





A.T. Shulgin

Clean notes: Book #8

Many found during post-Thursday clean-up.

Thursday, October 27, 1964 - the BIG day!

known quantities

H<sub>2</sub> → we found 161

H<sub>2</sub> → H<sub>0</sub> 133

5-Substituted

5-MAD-DIPT 108, 109, 124, 147, 265

5-MAD-DAET 264

Tryptamines:

MSS  
or  
NMRs  
↓

DSBT 58  
GIPT 56

$\alpha, N_2$ -TMS 5

NMT 22

MIPT 32, 125

MSBT 34

MPT 36 (DPT) 88, 84, 85

DAET 38 (NRET)

NET 54, 64 (di)

NSBT 58 (DSBT)  
(T)

NIPT 60

NPT 62 (DPTXT)

BMT

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

nox

pip

pyc

S-meO

T

102, 104

~~132~~ wip

132 wip.

MIPT quat 125

Chalk:

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

Peganum harmala: 48  
Tetrahydroharmane: 44

5-MeO-pyrr-T 120

index isogenic:

6,7(CNS)<sub>2</sub> { 138 amide  
139, 141  
140 → dihydro-<sup>try</sup>  
142 steric activity  
144 - phen  
143 spectra 162

NHE

6,7 MDO amide 145A spec 150

6,7 MDO ~~145~~ NDPE 156 → 190  
Kovamide 111

DMHT-4 NH<sub>2</sub> → formate 170

formate → BHQ 171, 172

→ THQ 176, 207







2.0 g indole carbonylalkaldehyde  
 25 g EtNO<sub>2</sub> Δ → sol.  
 0.5 g NitroOAc SB sol ~ 2 min 1/2 hrs done by TLC shift

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

ML's 0.81g

xtal f. 10 ml EtOH Δ ▽

→ small 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>

0.3g NitroOAc - on EtO 16:20 [120] v. dark already.  
 off (115)

↳ 1.58 g very red xtabs.

filtered on 25 ml denatured EtOH Δ to Sol. ▽ OTH

filter - work i small amt IPA -

↓  
 solution → ML → sweep

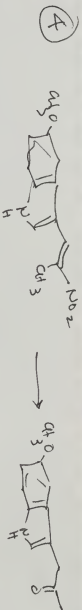
air dry. 1.24 g old gold xtabs

③

9g wet orange solids

0.57g

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{ 8 ml H<sub>2</sub>O<sub>2</sub> } on 5.8. add  
 { 2 ml H<sub>2</sub>O }

1.7 g electrolytic iron dust, when hot, add  
 0.5g NiO-NiS a glob at a time, over 2 minutes  
 10 min add NiS again - new starts at origin  
 & in between.

TLC  
 chloro  
 benzene

Into 100 ml H<sub>2</sub>O - 2 flasks and secondary incubation -  
 filter through paper - separate, 2x50 ml extract - wash  
 & set K<sub>2</sub>CO<sub>3</sub> - filter  $\rightarrow$  0.28 g film.

IR at 0.1 mm. 130°  
 160 (vac to 0.08)  
 215-230° over  $\rightarrow$  0.24g oil - off white

IR  $\nu$  = 0 at 1710  $\text{cm}^{-1}$  when nice OK.  
 stand a couple of months  $\rightarrow$  keep brown color.

Reagents:

30 ml H<sub>2</sub>O<sub>2</sub> }  $\Delta$  58 to 60°. add  
 5 ml H<sub>2</sub>O  
 4.55g Fe electrolytic - up to ~ 70° - bubbling, add  
 1.24g Ni hydroxide - 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 & collect. separate - separate - dist off  
 & 2x50 ml CH<sub>2</sub>Cl<sub>2</sub> - combine, Wash  $\rightarrow$  1.17g brown oil - dry at  
 achi acid barrel

Stand 3 days in cold lab  $\rightarrow$  stands! wip - int wgt by 95° - polymer?

TLC (CH<sub>2</sub>Cl<sub>2</sub>)  
 OK faster  
 known.

5

$\delta, N, O$ -TMS



0.22g Ketone (4) in

2.5 mL IPA

prepare

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

add

+ 0.4 mL 40% aq.  $\text{NH}_4\text{OH}$

+ 0.3 mL 25%  $\text{NaOH}$

stand a week.

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

To 4.3g  $\text{H}_2\text{SO}_4$  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 0.11 the, near end.)

into dilute  $\text{H}_2\text{SO}_4$  - wash & check

let evaporate

70 8:05

6:00 PM - 7 drops.

6:15 PM 5 more

2 PM next day 26 drops

2 PM next day 3 drops

2 PM next day 2 drops only (handful)

begin

Evaporate to 0.76g.

distill - 0.15 mm

then 0.05 mm.

to 160° - then over. - clear white oil

0.65 g white oil.

Wince out (ether, near, then)

→ 0.58 g pale yellow solid

let evaporate

5B

transfer to storage vial -

0.41 g 5D

0.76g -

120°

0.5 mm.

180°

0.5 mm

0.65.

play - IPA,  $\text{CO}_2$  no stab

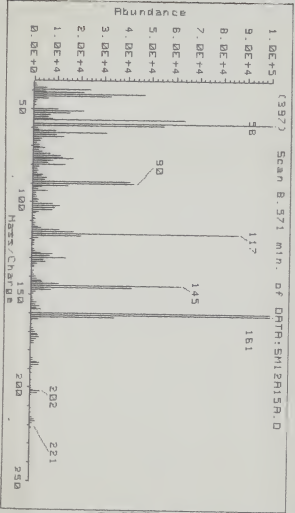
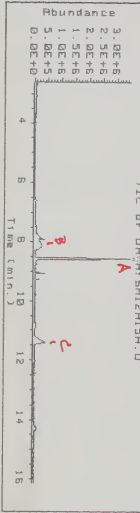
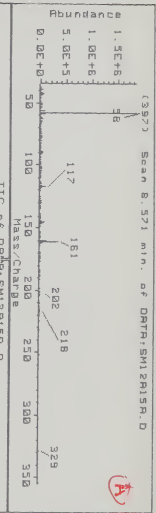
play - succinic acid, IPA

no stab

let stand.

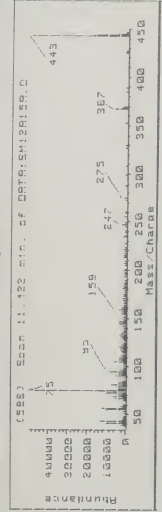
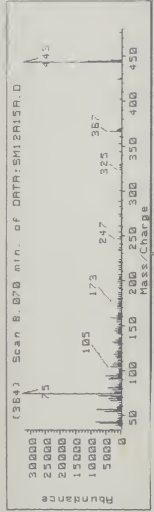
5C





PC = which 3  
CH<sub>3</sub>  
58





SIM of 203 → no Ketone.

205  
206  
207  
208  
209  
210



Properties of the celluloses:

Methylcellulose:

IR:  $C=O$  1680  $cm^{-1}$  fingerprint. 720, 748, 771, 932, 1043, 1096, 1127

(H<sub>2</sub>O)

wf, chunky 220 (amber) shr. 230 wfp 231 - 235  $\bar{c}$  dec. bubble

Sample 60g  
Nova Screen C<sub>1</sub>

G.C.M.S.: cracks ~ 60/40

149/58  $\rightarrow$  207

149/56  $\rightarrow$  205 delugna

See page 10, 11 MW 243

Dietingone:  
(H<sub>2</sub>O)

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

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

Sample 15.188  
Nova Screen D

G.C.M.S.: cracks ~ 70/30

149/100  $\rightarrow$  249

149/98  $\rightarrow$  247 delugna

See pages 12, 13 MW 330

t-BUTYLONE

IR:  $C=O$  1651  $cm^{-1}$  fingerprint: 988, 941, 1046, 1111

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

Sample VIII-107  
Nova Screen A

G.C.M.S.: cracks ~ 50/10

149/110  $\rightarrow$  249

149/98  $\rightarrow$  247

See pages 14, 15

MW 285

ARIPONE (vacuic)

Sample I-113  
Nova Screen B

Dane

fluorocalls

#2 April 14 1995 - BK -

We will alert we re

Pablo sources.

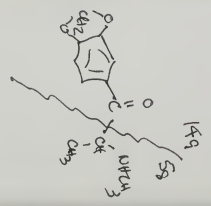
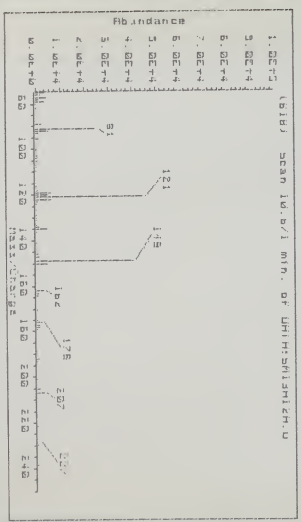
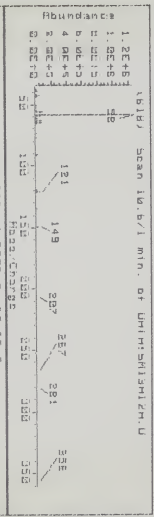
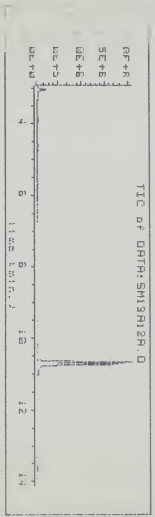
\$110 for Mikeo candy-pans.

