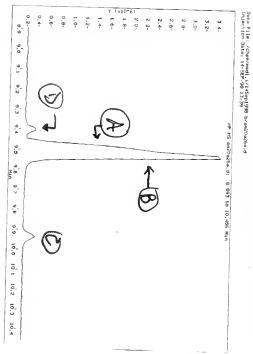
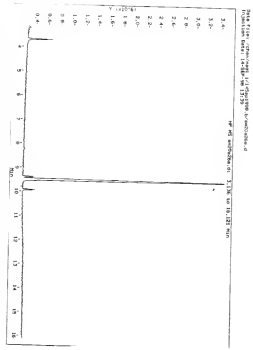
?



178

spectra from page 176 DMFH-4

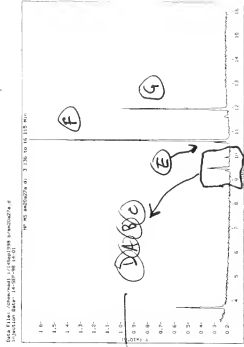
176A



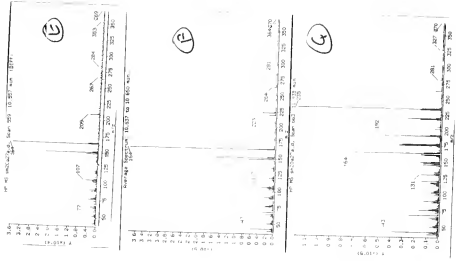
MW 207

Starcha film page 176  
 ve KR  
 (176X)

(179)



See they  
 = starch



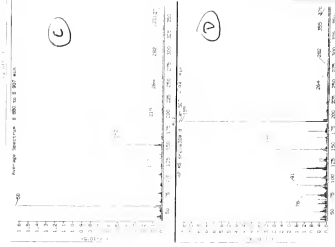
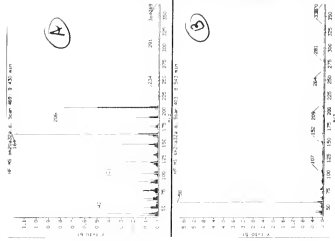
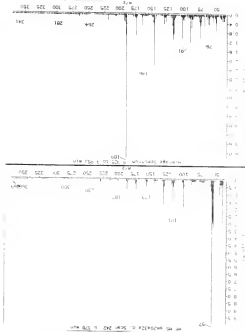
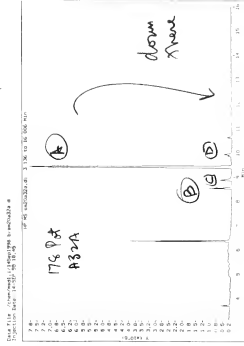
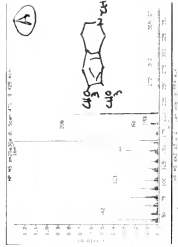
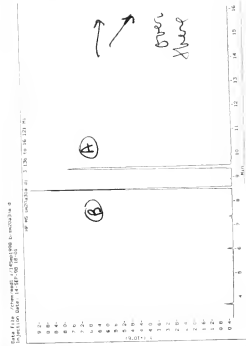
(176)

MW 107

150

Attempt to make the "phenolic" product 179.

177A C = 178.2 !!!



142

### mono acetylphen of 5-HT

→ 2.12 g serotonin.HCl

1.5g

1.5g

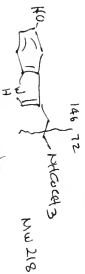
→ 1.52 ml pyridine - stirred slowly as it goes to a rather solid cake,

→ 1.04 ml Ac2O added evenly, drying time, over the surface of the cake. The reaction is almost a clear solution - and as it starts over, this begins to thicken. Time ~ 20 min. Add

40 ml H<sub>2</sub>O - homogeneous soln. extract 2x 40 ml CH<sub>2</sub>Cl<sub>2</sub> flash → 1.7g thick oil - 2 walls of pyridine - back into ether, - wash 2x 1.5N HCl - flash → 0.9g white oil almost no smell

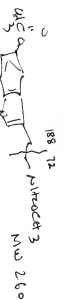
serotonin 176

N-monoacetyl



o-acetyl 155/30

o-acetyl



triacetyl

353/72 MW 302

take all 0.8g. Δ in rotary (50°) in a few mL conc HCl - add water - mix & chase. → 0.3g film.



(84)

# Diethylamine of Serotonin

2.12 g SAT.HCl

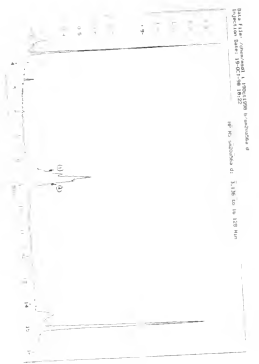
3.12 g. Aceto

found 6 to react? I don't remember

50 few drops - into sol.

1/5 wt slowly  
rest kinetic not in phase

what is 50 wt ether? (not wt sol, in oil) Serost  
sol ether anyway → 2.0 g viscous white oil.



Triethyl Serotonin.



(581)



1860  
 pink  
 p. 4416



SMO A61A

30g polymer  
 100ml H<sub>2</sub>O

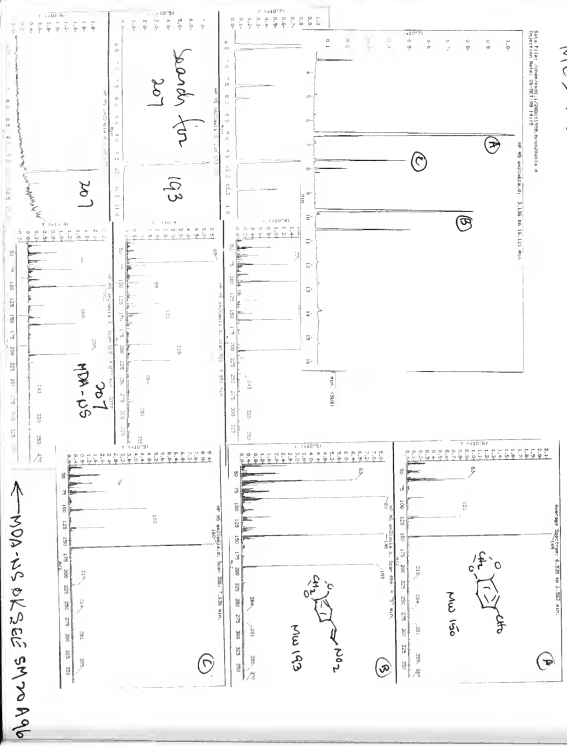
20ml water  
 to wd cyclohexanone (blank known!)

on 40 ~ 50 min - off at 16:00 - observed something → yellow solids

▽ RT. - rather heavy yellow solids.

Get fr. with 2 H<sub>2</sub>O - such a few dry -  
 crude wet weight 8.15g

MS stand on → some very light yellow solids. 1868 m/z  
 MS + water to turbid - stand → 3.7g wet 1868 m/z  
 See page 189



← MDA-MS DK 2.615 SM 70 A 96



4.8 g Al foil (n. 1 foot - 1" squares) +  
 0.12 g HgCl<sub>2</sub> in 150 ml H<sub>2</sub>O - last amalgamate -  
 already ready in 30 min  
 decant off H<sub>2</sub>O - wash 2 x 200 we H<sub>2</sub>O  
 1 x 200 we IPA.

add 100 mL IPA (RT)  
 10 we H<sub>2</sub>O

add 2g wet NS page 186 in 20 ml hot IPA.  
 stir - stand on - AM - gray sludge

filter - wash 150 we H<sub>2</sub>O  
 150 we H<sub>2</sub>O  
 20 we IPA  
 100 we H<sub>2</sub>O

small white solid - loss - not  
 soluble. un pale yellow

~~strip~~ add 200 ml + ~5 ml conc H<sub>2</sub>O.  
 x H<sub>2</sub>O 1 x 75 we CH<sub>2</sub>Cl<sub>2</sub> - add yellow into org -  
 strip.