MW 259



110

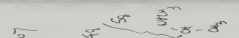
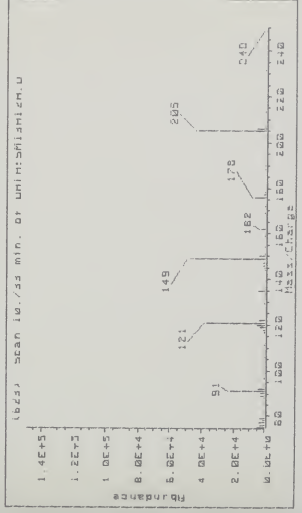
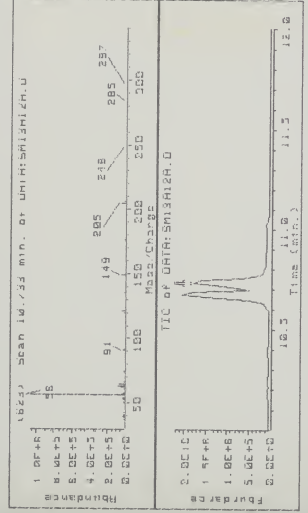
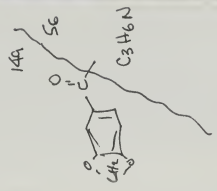
from methylene GCMS



mw 207

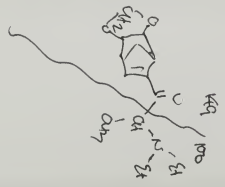
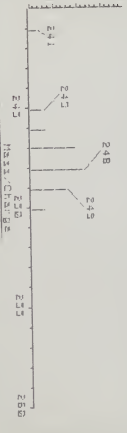
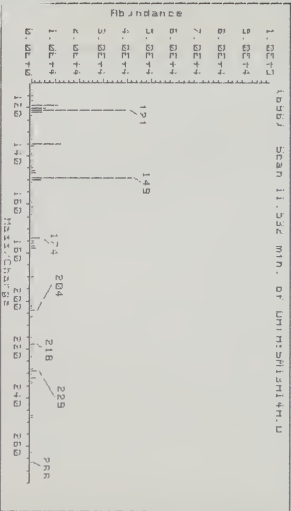
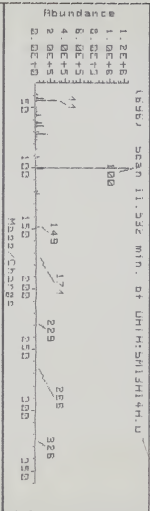
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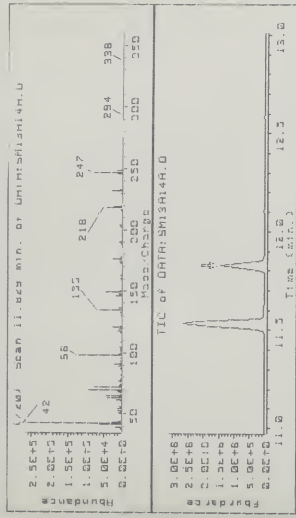
Nov. 20 5



(12)

DIETHYL OXIDE - See Page 9





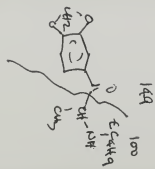
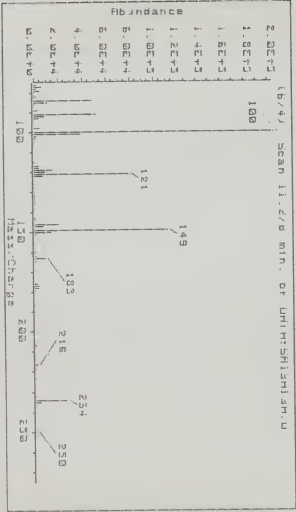
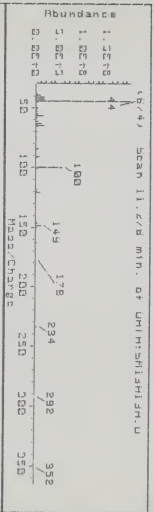
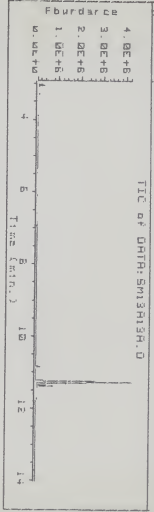
144 mw 247



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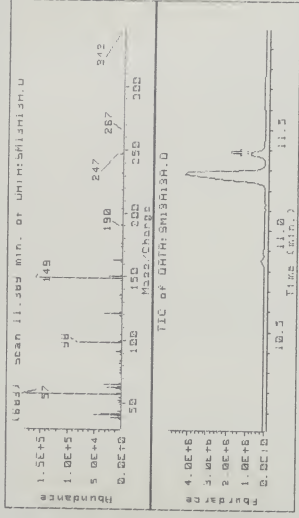


(NA)



MW 249

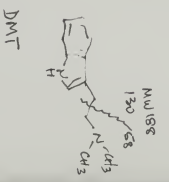
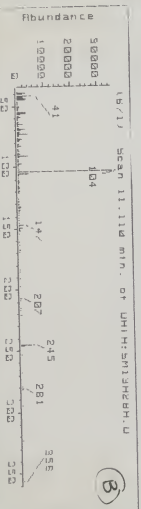
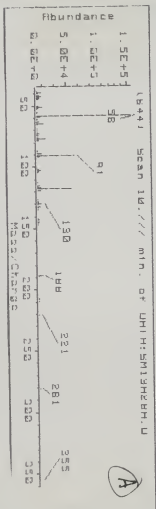
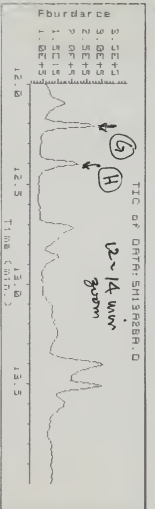
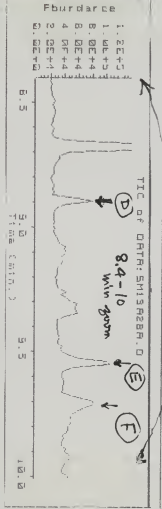
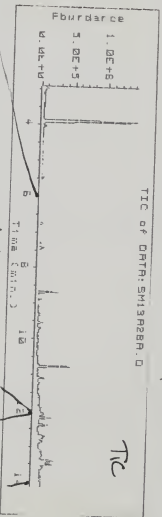
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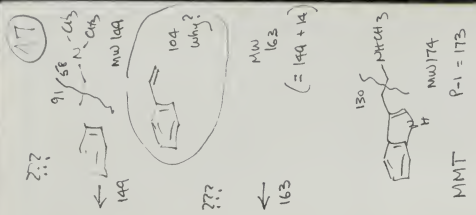
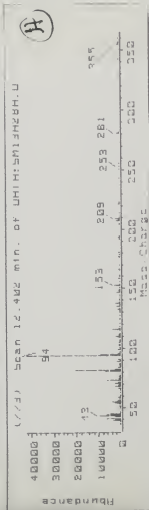
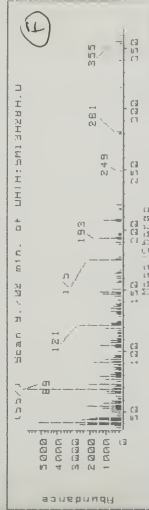
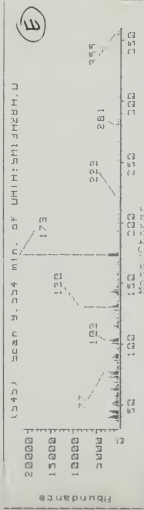
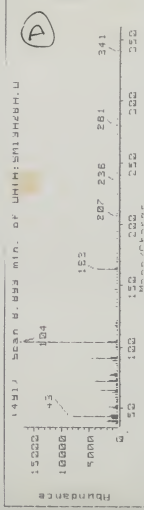
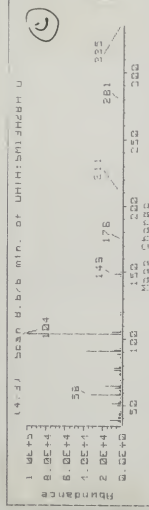


in  
C<sub>6</sub>H<sub>12</sub>N  
N

Ab

Acacia Saponaria, alkaloid fraction, ex D. Siebert





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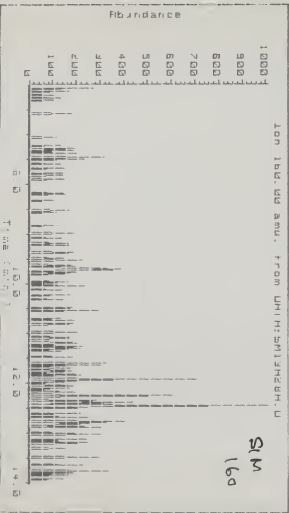
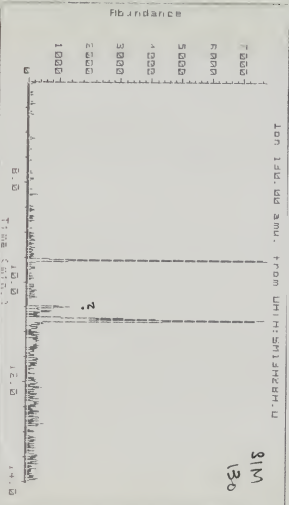
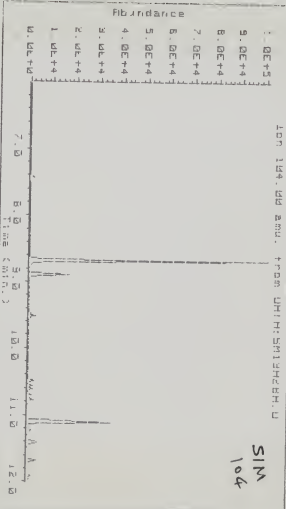
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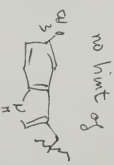
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16



MMT  
DMT  
probably was  
often undetermined



no 5-MeO-DMT  
5-MeO-MMT

100 104.00 200.00 FROM UH1H1SH1F2R2H.U

100 130.00 200.00 FROM UH1H1SH1F2R2H.U

100 160.00 200.00 FROM UH1H1SH1F2R2H.U

12. 180  
48 180  
12 16  
21.5  
192g  
candle



10 g tryptamine - abs'd  
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

xHCl 3 x 25 ml ethyl  
purpl - wash 1 x dist H<sub>2</sub>O. 50 ml.  
wash 1 x 5% NaOH 50 ml

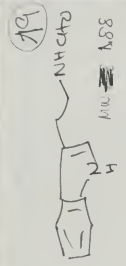
11.5g candle  
11.5g nothing  
13.5g .05 min. water  
40°  
45°  
45°

strip on PE. to hard vacuum → 11.5g black oil.  
into V.R. Vbr to 0.05 mm. distil  
220-240° oven - bad vacuum tubing?  
Almost color. Still stuff in pot.  
pink to 250° - no residue in pot.  
not x HCl ON - some wall oil in vial.

let stand - ven. hot weather - very slow x HCl.  
GCMS shows some inverted tryptamine -  
and, strongly - hints of di-ethyl.

a week later - slow growth of more x HCl  
Add ~30 ml Me<sub>2</sub>SO  
reflux oil - Mass Spec. now a simple formate peak - BUT  
a smut at 3 minutes? strip hard → 77 g. HCl

170 spot  
190° base  
0.1 mm

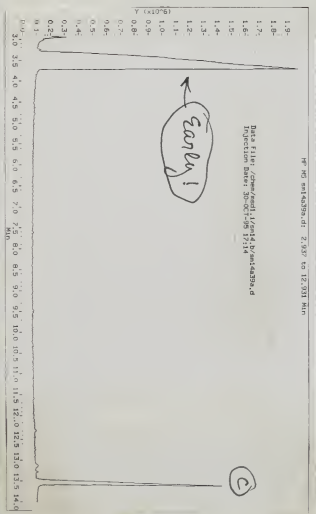
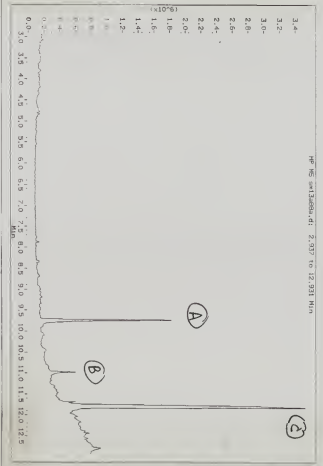


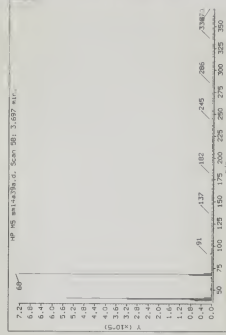
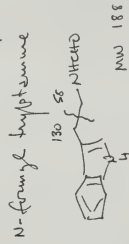
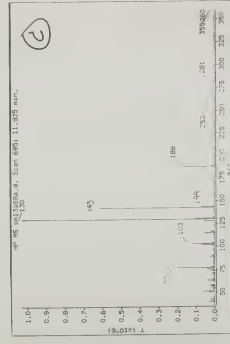
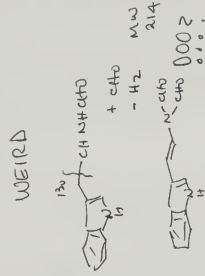
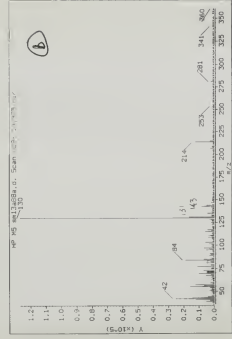
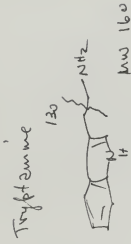
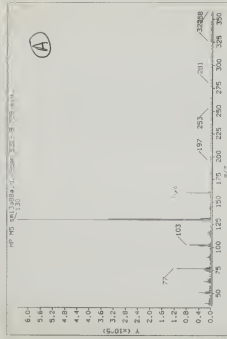
(purple paper 20, 21)

(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 Tm page (19)





early!

???

↑

(2)

22



In a PB 3 neck flask - Argon - stirrer, reflux, good gas flow. Add 100 ml 10 LHM in THF. At room temperature. (60 min)

Make a solution of 1.88 g Ferrocene in ~ 20 ml a.c.h. after. Δ LHM to near reflux, add ethyl sulfonium diphenylsulfide - 2 fold excess of LHM at next generation.

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

Remove a squirt of the soluble for a.c.h.s analysis →

Am - to RT. 24 hrs later - kill with

20 ml 50% a.c. THF. no more figs.

Filter, wash c 4 x 25 ml dump THF.

Check pH of filtrate - dump pH paper. DMSO

Scrap filtrate + washes to dryness in the rot. soap

→ 1.39 pale oil. waiting to decide to

acid of base - start of spontaneous xtals -

Sour Seed -

Distill at KR

0.11 mm / Hg 60° no

110° clear

120° wire beads

135 - 145° men. white oil.

1.22 g - spont. & immediate xtals to white solid.

4HTT NMT

di-tert

Magnetic bar w/ 90° wrap  
 w/ wrap 180°

Conversion to the 0.22 g base (no wt taken) (flaming really burning) + 1g IPA

Δ puts it into solution. add conc HCl - acid dump → green-blue color. Heat dissolution on stirring - when acidic - color stable - no xtals - add a 2<sup>nd</sup> and often → clearing - stir - smooth → xtals after yellow wash then silica wash - dry to constant wt -

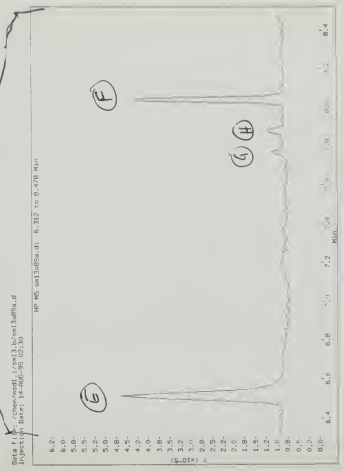
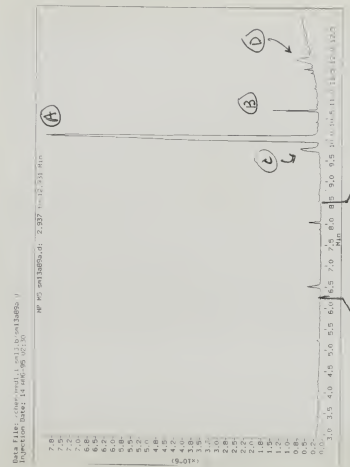
after 0.116 g.  
 64% white & blue  
 xtals. (grey  
 of Hg)  
 weight ↑  
 w/ 174-6.



GCMS of crude, subseq, of 22.

(23)

See separate analysis vs (A) in page 26



Abund

7.4

100.00

(1.0)

100.00

100.00

100.00

100.00

100.00

100.00

100.00

100.00

100.00

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100.00

100.00

100.00

BMSG

A. sub

to

peak

INT

100.00

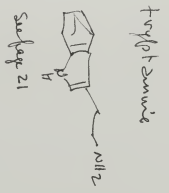
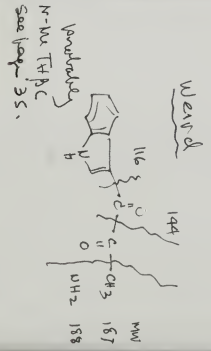
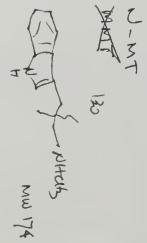
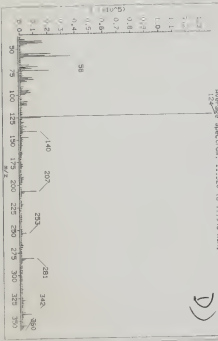
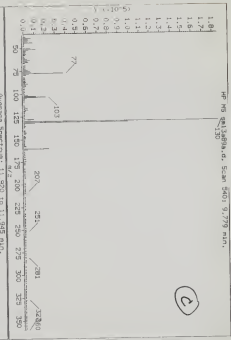
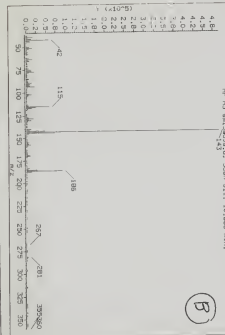
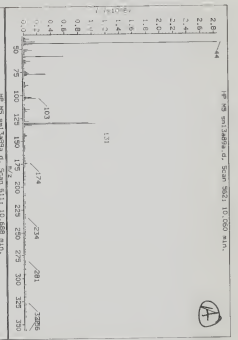
100.00

100.00

100.00

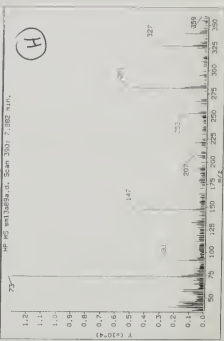
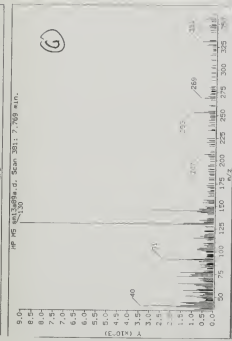
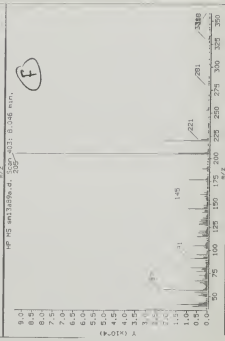
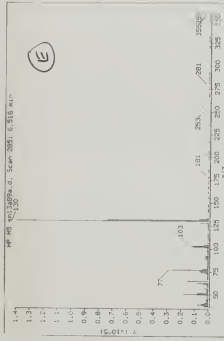
24

from page 22-23 under MT



See page 21

25



skatole ?  
 MW 131



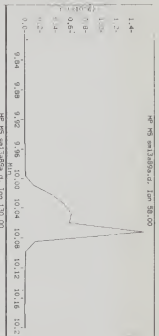
?

dimethyl-  
ethyl- } tryptamine.  
 MW 145

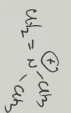
?