Make basic c 50% then 75% NaOH  
 strip c 75 we CH<sub>2</sub>Cl<sub>2</sub> (no color).  
 strip - SMA64A

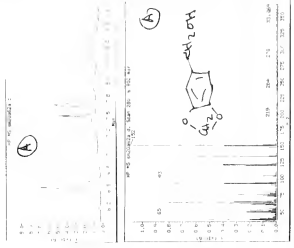
0.52 g sharp  
 solid  
 (H<sub>2</sub>M?)  
 vial yellow ore.  
 SMA62A

0.40 g viscous  
 white oil.

see  
 next  
 page

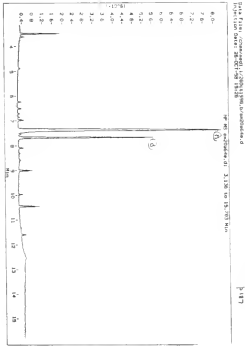
neutral

this is pretty  
 good time to start  
 to page 195



1.85

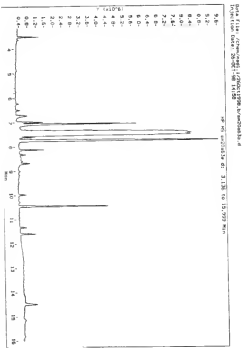
Benz fraction from Ad (lit) red. of NS  
(mass 187)  
SM 180 Ac4A.



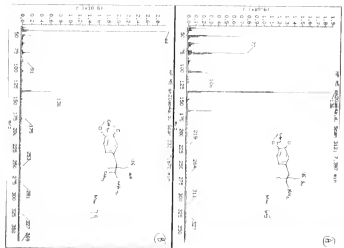
Ac4A

↓ diluted x 10

← overlaid



Ac4A



Ac4A



190  
190

Pyogran:



Start @ good 125 p 189

16.3 g good 125, 189A into 200ml gear, 10%  
w/1% in on steam bath we had -  
clean spec.

40 g of Ac form - 3" squares - amalgamate @

10 g Hg Cl<sub>2</sub> in 1.4 L H<sub>2</sub>O

1/2 hr - amalgamate taking - probably should have given  
more time of stirring, worst 2 x 1.4 L H<sub>2</sub>O, 1 x 1.4 L 18A -  
Acidic water - add:

1 liter 18A } - see RT, add:  
100 ml H<sub>2</sub>A<sub>2</sub> }

125 in hot water. add @ pretty good hard stirring -

1st group - immediate yellow dyeing (hot to cold? HCl to  
18A (?) add the rest over 5 minutes, stand 10').

AMN - clean the amalgam has been killed - allow RT -  
wash color still, yellow not to pink - just write strip

Follow all students - orange color water - wash RT @ 3 x 10 min

18A - combine - Reak → heavy yellow - red oil -  
xtds in it, into chels - xtd 3 x 100 ml dilute HCl - part -  
wash one @ chels - pale yellow

94% @ 25% base - xtd @  
3 x 50 ml chels - Reak → zinc  
of base fraction. To KBr,  
Serpige 198

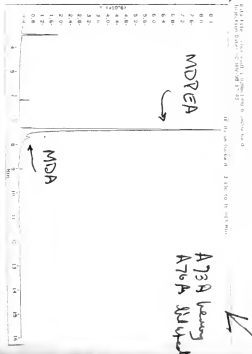
0.2 hr  
0.15 hr 80° wash

90° - 150° / 0.1 hr - 2.35 white oil  
Mauvelon

Picks up CO<sub>2</sub> like a shirt.

simulate to GCMS 191  
not to Gamma work - page 197

detectable MDA - 5400 in the Mando 2



Attempt 1:

11 to 170



2.3 g freshly distilled homophenylamine, run out of KR flask =

7.0 ml isobutyl formate - Immediate solids !! Must be a bit of carbonate - very quickly - rest of formate gives no more -  $\therefore$  not immediate Rx - no formate smell in formate. onto SB 5:30 - Off - 1.25 hrs

into 100 ml HD + H<sub>2</sub>O to pH Red. xlt 3 x 50 ml CH<sub>2</sub>Cl<sub>2</sub>

3.44 (small) 18 formate 40 yr 110° 40 yr 136°

flask  $\rightarrow$  7.44 g almost colorless oil - smells of 18 F to 14 - 0.05 mm immediately  $\frac{1}{2}$  white solids in pot. A

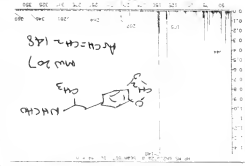
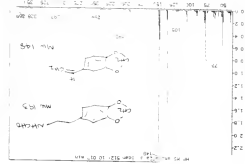
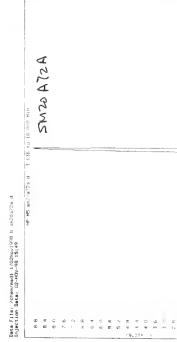
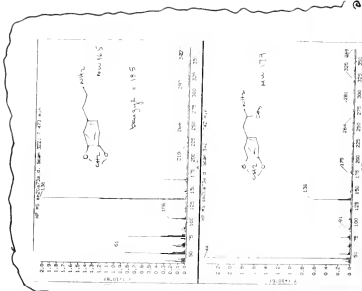
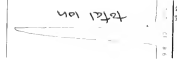
140-160° (higher) at 40 yr  $\rightarrow$  white oil that solidifies - 2.45

2.45

sample p(191)

2.40 isolated

2.30 on to D1A



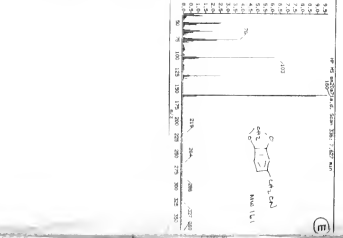
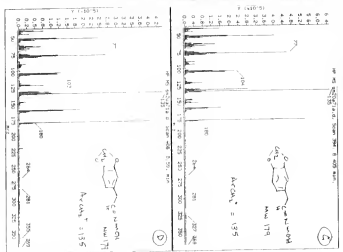
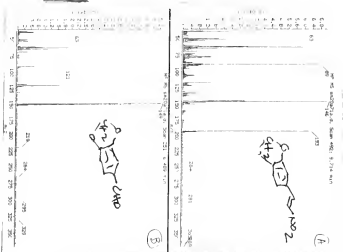
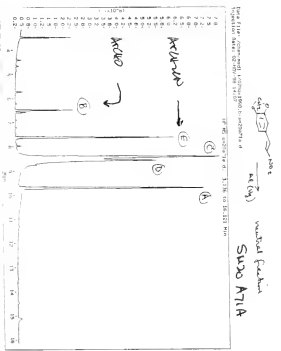
192

See page 190

residuals from c1ccc(cc1)C(=O)O + O=[N+]([O-])=O → CC(=O)N

taken to dryness  
10.36 g  
3 swabs cut 8.7 g

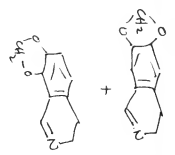
Same as  
homovergylamine  
crystal







104



2.20 g white formamide and 10 ml  $ROCl_2$  - onto 50.