2c

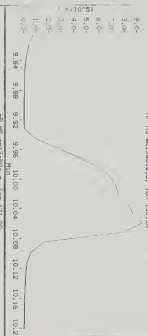
Contains four complexity in peak A, page 23



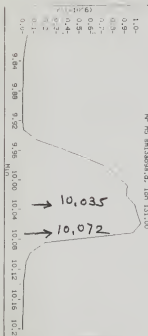
ion 58



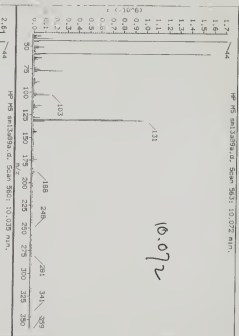
JMT



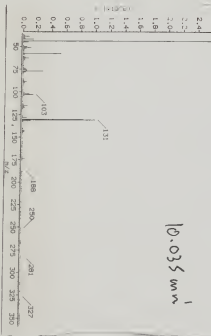
ion 131



ion 131



10.072



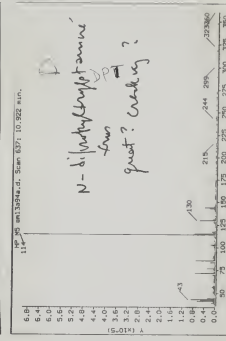
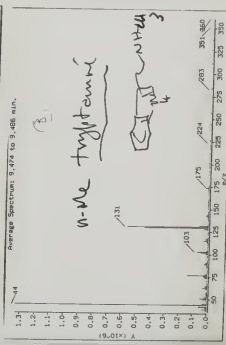
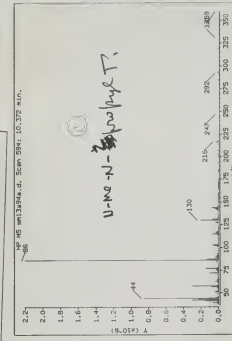
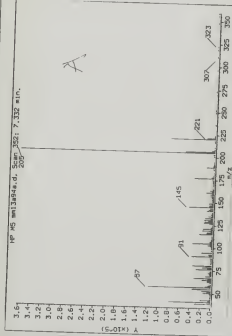
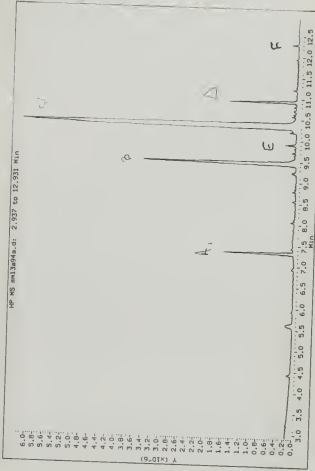
10.035 ppm

at SF.

add neat hydrolysis vial to a few milligrams of  
MMT - heat 100°C 1hr → GCMS that says 4/5 hydrolyzed,  
some trace Pr<sub>2</sub> (= <sup>125</sup>I-Pr + cracking) of bulk

27

Data file: /chem/msd/125125/251251.ms  
Injection date: 05SEP07:11



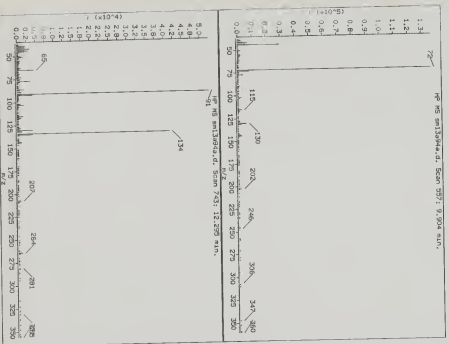
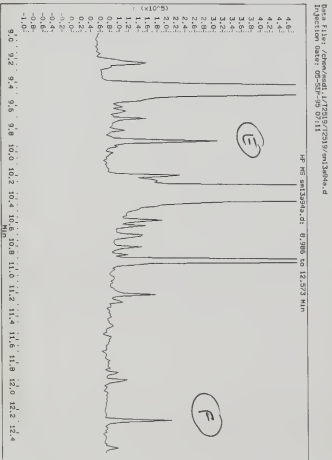
13

MT

7



28

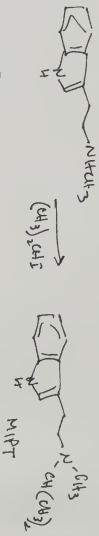


29

(  
20

3

Outcome:



0.3 g free base NMT (1.2 g batch; page 21)  
 dissolved in 4.5 g EtH - slow to dissolve RT, color  
 goes in first - ∴ 2 weights good for K react. solvent.

1.2 g verpurgel iodide (4x shot) 50 10 lbs.  
 TLC 95/5 chloroform → good done (wide separation)  
 50/50 chloro, strike however.

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

Shift to residue - sunbaked between 50 wet chloro  
 and 25 wet 5% NMT - all goes in - color mostly  
 to chloro - separate - shift base is a little white -  
 part - water is a 5% sodium base in the soln -  
 5mm, not all, color goes. nice water is 5% w/w -  
 Peak → 0.36 g (wash oil for cleaning).

Do full 0.19 g which ~~behaves~~ but still behaves  
 150-160° → a white oil - will not crystallize  
 0.19 g very viscous.

130 Blank  
 135 - long blank warm files  
 at 135  
 130  
 150 over.  
 0.05 w/w lbs  
 0.36 g excellent base 97% MPT  
 5% solvent  
 NMT



(33)

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of

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of

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of





36) repeat of SE run.

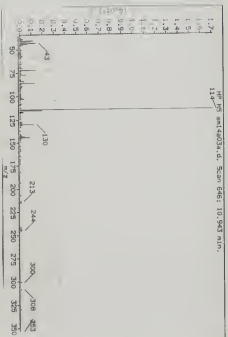
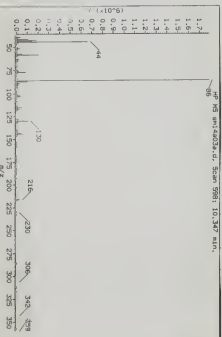


0.3 g N-methylmethylamine, into  
4.5 g IPA - acid

1.3 g 1-iodopropane. - reflux 1 1/2 hrs,  
strip - excess. were slow down

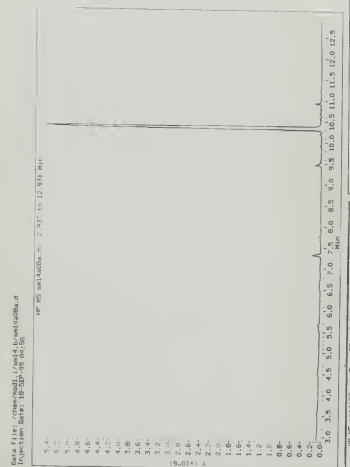
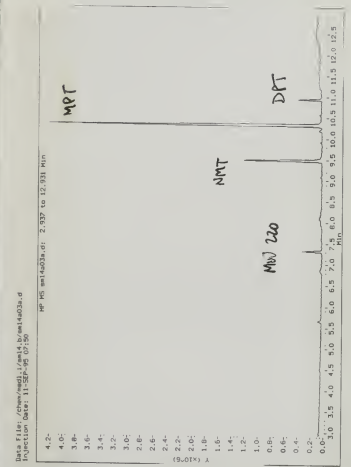
+ 4.5 ml IPA + 1.3 g 1-iodopropane - STB (IPA. water removed)  
5.5 g strip - IPA - 160-170°/0.05 → 0.19 g off-white oil 0.19 g

1.25%  
0.05mm  
190°  
100-110°  
even  
0.05  
0.19g

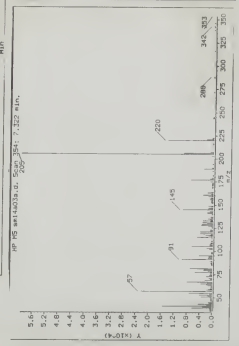


37

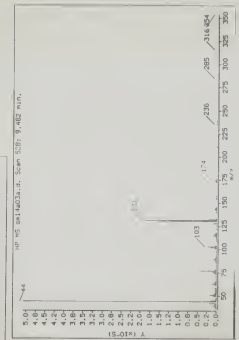
1/2 hr  
assay



12 hr  
assay



MW-220



NMT

mpf

NMT  
MW  
DFT  
0.8g

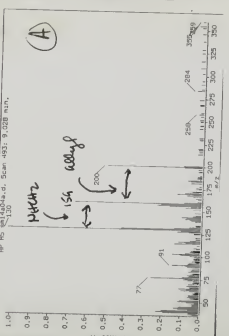
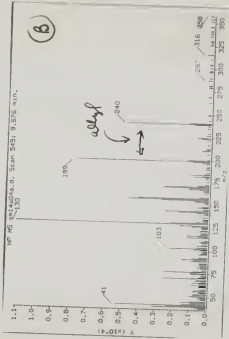
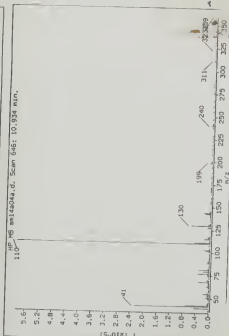
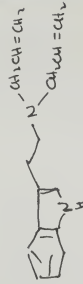
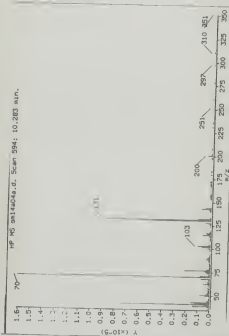
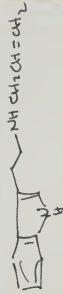
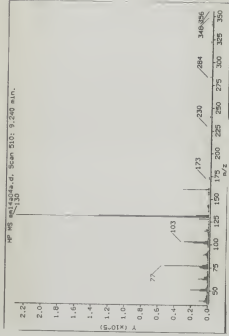
mpf  
NMT  
MW  
DFT

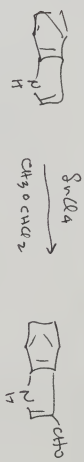
mpf  
NMT  
MW  
DFT





39





Dry snw ch<sub>2</sub> by boiling pool → low vol on SB. cover immediately.