$\Delta$  - 2.50  $\rightarrow$  fast - leave there a while 15 min off mid night.

to RT D20 - (cold room!). into 150 ml the (cold cell) = good showing - go to  $\sim 35^\circ$  - orange brown at - 2.9

film, an oil of insoluble. Ag. quite acid, darkens between the 2, 60 we check. 2.50 in 20 - yellow in solution.

All eq. to pH  $> 11$  = 2.2. 2.2 ml. add sets up solid 11.9. 5.7 has

also wd has to get stuff done - and almost into solution.

1.41 2.4 ml. 3.75 ml. - (when poured in sep funnel)

clean skin! flask!

20 ml

100% in.

no visible

to KR - starting to x half life

12-130  $\text{C}$   $\text{C}_6\text{H}_4\text{O}_2$ . white r.t. that

converting good to white x 100!

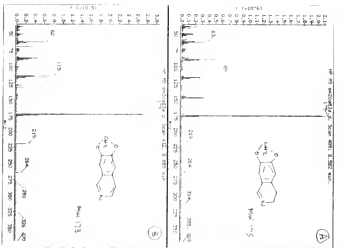
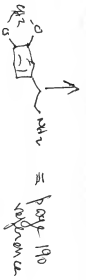
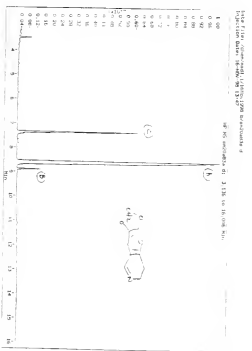
off - white still - viscous 1.2%  $\rightarrow$  (174)

neutral fraction

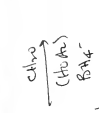
Park, 0.46 g viscous brown oil 194-A

to GCMS SM20 AS6A

slottis, 0.19 g, 2:1



From page 187.



195

0.40 g base - now broken - into  
[ 2 wt H<sub>2</sub>O + 8 wt MeOH ] to 100 wt stirrer add

2 wt 37% H<sub>2</sub>O -  
1.5 g Na B<sub>10</sub> - spatula - wine - over 20 min. - feeling over - add  
spurt y H<sub>2</sub>O - no more former - stir.

stir on R.T. - into ~ 100 wt H<sub>2</sub>O - + H<sub>2</sub>O → shing H<sub>2</sub> -  
wash 2 x 50 wt water

+ 25% MeOH  
X<sub>2</sub>H<sub>2</sub> < 100%

spin both times.  
0.25 g pale amber oil

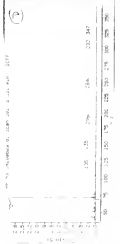
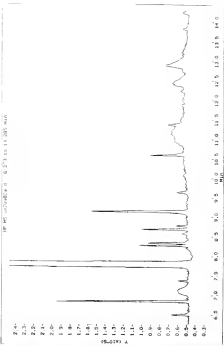
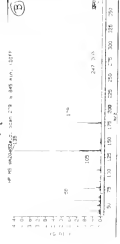
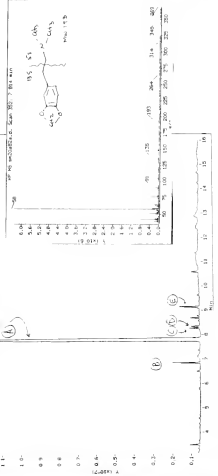
→ white.

Wt 1 wt MeOH + conc HCl  
2 wt water + 4 drops too much -  
stir 5 min + 2 wt ether - drain  
plates 1.75 wt white x<sub>2</sub>H<sub>2</sub>  
+ 1.5 wt ether

IR 0.02 μm 146-105 ~ 0.22 μm  
not quite white oil - No - it's white!

GCMS 195

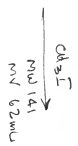
SM 20 AB2A



196



Mu 175



I-

0.6 g DHA (19A) into

1.0 ml MeOH -  $\Delta$   $\rightarrow$  still solid; +

0.3 ml  $\text{CH}_2\text{I}_2$  (250% xs) immediately yellow color -  $\Delta$  on Rot. Evap -

$\nabla$  - no ring -  $\Delta$  again  $\nabla$  RT  $\rightarrow$  fine white - immediate wine

and wine - add soon solid - add ~ 3-4 ml MeOH - solid disappears

but not all derivative. Stand. RT a few hours - yellow solid.

filter - wash each org.  $\nabla$  MeOH -

wet weight (yellow + hb) 0.55 g.

0.42 g dry

0.35 g on to

196

same 0.04 g

ref. selection

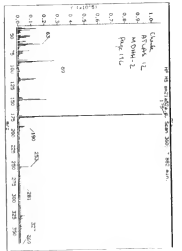
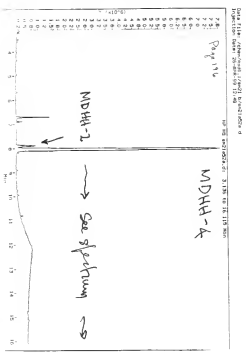
Calcat -  
on page  
222

from Mu 071

(19A too weak)

Mu 5  $\rightarrow$  yellow  
kinds - ends  
plate. 196A  
106 mg

Wash up 0.149 g (197A) - now turned solid of white. Absorptive id 3 we had 19A (1/2 not analyzed) add 12/14 sample entire  $\rightarrow$  white, off white. Filter without other - wash  $\nabla$  small amount 19A - then filter.  $\nabla$  small amount other  $\rightarrow$  0.43 g of off-white MDHT-4. 192 (197B) . SW21A52A





NaBH4  
HCl.



MDHH-4

197

0.6 g DH19 194 into  
~ 10 ml H<sub>2</sub>O - HCl dropwise → yellow solution (~10 drops) add

0.6 g NaBH<sub>4</sub>. immediate yellow loss - foam -  
then towards end - more of a froth. add

20 ml H<sub>2</sub>O - not much change - add  
Several cc 1 N HCl. some foaming - solution largely  
clears, and no more response to HCl.

strongly acid - stir 2 x 10 ml ether → vents

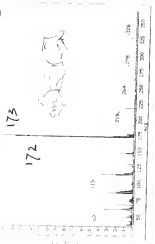
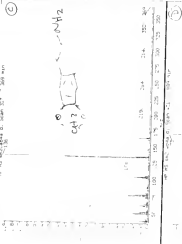
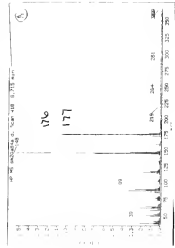
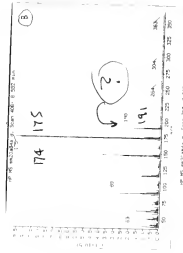
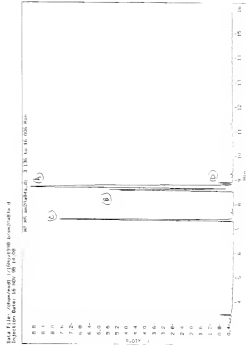
OH = 50% NaOH (get new pH paper!) - stir

3 x 40 ml ether - flask → 0.56 g white

crystalline solid. to IR.

oven 90° - 100° 60 min. → 0.49 g fused white xrb  
into 50 ml vial. (197) small sample for GC/MS 197A  
SM20 ABGA

50 μ 50°  
60 μ 90° oven



198



0.38 g yellow solids, 10 wt H<sub>2</sub>O - w/ completely soluble - add some 10 wt H<sub>2</sub>O - no better - add

0.39 g NBSH<sub>4</sub> (Rau et al. add more H<sub>2</sub>O - finally precip - when halogenate sample gets gone. + H<sub>2</sub>O + H<sub>2</sub>O - xH<sub>2</sub>O x 50 wt H<sub>2</sub>O

0H i 50, from 25% base  $\rightarrow$  white cloudy - hot 3 x 40  $\rightarrow$  we ch<sub>2</sub>2