In a 250 RB flask - veg stirring - add

10 ml dry ch<sub>2</sub> - add  
1 g indole - stir → solution (under hot) - cert  
to go to extracted ice bath, add

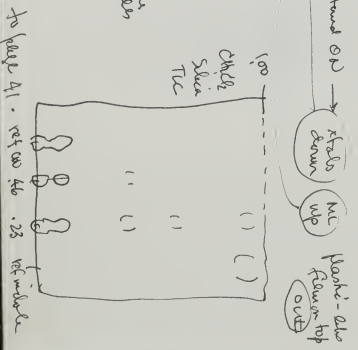
12.5 ml 1 M SnCl<sub>4</sub> (in ch<sub>2</sub>) of over ~ 2 minutes, dispense - turn a slightly cloudy grey color. Then add:  
1.0 g neat reagent's slowly (stir, still at 0°) turning a tomato color.

stir 15 min 0° - then  
1 hr RT. Pour into receiver, wash  
basic & ag. wash → orange ch<sub>2</sub>  
phase & yellow ag.  
Solvent extract ag. 25 ml ch<sub>2</sub> - combine  
wash in 100 part. Separate & flash.  
pale yellow phase → 1.78 g crude  
trans oil.

Into a 10 ml vial - rest stand 0°

grind up.  
→ 0.4 g  
brownish-  
linear solids

into  
big leftish skin  
oily oil →  
wet oil & stir  
with 2 x 5 ml  
hexane → washes  
brown solids  
with 0.25 g  
st. granular solids



to page 41. rest on 46 .25 rest on 46

from bags to

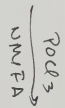
Combine 0.46, 0.23 g - grind under 10 mesh IPA - stand over  
→ brown solids after filtering, working in IPA. 0.34 g  
Feinble, free of indole & .5 ref shift -  
forgot to TLC MW - vod known oil as vendor

100  
( ) ( )  
" "  
( ) ( )  
( ) ( )  
8 6  
ref .23 .34 indole  
CHO

(4)

(42)

try



11 Picked 580

7.2 g ROCl<sub>3</sub>  
6.7 g NMeFA

Let stand RT start 1700 on clock.

by 1735 just clear enough - into 583 Temp add:

↳ 6 minutes - was really - to body binding.

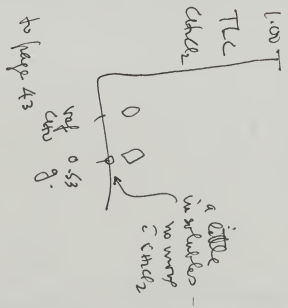
2.5 g imhole - vagnon - gfs very hot - waste - binding.

let go ~ 5 minutes - into 150 we had - 5th 01.

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

Meat - scrape, scratch → Strids.

↳ 0.53 g Rauscher shell.



Reheat, but cold.

page (42)

7.2 g POC 3

6.7 g NME FA.

~~2.5~~ 2.5 g Indole

still very exothermic, even at 0°C.

stir until all at ~0°, no more rx -

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

stir → water wash out - red color into water.

decant, + water 2x

blebby oil + almost ~~blebby~~ no red into water.

decant - then + 10 ml MeOH - strands OK.

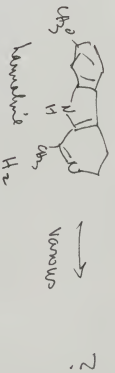
(43)

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



44

trial:

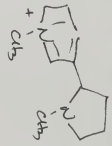


A bunch of trial changes of norbornene, different endo, exo, to challenge its end, the stability. The more hydrogen, the more norbornane

- (A) H<sub>2</sub> in water, low depth the **high**.
  - (B) H<sub>2</sub> in water, low depth 25% **high**.
  - (C) H<sub>2</sub> in water,  $\bar{\epsilon}$  Ultraviolet C. **high**.
  - (D) H<sub>2</sub> in water,  $\bar{\epsilon}$  water: **high**.
  - (E) H<sub>2</sub> in H<sub>2</sub>O,  $\bar{\epsilon}$  strip of gas: **high**.
- add - submerged the by acans
- (F) into acidified (H<sub>2</sub>O) water, + Adams catalyst, + ag. phosphate salt - hot - sparsely - make basic, xnt  $\bar{\epsilon}$  90/10 Adams Reagent. acans. extending tetrahedral lowwise Ha.



(46)



24.58 g methine (just over 15 mols)  
use ~~2.3~~ 3 mols HI (to pH ~ 2)  
at 50% strength.

into 200 ml kanthard

add 75 ml 50% HI (try to get pH ~ 2)  
failed on the 1st attempt - have been  
water - wet first - 2 night. was it in  
strongly acid.

Play: other, 2 floors - no xtds - plant - dilute  
in water

add to 1 L.  $\epsilon$  devalued stuff - other to seed,  
ready to add 4 L. other  $\rightarrow$  xtds - stand on water -  
a bit of a  
time,  $\epsilon$   
other

fill to, water = 50% other/seed  
 $\rightarrow$  light yellow xtds  
air dry 2:1  $\rightarrow$  38.50 g

Mix + other again  $\rightarrow$  2:1 other/seed ratio -  
were light yellow xtds  
filter, wash  $\epsilon$  2:1, air dry 2:1

$\rightarrow$  14.0 g

Mix of M.L.S. -  
turning very dark  
OUT  $\rightarrow$  52.50  $\rightarrow$  53% yield

Continue 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  $C_{12}H_{21}I$  (10% xs) - into SB vial at 4:45 PM.

slight darkening in evening - let go ON -  
off noon. play a bit, scratch, then,  $\rightarrow$  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 20 mL portions of MeOH -  
Air-dry to constant wt. 98.24 g pale yellow  
shards

98.24

MW's

nicotine	162
N.HI	290
N.(HI) <sub>2</sub>	418
$C_{12}H_{21}I$	142
<hr/>	
$6 \cdot C_{12}H_{21}I$	304
N. $C_{12}H_{21}I$	432
HI	

72.84 g  $\rightarrow$   
N.HI

(47)

(18)

*Pogonum hamada* → alkaloids

1.5g *Pogonum hamada* seeds - grind to powder in water -

5 mL SAT NaHCO<sub>3</sub> +

5 mL ether - boil for 10 minutes  
add ether 5 woods.

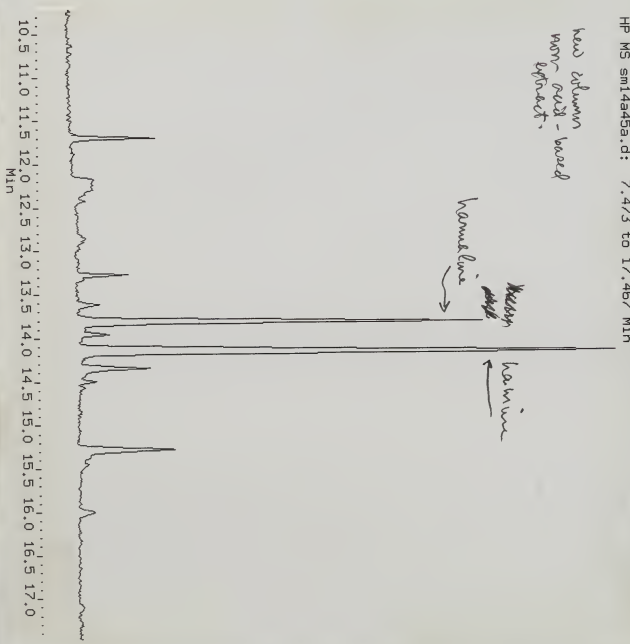
decant - wash with ether → rot → Resin  
→ 0.11g Resin -

Swelling oil?

Residue in 2x2 mL ether - Resin → 0.055g - least oil

HP MS sm14a45a.d: 7.473 to 17.467 MIN

head column  
non-poll - based  
extract.



49



50

(5)

52

Attempt:



Ac2O  
Et3N



NH  
CH2CH3

hydrophobic

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

25 g Et3N (MW 101) 0.25M - >100% xs. , added  
spontaneous  
good  
strong

11.2g Ac2O (100% xs). very visc - gets  
thick

stir until acylation done - onto Sephad

MN - 2 phases on 48. stir down to a thick, gummy  
sludge - spread a, feed sharp - add w/ 50 wt chices - very sludgy  
to go into solution - then clean solution - add more chices -  
add 100 wt 100, 110 → pH acid.