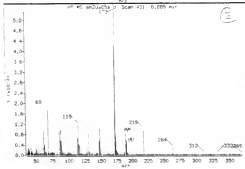
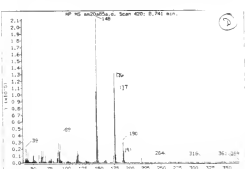
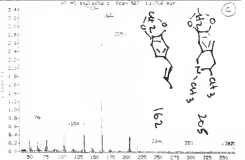
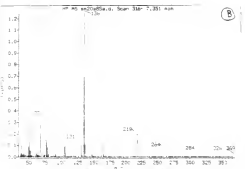
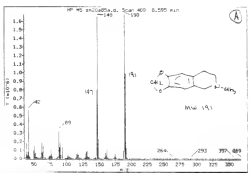
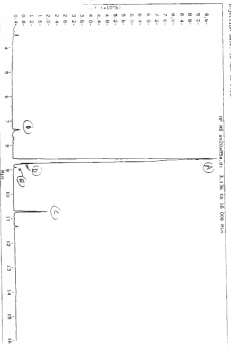
0.16 g brown  $\rightarrow$  white solid 95-95<sup>o</sup>/50<sup>o</sup> in R<sub>2</sub>S.

small sample  $\bar{c}$  dry ice  $\rightarrow$  xH<sub>2</sub>O - tubers.

small sample 2 dry ice  $\rightarrow$  xH<sub>2</sub>O - tubers.

small sample 0.08 g 108 SM20ASSA

0.16 g crude sample 95-95<sup>o</sup>/50<sup>o</sup> white solid 1 part T. still part of it. nothing sample.



Refact.

See 170  
171



199

10 g DMPEA, saturated i carbonate - the ancient bottle of Medich chem. stuff - all that would swim - carbonate now solid in bottle.

25 g isobutyl formate → cloudy - Δ 50 ± hr → brown soln. - add to 200 ml H<sub>2</sub>O, @ → pH 1 - extract 3 x 75 ml CH<sub>2</sub>Cl<sub>2</sub> - pool - wash c 5% NaOH - pool → ~~10.85 g~~ 10.85 g crude

KR 15-160°/120 ju. → 7.52 g vacuum oil pale amber. into 45 ml POC<sub>2</sub> - spill - nasty - into 50 ml (1.5 hr actually.) - off - cool oil. Into 400 ml cold water - emulsion of course - Δ c 1 hr - xrt c 2 x 50 ml CH<sub>2</sub>Cl<sub>2</sub> OH c 25% NaOH to pH 13 - Δ → solids, H<sub>2</sub>O to 1300 ml to dissolve - xrt in 1/2 batches c 3 x 50 ml CH<sub>2</sub>Cl<sub>2</sub> each - pool. wash c 1 hr - flush → 4.24 g vacuum brown oil. KR.

200 ju 130-140° 273 g. Save ~ 0.2 g to see how the oil dissolves colors. 250 g to page 200

this all from memory - lost the paper in the notes and weights. Flash of ether → a lot of material - 2-3g that tends to go solid. TLC a bit checking against reference sample of formamide. solvent 80/20 hexane/ether. Amide ~ 0.2 Rf. thin stuff only. Could the salt of DMHT-2 be extractable into ether? Partition ~~of~~ in OH:H<sub>2</sub>O & CH<sub>2</sub>Cl<sub>2</sub> - ether to dryness ~ 1.5 g? to KR. So, 50 vacuum br (N 200 ju?) and push to ~ 200°. Some 60 mg came over and the pot was left with a dark residue - too much - with some fluidity. OUT.

216

100



2.50g DMHH-2 - dissolved in  
 5 mL MeOH - add  
 1.2 mL Et<sub>3</sub>I.

A on edge of SB - sudden &  
 yellow starts - off - let extend to RT - several hours.  
 of orange starts pushing up through the yellow! Stir  
 it all afternoon - let stand. working the stirring more  
 does not remove the yeast.

Filter (after adding a couple of mL of  
 acetone. → gold colored starts - wash 2 x 2 mL  
 acetone - suck dry - air dry.  
 → 4.29 g wet dry  
 4.20 g dry





A.R.

book

A.15

in the

series

A.15

with

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## THE CONTROLLED SUBSTANCES ACT

### A RESOURCE MANUAL OF THE CURRENT STATUS OF THE FEDERAL DRUG LAWS

Including:

A 215 page chapter that lists every formerly and currently scheduled drug, the authority for its inclusion in the law, its exact chemical name, its Chemical Abstracts Identification number, and all known synonyms for it.

A history of the development of the currently employed Federal law, and the many amendments, changes and regulations that have brought it into its present form.

A complete listing of all chemical structures of the controlled substances, arranged by structural families. Some 46 pages of Drug Code Numbers assigned to drugs, solvents, vitamins, food stuffs, and even things unrecognized.

All Federal Register Citations that have been used to justify changes in law and regulation.

A complete empirical formula index of all compounds mentioned in the current statutes.

Four appendices, detailing the original law (1970) and the present form it has taken (1987), the wording of the Emergency Scheduling Act, the Analogue Enforcement Act, and the currently available and



100%

100%

100%

100%

202

accumulate  soon

1891 B

5.73 g crude -

crude M<sup>1</sup> of 1

Δ 98 = 75 mg ~~1820~~

AcOH

Δ SB 1/2 hr.

fill to just - neurons

some w/acid's

□ OP RT

3.34 g

25 g polymeral

75 mg cis 202

2 1/2 g NHTCKT

Δ SB 2+ hrs.

2 x 1 hr

an Rng.

- into beach - ▽ add less

fill to - wash = a little more

203

Arguing into the "Pharmaparc" available in Pharmacy  
LH pill.

S-NaoDMT piece

S-NaoDMT piece

LH<sup>2</sup> pill.  
dirts yellow

235mg

lost the quan-  
spray or  
from

white left?

30mg white solid

to Karmelini - 146  
Sigma lot 1231437.7  
95%

to Hornum - 144

~~144~~ lot AJ01  
H0002





Handwritten notes in Chinese characters.

3.36 g NINE QUAT DMMM-2 + I - into 100 ml H<sub>2</sub>O - add

just > 10 ml H<sub>2</sub>O HCl (not ~~not~~ under quantity) and add 1 g NaBH<sub>4</sub> a bit at a time.

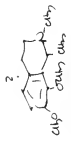
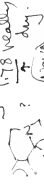
quart in water cloudy. add ~ 12 ml H<sub>2</sub>O - still cloudy & no color at all! pH red. add 100 mg pKa's of NaBH<sub>4</sub> - in acetonitrile ~ 1/3 of BH<sub>4</sub> added now green, spirit x4 N H<sub>2</sub>O → red - add white 100 mg BH<sub>4</sub> + spirit the always letting the pH dictate which next. 1/2 way through - totally clear orange - 1/2 way there of water - add rest of NaBH<sub>4</sub> + acid to lower the pH red. - xH<sub>2</sub>O 2 x 50 ml chloro (let evaporate) BH<sub>4</sub> to blue ± 5 ml NaOH. xH<sub>2</sub>O 2 x 15 ml chloro - no emulsion - clean - pink - flesh → 1.56 g pure yellow oil.

Carbazine  
Methylindole  
#1896  
viscous oil  
H<sub>2</sub>O 14.50 g  
dist. 50%  
imp 20.7  
amb. 22.1  
and add

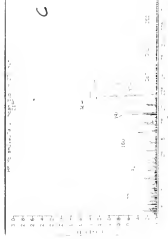
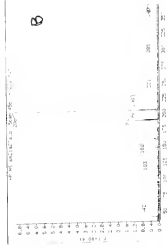
Soln.  
100 ml  
115-125 ml  
1250 drops  
White turbidite  
clean part.

1.56 g (205A) same small sample GCMS. See page 204

best w/5 beaker ± 8 ml IPA + some H<sub>2</sub>O 21 drops not enough. 24 more than enough - then - wait x 1/2 hour start to form - in 2 min. very thick - dilute ± 5 ml acetone - filter - wash ± acetone - acetone (spring 5)



178 really dry.



DMMM-4  
MIX  
H<sub>2</sub>O 219  
MIX

C

B

1.04 dmw  
100



206



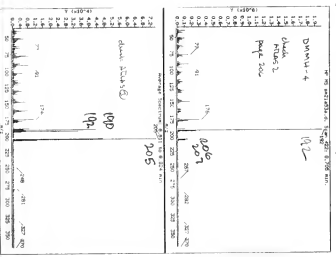
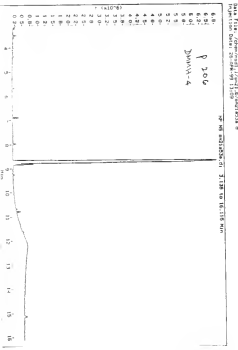
205

2.6g DMMH-2 (page 165 C) was  $\text{Zr} \sim 12 \text{ cm}$  into  
 100 mL H<sub>2</sub>O (IT IS WATER SOLUBLE!!!), acidify with  
 1 M HCl ( $\sim 12 \text{ mL}$ )  $\rightarrow$  red pH buffer)  
 1 g NaOH. add a bit of lime (good stirring) + N<sub>2</sub> try  
 as needed to keep the solution acidic. No exoN<sub>2</sub>.  
 work  $\bar{c}$  50 mL CH<sub>2</sub>Cl<sub>2</sub>, boil  $\bar{c}$  5% NaOH - extract 2x  
 so we collect - stay on R.E.  $\rightarrow$  "1.17 g" crude - normally  
 I would have cleaned, see below - KR 110-120/140g  $\rightarrow$   
 2.13g free base - white oil - on start of recovery

Purification between cycles

and also NaOH - 3 hrs -  
 2.24 white oil - loss of brown 8 mL IPA - 30-32 drops H<sub>2</sub>O  $\rightarrow$   
 pH red. no xtho - add ether to turbid - no xtho - repeat 5x out  
 eventually - seed - some takes - I feel I am picking too thick?  
 (hydrolysis) were ether  $\rightarrow$  wash of solids - filter - wash  $\bar{c}$  ether -  
 an dry  $\rightarrow$  2.70 g albertan white solids - also stain to card, 10 ths?  
 206B active solids!! was the substrate in water!

picking up CO<sub>2</sub>? GCMS came 131A (SM2)  
 Saw a bit of free alkene 133A  
 base solids - contains 202 (206A)



207



1.5 g DMHH-2 + HCl (from p172A) - into 50 ml H<sub>2</sub>O

0.5 g NaOH + 1N HCl

with good stirring add hydroxide a bit at a time, keeping the Rx mix acidic  $\bar{c}$  5 drops of H<sub>2</sub>O. Work  $\bar{c}$  18:30 w/ EtOH, extract  $\bar{c}$  2 x 40 ml EtOH. Wash base  $\bar{c}$  5% NaOH, extract  $\bar{c}$  2 x 40 ml EtOH - flask on R. coat  $\bar{c}$  residue that crystallizes - Distill at KR 110-130 $\bar{c}$  100 u - all sublimed over as white solids. Wt (in flask) 1.12 g

Scrape out. 1.00 scraped out - not sol. IPA, Sol. H<sub>2</sub>O - Et. must be the carbonate - into 2 x 100 ml H<sub>2</sub>O snow soluble - add 5% base  $\bar{c}$  H<sub>2</sub>O blue - xht 3 x 40 ml EtOH - flask  $\bar{c}$

0.86 g oil that sets to white xtal - dissolve in 2 ml hot IPA into beaker - remove  $\bar{c}$  2 ml IPA - clean soln - add 10 too little 13 too much HCl (solids at 3!) + a bit of ether - stand - filter - wash  $\bar{c}$  ether in dry  $\bar{c}$  beautiful white crystals of DMHH-4 + HCl 0.92 (207A)



11/11

11/11

11/11

11/11

Citricolium  
6, 101 Neo Neo

6,7 MDD

61,14  
207A  
MUD 2004  
193



1,7 D  
MUD 2004  
171

193  
192  
164  
100%  
60%  
55%

~~61,14~~  
H, M  
201  
MUD 2004  
201



223  
MUD 2004  
191

207  
206  
164  
50%  
15%  
100%

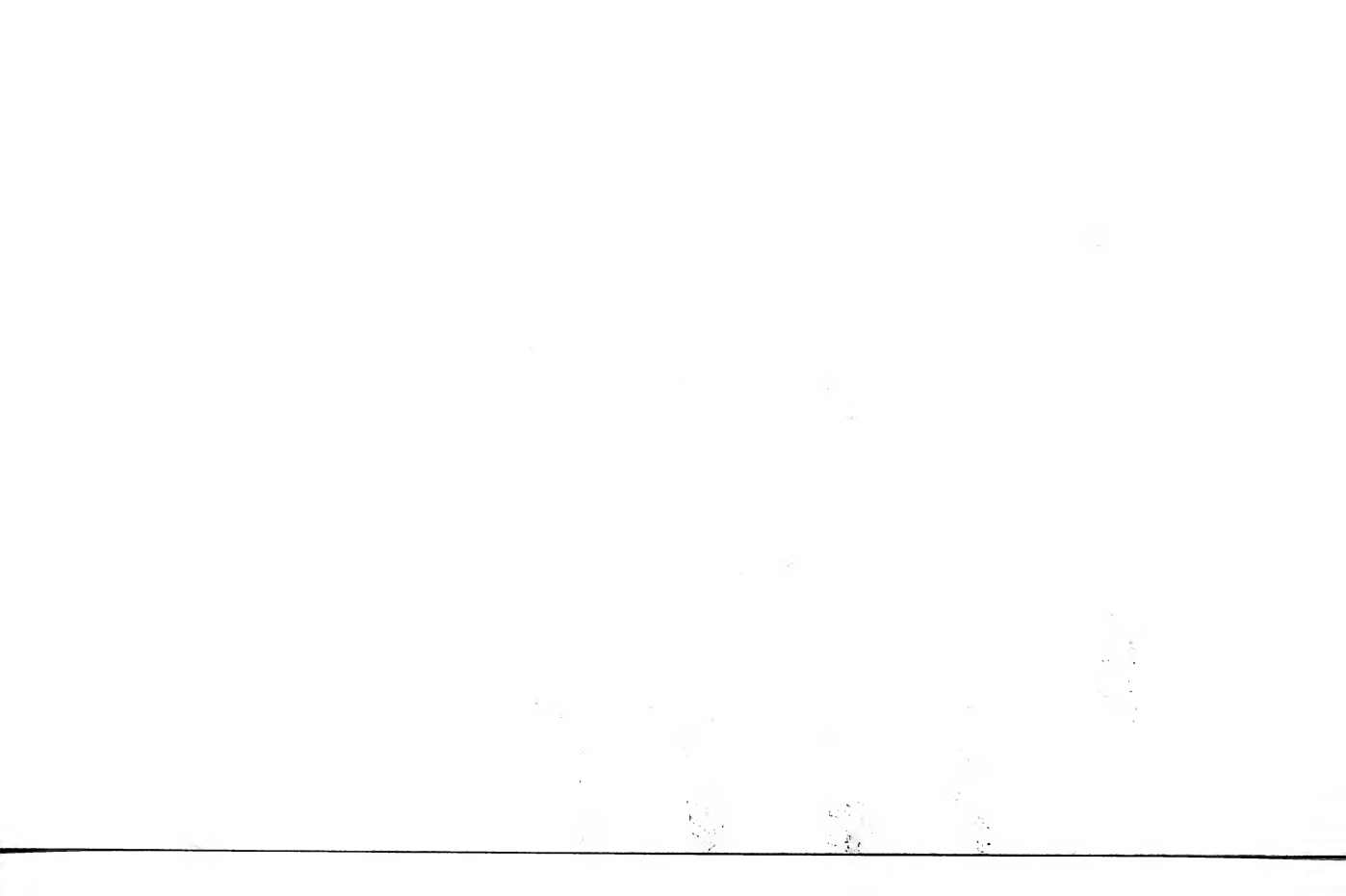
~~61,14~~  
Melt  
206 B  
MUD 2004  
207