Separate - extract aq = 2x50 wt water chices?  
wash chices phase 1x w/ 1/4 sat NaHCO3 - very sludgy pH 10/11 -  
separate - stirp → 12.57 g dark viscous oil - to KR3  
Use sludgy form 0.5 - acet 0.2 → 0.1 min. dilute.

183-200° / 0.1 mm/Hg. about 9/10 clear - residue  
lookes increasingly sticky. Oil in vacuum canben. lookes  
good.

→ 9.45 very vis oil - canben.

1330 hours .  
22:47

9.37

Flame out → 9.37. extracted play → white chads  
grass

no in food  
stick.  
ketone → carbon  
left → xthals.

oil → visc solids

mp 70-73

mp 71-71

73-74

53

1/18  
1/19

1/20

1/21

1/22

1/23

1/24

1/25

1/26

1/27

1/28

1/29

1/30

1/31

5A

Attempt:



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

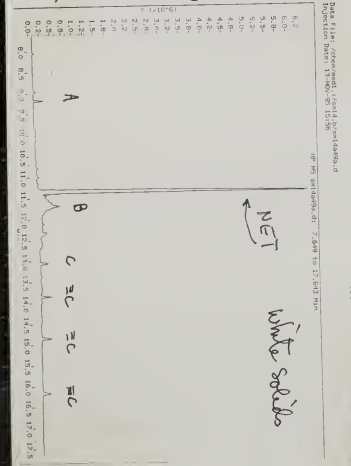
11 May 22

13

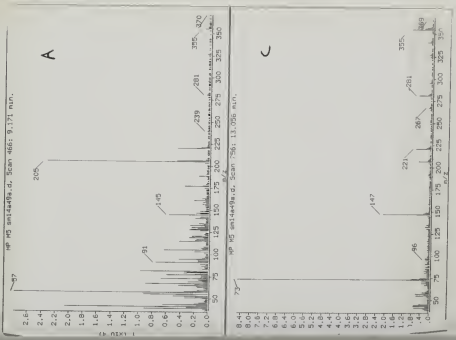
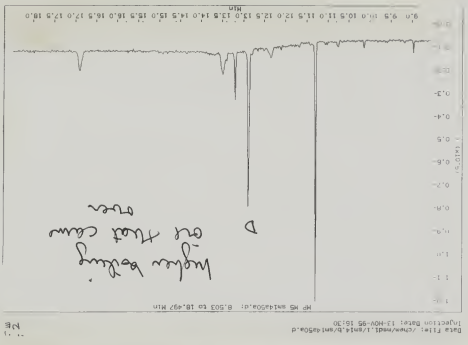
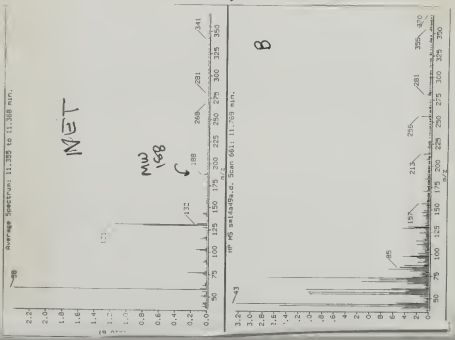
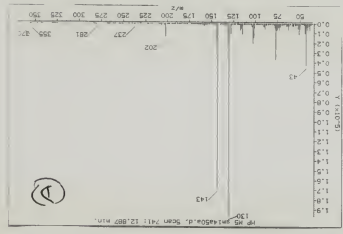
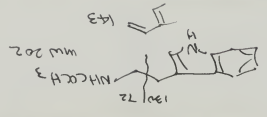
A solution of the hard glass acetamide  
 The 30 ml dry THF, water Ac. Heating? (pale yellow  
 solution (water 2.02, used 2.315 - levible glass),  
 A solution of LAH in THF (1M) 60 ml - brought up to  
 during reflux, under N<sub>2</sub>, stirring, 3 ml RB soave.  
 Add amide dropwise - good stirring. Hold at  
 reflux 10 pm.

Stir 20. Sample at ~ 10 min - (let cool, let stir) here  
 Next day - add 50 ~~ml~~ g. THF + stirring (total 200 g)  
 Filter through paper - wash 2 x 25 ml OK THF.  
 ML base to wet lit paper - strip → 160 g pale  
 cream-colored oil.  
 (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>: 125-135°/0.1 mm Hg → white rice treat  
 x holding in the vacuum.  
 → 1.58 g. product for fur - table 2. drop by

160 g  
 styrene  
 90° w  
 125° 0.1. start  
 white,  
 little more by  
 44° w.  
 Free base white and  
 (Acromo benzene 2) hard  
 as white, w/ 80-81°  
 150 mg into 750 mg IPA  
 to sample the  
 a analyt. often →  
 of clearly - show K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>!  
 10 days (81-182°  
 w/)



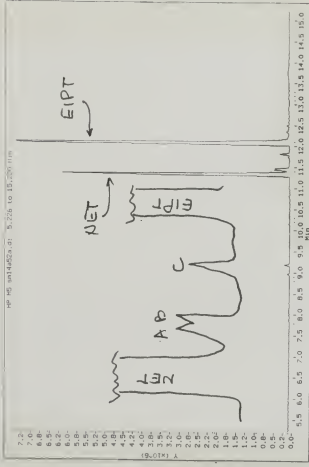
55



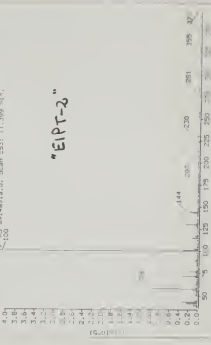
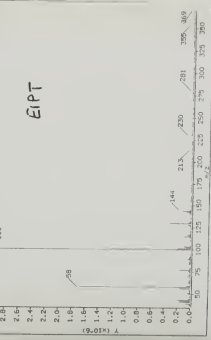
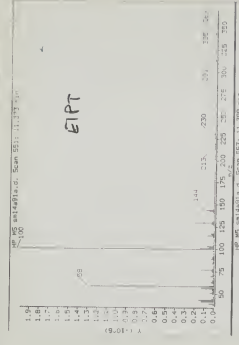
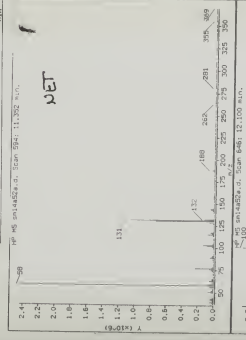
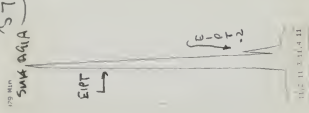




Date: 1/10/1988  
Function: Data: 13400000.D



ST  
DWA 041A



- (A) 72 130(31) 202 242N(C2)
- (B) 72 130(31) 202 242N(C3)
- (C) 44 86 130 small 210 ext. 242N(C4)

(neutral) displacement of alkyl on I?

6600  
44(86)

580

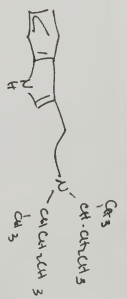
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58

Attempt:



See Br.Br



DSBT (vapor & recoverable)

1.0g hydrochloric acid, into  
 8g IPA - warm, no, build to give solution. slow, not low;  
 5.5g 2-bromo butane - mix vial by SB - 15 hrs total  
 into 150 ml pro, add 150 ml water → give color &  
 clarity - xrt 2x 50 ml water - wash-water: 1:10  
 flash → 1.72 g clear residue - to IR.

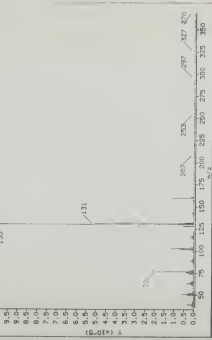
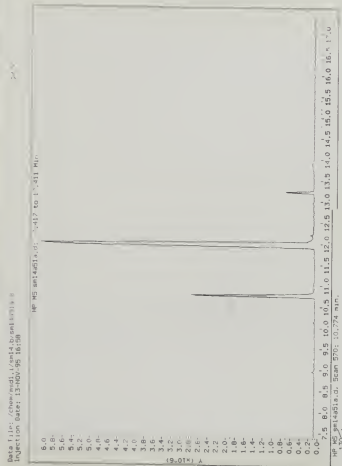
Starts over at 120°/0.05 mm - very faint, carbon, then column.

end at 150° → 1.17g white oil.  
 Residual  $\frac{1}{2}$  least 1.1g

in a waste on so - two glets of low melting white solids  
 small amt,  $\bar{\epsilon}$  IPA,  $\bar{\epsilon}$  the same,  $\bar{\epsilon}$  skin → fine white  
 NP 100; xrtals  
 SA 100;

All into 6 ml IPA - clear skin. add cover the (21.7m dtt)  
 Add ether to make (16 ml) skin → solids. stand (1 hr -  
 filter, wash & ether) → 1.27 damp, 1.07 dry white solids.

59

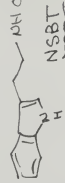


MW 160

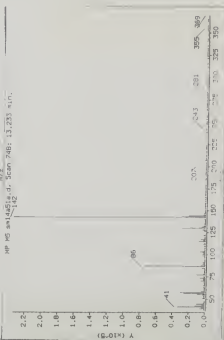
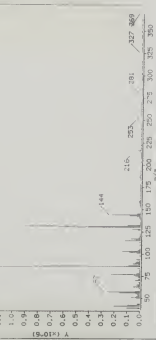


tryptamine  
I

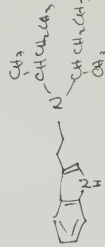
MW 216



NSBT



MW 272



DSBT

(60)



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

6.8 g isopropylaldehyde (20mm) - into reflux 500 PM.

off - Seulp → dark oil. IR E slow off. darker very dark -  
play seed → no

into  $\text{Na}_2\text{S}_2\text{O}_8$  + 150 ml water (pH → acid to base). sh 2 x 50 ml

chisel - separate. ~~separate~~ → separate again - dry ↓ -  
slow, careful. not a good extraction. little chisel - separate  
flam → dark oil. - base very dark water.