218 A  
MUD 2004  
111

2

Mel Nu  
200  
MUD 2004  
221

219  
MUD 2004  
205





208



8.2 g Al<sup>1</sup> Squares (n2 ft foil) - add solution of  
 2.2 g HCl<sub>2</sub> in 300 ml. H<sub>2</sub>O, at RT ~ 1/2 hr → heavy spray, bright  
 spots - 1h ft. - decant H<sub>2</sub>O, wash  
 300 ml H<sub>2</sub>O - drain, ~~wash~~ wash

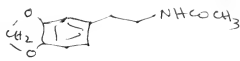
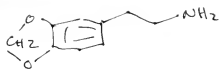
100 ml IPA - drain - add  
 + [100 ml IPA] - add - over 1 minute;  
 3.34 g (see page 193) NS. in 40 ml MeOH  
 40 ml IPA Δ 50 - mix in solution

4th round extraction - 2 ext. H<sub>2</sub>O, 20 ml each, adjust w/ HCl.  
 add over the white res. in PH paper. Try extracting 2 cubes  
 (discard top phase!!) separate in bottom jacket - GCMS on  
 these washes see page 193 adjust to correct square volume H<sub>2</sub>O  
 lets water - discard on bottom - extract again → recovery  
 org. waste = 25% MeOH - x 4 x 3 x 75 ml cubes (done in red waste  
 these batches - discard a/c and appropriate - fresh - flash)

on vft. sample → 1.5 g known oil  
 90° 0.04  
 white oil  
 dissolve at 40° 90-100° white  
 oil 0.95 g pot. lets ~~even~~  
 up to 140° - flash  
 5 ml GCMS (208B) → OOT  
 2.32 g  
 Same wash  
 under the  
 amine  
 see p 193  
 208A

Synthesized → white solids.  
 Can Market (208)

run # 1 1.54 white oil used for octanoids 215  
 run # 2 1.46  
 run # 3 1.57 g  
 3.33 g  
 400 90-100° → white oil 2.54 g all to Formamide page 220  
 50 ml per wash  
 4 0.712 pot visible  
 wash vft. at  
 140° C.  
 conditions: (208D)  
 See page 261



(209)

0.95 g white (208C) into 2.5 ml pyridine - add  
 1.0 g Ac<sub>2</sub>O. Δ S.B. 1 hr. (tried to strip it, no go) - add to  
 30 ml H<sub>2</sub>O - add gls of NH<sub>4</sub>OH - stir - oil goes to  
 Solids. color pale tan, but that may be some of **Jill's**  
 sink out of the snorkel of the rotary evaporator - look  
 for that awful p-nitroanisole  
 filter - wash w/ H<sub>2</sub>O - kinda dry → 0.92 g off-white solids  
 w/ H<sub>2</sub>O - stir w/ ether - wash acid (1M HCl) 1/2 flash  
 → 0.19 g  
 distl at KB 165-180° at 0.05 mm. white oil over.  
 Say 165-175°! short. stals.  
 0.93 white crystals (209)

0.150 g





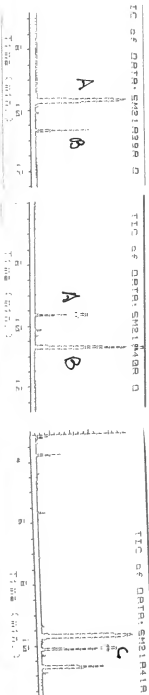
no steamover & Rn. n. 2 PM darkened - 2 phases? sets to xthls  
 of (11 PM = 19 hrs. very dark - 2 years? K. H. 40 for the 200 wt work -  
~~sets to~~ in strongly basic - xth is 3 x 50 wt chcl<sub>2</sub> - all set in the org.  
 xth is Dil. HCl 3 x 50 wt (color strong in chcl<sub>2</sub>) - part of  
 work is in 50 wt chcl<sub>2</sub>, water eq. waste (color) is 25% Na Pt.  
 cloudy. xth 3 x 50 waste - amber colored - fresh. At 80°/0.05 gm -  
 all the xth is gone → black heavy oil in part. At 90° -  
 notice some colorless oil in the residue - some it, there at  
 90°? - type probably - lots up for Swartz - no small - want for  
 0.5 gm  
 or use Pt one (1.2 gm) and more. New reaction.  
 (210B) 100% CEMS

140-150° over + 0.76 g pale amber - part black! out with 4 wt IPA  
 110° standing?  
 distillates slowly - OK + 7 hr little 10 hr wash, darkening  
 came HCl. Spent xthls + a little 5470, nice white  
 xthls - wash 2 ethan/acetone. see dry → 0.52 g xthls,  
 219g yield! ygh  
 (210C)

(210 C) #39

(210 D) #40

ML's 2<sup>nd</sup> crop  
 0.13 g white xthls (210D)  
 (210B) #4





REPORT FILE

SM-21

REV. 1-2 12-MAY-68

Sample name file name:

~~XXXXXXXXXX~~

22 MAR 69 4:55 PM

Index Sample Identification

- 1 OVEN 4 FROG C06
- 2 OVEN#4 PAGE 247
- 3 #21 DILUTED CSW
- 4 #12 DILUTED SW
- 5 CARBONION MICROLYTIC "MICROLYTIC" 1000/ML
- 6 SPECTRUM ETHYNO-DIOLYL METHACRYLATE 1000/ML
- 7 CONTINING 1000/ML
- 8 #101 1. BPE/EPH/PH MEMPH
- 9 #101 OVEN 4 WITH CONTINITY
- 10 #101 OVEN#4 WITH HIGH PROBABILITY
- 11 OVEN#2 EXTREPT OF DISCREPANT RADIUS OF WHITE SEED PRO
- 12 #101 #11
- 13 1A POU
- 14 SEP-BAT
- 15 SUSPECT F-REP-OHT
- 16 SUSPECT TROV#1-INC
- 17 #101#1-SWELL-INC
- 18 W-1#-100 PAR-02, F1089
- 19 #101#1#100-100
- 20 #101#1#100-100
- 21 SUSPECT #101#1#100-100 (SEE 16)
- 22 #101#1#100-100
- 23 #101#1#100-100
- 24 #101#1#100-100
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- 96 #101#1#100-100
- 97 #101#1#100-100
- 98 #101#1#100-100
- 99 #101#1#100-100
- 100 #101#1#100-100

95 page 425 T. D.  
96 page 39 as in T. E. D.  
98 page 39 (same) D. T. E.  
99 page 41 (same) F. T. E. book