2.19 g dark oil. NIPT crude (shipped out)

distill at 14R. 0.05 mm ethylaldehyde. 0.2 mm burning.  
80° no 0.04 mm 115° start. 1st open ethylaldehyde. white oil now at 130° - distill 130-150°

151g total weight 1.51g - drain out 2 traces → 1.50g - whole

1m-140° distill in 6 ml IPA  
1m-140° H<sub>2</sub>O → blue/red 30-34 drops  
1m-140° H<sub>2</sub>O from 1/4 way through. add

4 + 30 ml ether → chanelade water -  
+ 30 ml ether → chanelade water -

filter - wash 2 ether (2 → canals, wash). can dry.  
↳ 1.55g. H<sub>2</sub>O mp 22.4-22.7°

Small amt -  
94% IPA flash  
mp 94-95°

(61)

1878

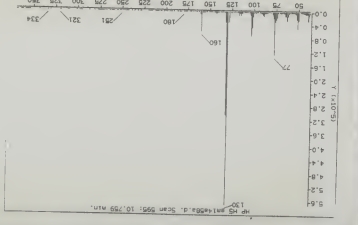
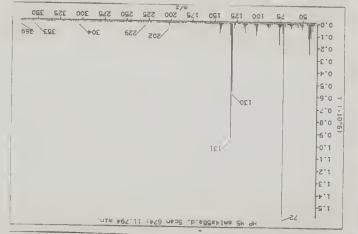
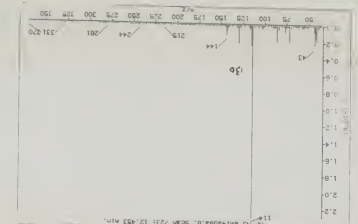
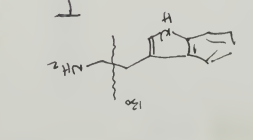
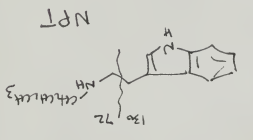
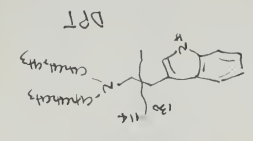
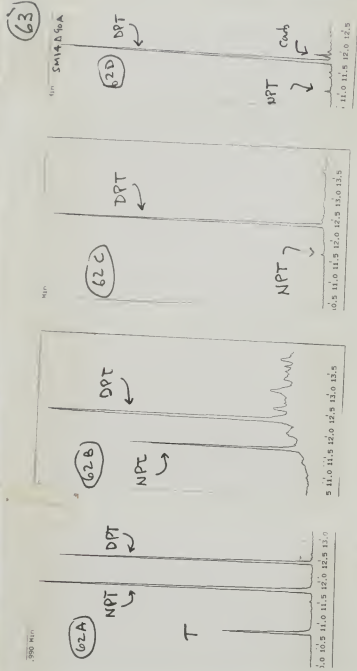
(62)

1878

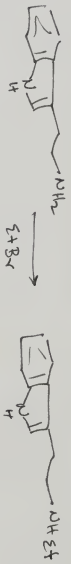
1878







164



3.2 g triptamine (2 mks) into  
 25 µl of IPA - add  
 8.72 g EtBr. onto gentle reflux in stirring 8PM Friday.

reagent:  $K_2CO_3$

2.0 g triptamine

5.6 g EtBr

15 g NaOH

8.5 g aash,  $K_2CO_3$

skin RT 2:45

pulver - slushy  $\rightarrow$  3:45  
 5 hrs - off skin, filter through

2:10  
 1:01

broil in  $4 \times 2.5$  ml chex - rub - convert to black solid  
 down white solid

filter through paper,  $\rightarrow$  0.55 black rd - (lyso)  
 clean  $\rightarrow$  1.67g  
 dunk  
 oil.

0.16 mm

0.05 mm

180-190°

KR. 0.05 mm 180-190°

$\hookrightarrow$  1.15g white oil (A)

dispense in 5 ml hexo -  $\Delta$  briefly on SB. (micro scale 37°  
 ACSMS (B) 4 ml

acid-base (into 0.5 ml  $H_2SO_4$  - wash 2x chex)

(boone  $\bar{c}$  6 µl  $H_2SO_4$ ) - xH<sub>2</sub>O - skin  $\rightarrow$  0.31g

KR. 155-160° / 0.05 mm  $\rightarrow$  7.19 g oil  $\rightarrow$  waste xH<sub>2</sub>O  
 $\Delta$   $\rightarrow$  white solids 0.175 g removed

1:64

31 counts

130°AP

0.08

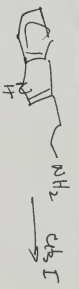
44-100

0.05

.9



(16)



1g hypofluorine -  
 10g (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SO  
 3g C<sub>2</sub>H<sub>5</sub>I  
 stir 18 hrs,  
 white solid under acetone

10g hypofluorine,  
 40g warm acetone (Spel)  
 36g C<sub>2</sub>H<sub>5</sub>I,  
 200mm - add  
 15g (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SO - in OK.  
 stir  
 ~ 12 hrs.  
 Solids in a minute.

17.5 g 20A KOT

Paper, work in acetone → cream solid - wet

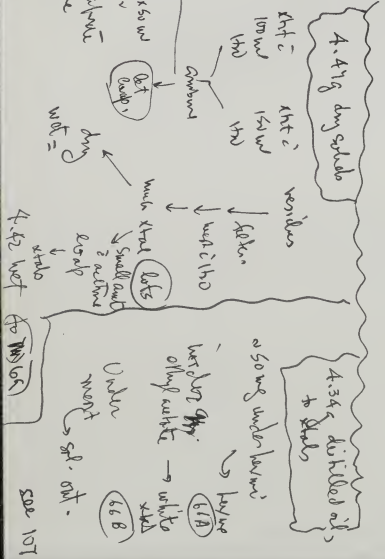
9.07 pap/KP  
 0.4mm

fill into chex, dilute base - seen gelation between dry  
 acetyl 75% ~~acetone~~ chex & ag. base. finally two  
 phases - separate  
 → chex phase (3500 wt) - stir 1/2 - was 100 wt KR  
 bubble  
 0.4 ~~water~~ mm. w.w.  
 80°, 105° no trouble  
 140° up → 170°. ~~some~~ some  
 1st variable? T<sub>1/2</sub> R up to 90/140 wt%.

100 chex!  
 105° slowly.  
 90°  
 (50 heavy?)  
 100 hours?

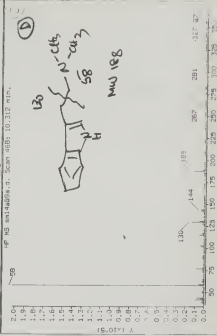
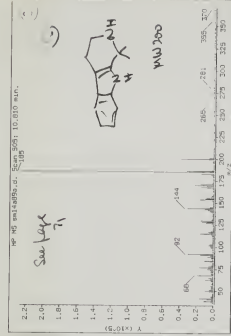
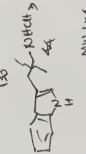
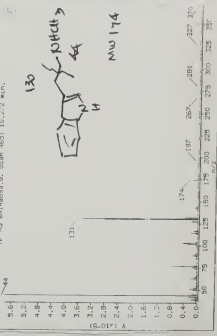
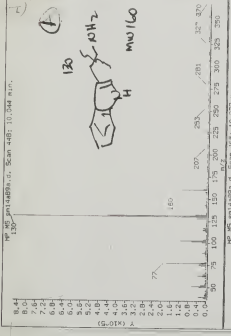
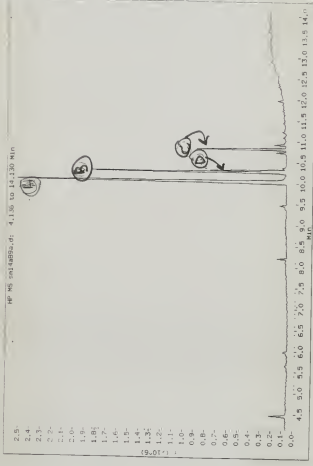
4.34 g acetone  
 MT.  
 K<sub>2</sub>CO<sub>3</sub>

← w/v  
 K<sub>2</sub>CO<sub>3</sub> w/w  
 CH<sub>2</sub>Cl<sub>2</sub>  
 let acetone  
 outside





MS 1111: C<sub>12</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub> (MW 210.21) 10.000000







Solids from Hydroxamine + MeI

(69)

4.42 g wet. This has been twice extracted c 100%  
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

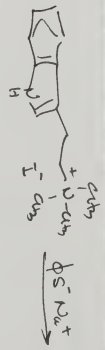
NH 3400 cm<sup>-1</sup> sharp.

finger print  
767 large  
919 }  
953 } medium  
978 }

in file

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

70



2.4g of grade I<sup>-</sup>, suspended in 40 ml MEK. add  
 { 1.0g pSt (n1000x5)  
 1.0g NaOH (n2000x5) }