Sample table file name: 2800000000

2 Nov 98 3:40 pm

## Index: Sample Identification

1 ACETONE-D6  
 2 ACETONE-D4  
 3 ACETONE-D4  
 4 ACETONE-D6  
 5 ACETONE-D8  
 6 METHANOL-D4  
 7 METHANOL-D4  
 8 METHANOL-D6  
 9 METHANOL-D8  
 10 METHANOL-D10  
 11 DIETHYLENE GLYCOL-D4  
 12 DIETHYLENE GLYCOL-D6  
 13 DIETHYLENE GLYCOL-D8  
 14 DIETHYLENE GLYCOL-D10  
 15 DIETHYLENE GLYCOL-D12  
 16 DIETHYLENE GLYCOL-D14  
 17 DIETHYLENE GLYCOL-D16  
 18 DIETHYLENE GLYCOL-D18  
 19 DIETHYLENE GLYCOL-D20  
 20 1,4-DICHLOROBENZENE-D2  
 21 H<sub>2</sub>O  
 22 H<sub>2</sub>O  
 23 H<sub>2</sub>O  
 24 H<sub>2</sub>O  
 25 DMSO-D6  
 26 DMSO-D6  
 27 DMSO-D6  
 28 DMSO-D6  
 29 DMSO-D6  
 30 DMSO-D6  
 31 DMSO-D6  
 32 XYLIS FROM PLOT OF M31 178-PORT  
 33 TOLUENE-D6  
 34 TOLUENE-D8  
 35 TOLUENE-D10  
 36 TOLUENE-D12  
 37 TOLUENE-D14  
 38 TOLUENE-D16  
 39 TOLUENE-D18  
 40 TOLUENE-D20  
 41 NITROBENZENE-D6  
 42 NITROBENZENE-D8  
 43 NITROBENZENE-D10  
 44 NITROBENZENE-D12  
 45 NITROBENZENE-D14  
 46 NITROBENZENE-D16  
 47 NITROBENZENE-D18  
 48 NITROBENZENE-D20  
 49 NITROBENZENE-D22  
 50 NITROBENZENE-D24



SAMPLE TABLE

2419

Rev. 3-2 12-14g-89

Sample table file name: INDEXINDEX

7 Aug 78 6:51 pm

Index Sample Identification

- 1 YELLOW SOLID
- 2 YELLOW SOLID
- 3 RED SOLID
- 4 RED SOLID
- 5 RED SOLID
- 6 RED SOLID
- 7 RED SOLID
- 8 RED SOLID
- 9 RED SOLID
- 10 RED SOLID
- 11 RED SOLID
- 12 RED SOLID
- 13 RED SOLID
- 14 RED SOLID
- 15 RED SOLID

5

- 16 Name Info: DIMETHYLMETHOXYPPA AND CHCl3 AND BASE, RT A FEW MINUTES
- 17 Name Info: 4-METHOXYPHENETHYLAMINE REFERENCE
- 18 Name Info: 4-METHOXYPHENETHYLAMINE AND CH2O AND NASH4 AND HOAC IN H2O
- 19 Name Info: REAL RUN OF 18
- 20 Name Info: EXTRACT PACH 25% BASE DICHLO
- 21 Name Info: 20 ACID BASE -- ENTIRE SAMPLE
- 22 Name Info: 146A MD-MH
- 23 Name Info: 10,000 CONC RAZA D'ORO
- 24 Name Info: CONCENTRATE OF SR
- 25 Name Info: D2-NICOTINE FROM TARTRATE -- FOR CLINICAL TRIALS
- 26 Name Info: D4-COTININE DISTILLED BASE -- FOR CLINICAL TRIALS
- 27 Name Info: (MS 7)
- 28 Name Info: D4-COTININE, REMAKING OF THE PERCHLORATE -- BEFORE REXTAL
- 29 Name Info: D4-COTININE, REMAK PERCHLORATE SALT -- AFTER REXTAL
- 30 Name Info: D4-COTININE, SAMPLE 29, MUCH DILUTED
- 31 Name Info: D4-COTININE XTALS FROM ACETONITRILE
- 32 Name Info: D4-COTININE XTALS #31 DILUTED 10X
- 33 Name Info: D4-COTININE XTALS #32 DILUTED 10X
- 34 Name Info: 90/10 BLANK
- 35 Name Info: 5-HT AND ACETIC ANHYDRIDE
- 36 Name Info: 35 WASHED WITH AQ. BICARB.
- 37 Name Info: 90/10 BLANK FOR 3/2/98
- 38 Name Info: P. HARMALA SAMPLE B EXTRACT
- 39 Name Info: #39 SAMPLE B DILUTED SEVERAL TIMES
- 40 Name Info: P. HARMALA SAMPLE A EXTRACT
- 41 Name Info: #41 SAMPLE A DILUTED SEVERAL TIMES
- 42 Name Info: XENNET FROM BARK OF VIROBA THEODORA
- 43 Name Info: 90/10 EXTRACT OF SERUM TUBE #1
- 44 Name Info: 43 AGAIN -- METHANOL AND BASE
- 45 Name Info: 44 DILUTED MUCHO
- 46 Name Info: 90/10 EXTRACT OF SERUM TUBE 2
- 47 Name Info: D-0-MECAMYLAMINE
- 48 Name Info: D-3-MECAMYLAMINE
- 49 Name Info: D-3-MECAMYLAMINE
- 50 Name Info: O-ETHYL-5-HT

51 Name Info: O-ETHYL-5-HT DMF 5 HR  
52 Name Info: O-ETHYL-5-HT DMF 3 HR ACID BASE  
53 Name Info: O-ETHYL-5-HT MEONA P 157  
54 Name Info: 5-ETHO-DET FROM P 157 AFTER ACETIC ANHYDRIDE  
55 Name Info: NEUTRALS FROM P 157 AFTER ACETIC ANHYDRIDE  
56 Name Info: STEAM DISTILLATE OF T. GRANDIFLORUS  
57 Name Info: PU X-87 3,7,5-DIBROMOTRIDEUTEROPYRIDINE (LOOK FOR Br, Cl)  
58 Name Info: PU XII-83 3,7,5-DIBROMOTRIDEUTEROPYRIDINE DIRTY (L.F.Bz, Cl)  
59 Name Info: 57 A LOT MORE DILUTE  
60 Name Info: 0.43 G (0.34 G?) SAMPLE FROM KR  
61 100% CITRATE DC 15  
62 100% CITRATE -- BILCHENWÄTTE  
63 AC BRCE  
64 AC BRCE AND BWA  
65 100% TIC LOT 1001  
66 100% TIC LOT 1001  
67 100% TIC LOT 1001  
68 100% TIC LOT 1001  
69 100% TIC LOT 1001  
70 100% TIC LOT 1001  
71 100% TIC LOT 1001  
72 100% TIC LOT 1001



Sample table file name: sgms01st05

13 Oct 97 7:46 pm

Index	Sample Identification
1	METHANOL OR MORGIC BE
2	DIETHYLENEGLYCOL
3	GLYSEROLNE DISTILLED
4	2-NITROGEN SOLIDINE TRING
5	D-CYCLOHEXANONE 1 KG/ 4 MS
6	POLYURETHANE 24H
7	2-CYANOPURIDINE
8	DIETHYLENEGLYCOL
9	DIETHYLENEGLYCOL
10	DIETHYLENEGLYCOL 5-HA
11	MIXED FOR METHANOL 07/30/97
12	100%ETHEL VLN 1807/97/MS1
13	100%
14	HE-PROLV-N-NDH8 NEM REAGENT
15	HE-PROLV-N-NDH8 OLD REAGENT
16	110 D TROVLE NEM8 OLD REAGENT RECONCT 07/20 07/97
17	HE-PROLV-N-NDH8 OLD REAGENT REBOT 1 00/7/97
18	HE-PROLV-N-NDH8 OLD REAGENT REBOT 100 00/7/97
19	HE-PROLV-N-NDH8 OLD REAGENT REBOT 10 00/7/97
20	110 D TROVLE NEM8 OLD REAGENT REBOT 100 00/7/97
21	HE-PROLV-N-NDH8 OLD REAGENT REBOT 10 00/7/97
22	HE-PROLV-N-NDH8 OLD REAGENT REBOT 10 00/7/97
23	HE-PROLV-N-NDH8 1 00/7/97, NEM803 + ET3H
24	HE-PROLV-N-NDH8 1 00/7/97, NEM803 + ET3H
25	HE-PROLV-N-NDH8 1 00/7/97, NEM803 + DMPE
26	HE-PROLV-N-NDH8 1 00/7/97, NEM803 + DMPE
27	HE-PROLV-N-NDH8 1 00/7/97, ET3H
28	HE-PROLV-N-NDH8 1 00/7/97, ET3H
29	110 D TROVLE NEM8 1 00/7/97, ET3H
30	110 D TROVLE NEM8 1 00/7/97, DMPE
31	1000 + PYRROLIDINE
32	1000 + PYRROLIDINE
33	1000 + PYRROLIDINE
34	1000 + PYRROLIDINE
35	1000 + PYRROLIDINE
36	1000 + PYRROLIDINE
37	1000 + PYRROLIDINE
38	1000 + PYRROLIDINE
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40	1000 + PYRROLIDINE
41	1000 + PYRROLIDINE
42	1000 + PYRROLIDINE
43	1000 + PYRROLIDINE
44	1000 + PYRROLIDINE
45	1000 + PYRROLIDINE
46	1000 + PYRROLIDINE
47	1000 + PYRROLIDINE
48	1000 + PYRROLIDINE
49	1000 + PYRROLIDINE
50	1000 + PYRROLIDINE

- 51 PYRIDINE/S-NH<sub>2</sub>, 24H  
 52 3-CYANOPYRIDINE  
 53 NICOTINAMIDE, SYNTHETIC  
 54 NICOTINAMIDE, 1,2-DIC  
 55 NICOTINAMIDE \* SUGAR, 51H  
 56 METHANOL  
 57 METABOLIC NICOTINAMIDE FROM KOL  
 58 METABOLIC NICOTINAMIDE FROM NICOTINIC AC  
 59 NICOTININE STANDARD  
 60 NORNICOTINE-DE SUBSTRATE FROM TLC, KOL  
 61 METABOLIC NICOTINAMIDE FROM KOL  
 62 NICOTINAMIDE STANDARD  
 63 DIBENZOARGANOLSOLIDINE  
 64 SOLSOLIDINE  
 65 DICICL ESTER OF P.P.  
 66 METHYL NICOTINATE  
 67 ON COLUMN METYLATION OF N.A.  
 68 SOLSOLIDINE VIA ACETIC ACID AND NABA  
 69 180C/TOL/7,12-D-C  
 70 NINE-AR-S  
 71 NORNICOTINE-DE  
 72 P-EPOXYE/OL/KOL VIA NAB RYN  
 73 P-EPOXYE/OL/KOL VIA NAB RYN 51H, 100, 150, 180, 190  
 74 P-EPOXYE/OL/KOL VIA NAB RYN  
 75 NORNIC-DE SYNTH  
 76 METYLATION OF ACIDIC CC  
 77 DIBENZOARGANOLSOLIDINE  
 78 SOLSOLIDINE DISTILLED  
 79 N-METHANOLSOLIDINE CRUDE  
 80 NICOTINIC ACID ETHYLE  
 81 AR BASE AR-S-PHINE  
 82 TIMING -- P.D.  
 83 TIMING -- N-METHANOLSOLIDINE  
 84 AR ONE #1450  
 85 AR-ENE #1458  
 86 AR-ENE #1468  
 87 ZMITTEPION FRACTION 137C UNDEPARTIZED  
 88 Z FRACTION 137C WITH ACETIC ANHYDRIDE/PICOLINE  
 89 SALLES FROM ACETIC ANHYDRIDE/PICOLINE  
 90 AR-S tert-PHINE  
 91 TOLMETHEPION : HEDIA  
 92 MIAKON FOR NABT RYN  
 93 AR-S-HOC  
 94 AR-SHOC ?  
 95 AR-SOC HOC ?  
 96 HNS 6,7-EPIETHARY-1,2-DIMETHYL-TOLU  
 97 CRUDE HARNOLINE  
 98 96, 145? DILUTED 10X  
 99 1, 180 C/DH NICOTINIC AND PICOLINE ACETIC ACIDS, 50:50  
 100

che  
 Page 145A

6,7-Dimethylidene

6,7-Methylidene

	HH	HM	MH	MM	HH	HM	MH	MM
	chem 171 194 172	chem 200	chem 140 169	chem 204	chem 194	chem 196	chem 144 145 216	chem
MS	178 173		142 (?) 144 (?)		194 196 RTB	<del>196</del> ?	150	
ATLAS	(3) (23)		(3)		(12)		(6)	
	chem 207 176	chem 177 201	chem 206	chem 143 205	chem 197	chem 198	chem 146	chem
MS	178	181 201	142 162	162 204	?	198	151	
ATLAS	(24)	<del>738</del> ATLAS 27	conclit ATLAS (2) (17)	(3)	(13)	(14)	(9)	(25)
ATLAS	(19)	(8)			(13)	(14)	(9)	(25)
MS	173 178				194 ATLAS (11)		ATLAS (8) (20)	

MS

MS

MS

51 mm  
 4 mm  
 3 mm  
 2 mm  
 1 mm  
 0.5 mm  
 0.2 mm  
 0.1 mm  
 0.05 mm  
 0.02 mm  
 0.01 mm

Chemical Final Recipes:

6,7 Neo I Me	6,7 Neo I-H	6,7 MDO I Me	6,7 MDO I H
DMPEA Acetamide (168) (141) (138)	DMHH-2 (170) DMHH-4 (176) DMHM-2 (200) DMHM-4 (201)	MDPEA-acetate (209) (145) (215)	MDPEA NS (183) (186) (208) MDPEA (187) (190) (171) (DM-MDPEA) (195) (207) <del>MDPEA</del> (MDPEA PEH) MDPEA Forward (171) 191 220
DMMM-2 (140)	DMHH-2 (172) -173, 174, 175, 190, DMHH-4 (176) DMHM-2 (200) DMHM-4 (201) 171 140	MDMM-2 (145) (210) MDMH-4 (146) (218) MDMM-2 (211)	MDHH-2 (194) MDHH-4 (197) MDHM-2 (196) MDHM-4 (198) 223 23
DMMM-2+I (204) DMMM-4 (205) DMMH-2 (169) (159) DMMH-4 (206) Inactive Solid state done			

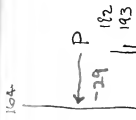
in Green: NIOSH & NMR samples

505  
15(16)



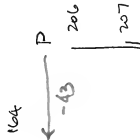
di me moxy

HH  
1780  
MW 193



104

HM 180-201 143  
MW 207

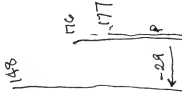


104

105 190 192

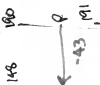
wetly low dioxy

HH  
MW 177



148

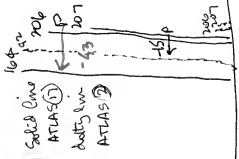
HM 198  
MW 191



146

141

M(H) 162 (162) (144) 143  
Loc MW



104

MM 162-204 143  
MW 221



206

190

106

220 221

MH  
MW 191

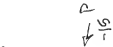


176

140

191

MM  
MW 205



190

101 102 103 104