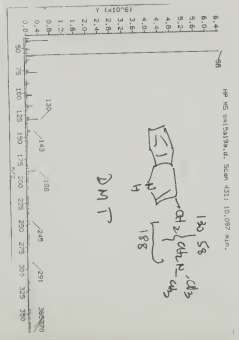
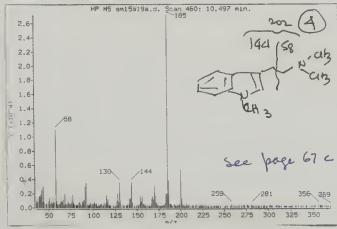
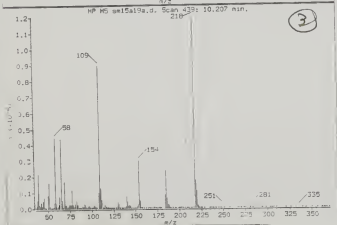
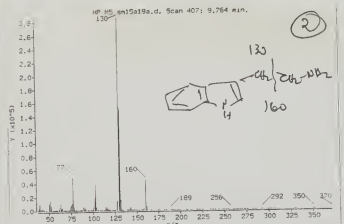
stir - to reflux at 45 °C.

have in flask from removal into.

have in my memory  
 reflux in white (3 hrs? 200?)  
 strip → ~~see~~ residues - white  
 tho? basic? strong NaOH -  
 ketones - strip →  
 residues -

IR - → reasonably high  
 making fraction - white oil -  
 weight ~ 0.10g slow, but  
 feed x100. IR - still oil  
 & no spectrum,  
 Ferriable yield,

70 high

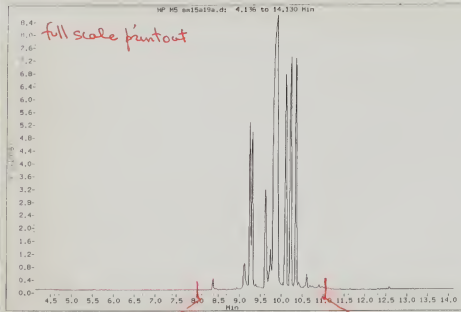


DNT

see page 67c



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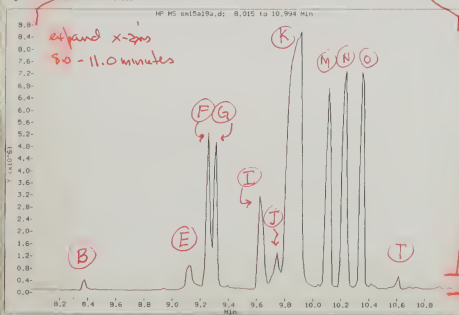


~~Archives~~  
 Pachycaerus bringleri

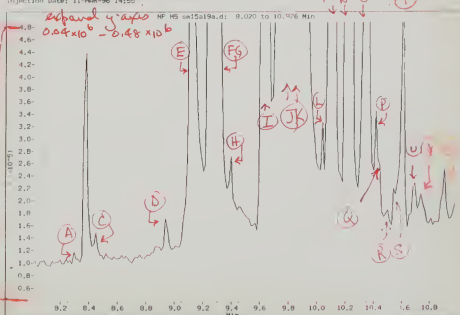


Total extract 1 part P.b. aqueous  
 1 part 6 N NaOH  
 1 part 90/10 toluene  
 butanone

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 Injection Date: 11-06-96 14:55



Data File: c:\chem\msd11\3522\3522\ms15a19a.d  
 Injection Date: 11-06-96 14:55



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 Z



Compounds reported in P. p.

(73)



NW 193 Heliammonium

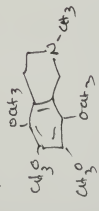


NW 193 Leandireocarine



NW 237 Tobaamine

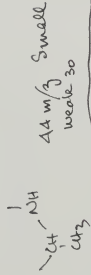
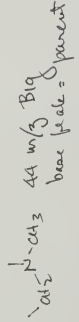
PUCM reports  
N-oxide  
NW 253



NW 267 Weberine

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

note - from M-8501



MW TALLY (Components)

152 (1561) 8	182 192-3 J (+44)	192	206-7	(267)? 219 (236) T	234	266
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page 74

page 75

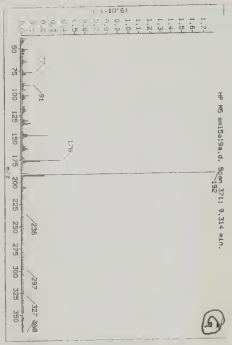
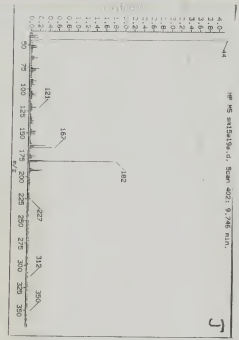
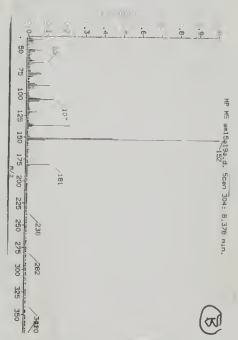
page 76

page 77



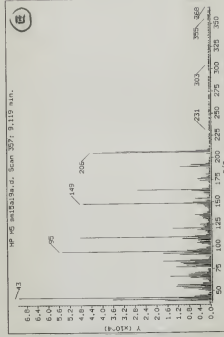
74

lower ml components



206.7 MW components

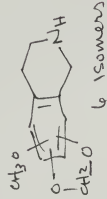
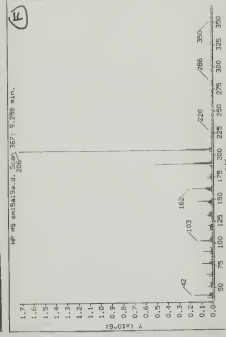
(75)



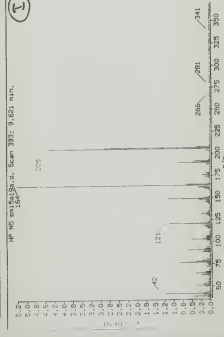
possibilities



12 isomers

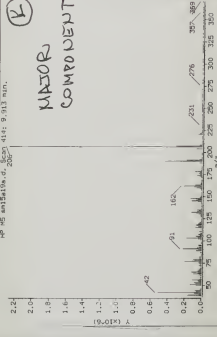


6 isomers



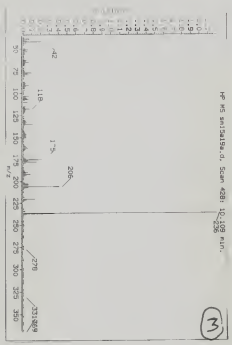
6 isomers

none of these have been reported in P. p.

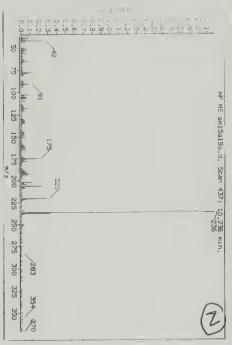


MAJOR COMPONENT

76



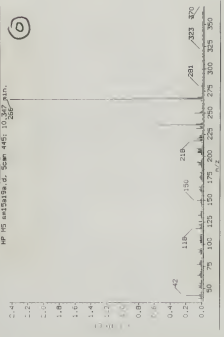
(1)



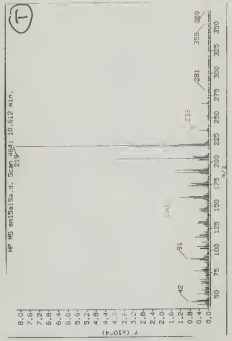
(2)



Toluene is a reported component of R.P.



MW 266



MW 266  
236  
266?



Wetberine is a  
valerianol component  
of P.p.

98



10g N-ethylamine - into  
20ml water - stir a long while

→ 14g water left  
some adsorbance ↓

3.6g T. add.

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

residual CH<sub>3</sub>I

Snake-vent graft - 15.154g.

water ~ 10 ml  
water ~ 10 ml

black oil = big crystals

ethanol  
N < 1g  
oil  
shift  
off

water ~ 10 ml water - no adsorbance  
water ~ 3 x 10 ml CH<sub>3</sub>I - stir, pp

white solids  
water ~ 10 ml  
white solids 3.40g

polymer  
under  
orange organic.

fish

→ 2.63g

diazo (2 grafts)  
grain water

grain HI = 330 mm.

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

Salt water wash a bit (100)

Snake skin - dry = 3.07g

0.3g  
0.3g  
0.3g

very little

P<sub>2</sub>O<sub>5</sub>

2.77g

tan graft  
graft

Snake graft  
let dry.

6.4g snake  
skin

grain under 2 x 5 ml  
CH<sub>3</sub>I

crop

-CH<sub>3</sub>OH.  
→

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

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

Filter - wash in boiling MeOH (another 20 ml). to  
slightly opalescent yellow brown solution, add  
1.0 g PSH. opalescence disappears. strip on RE. → 2.12 g  
vacuum oil. Δ in flame - milk w/ 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% MeOH to pH blue - slowly!! - xht in 3 x 20 ml  
ether - good - flask → amber oil 1.04 g.  
slight PSH smell. to KR.

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

- 1.04g
- 115°
- 2 mm
- no
- 145°
- 1.6 mm
- over
- 170°



5gamphetamine → into 25g MeOH Δ see Δ RT

5g 37% chro pH very green.

2.5g NaCO<sub>3</sub> - stirring - pale green pH → sh. green  
quite exothermic; Δ = running tho - add

50/50 conc tho/MeOH until pH ≈ yellow. IT does not

kn - still yellow! strip RE → reddish thin  
only sludge

9.6 g tryptamine (60 mm) into 100 ml MeOH add  
34g CH<sub>2</sub>I<sub>2</sub> (4 eq) (used 42g) - add  
40 ml 2.5% NaOH - onto 35 - reflux → solids, off at 4 hrs, ▽

filter → 52.55 g sludgy wet solids.  
dissolve in 100 ml boiling MeOH, ▽ twice → pale cream-colored  
solids  
filter, wash sparingly w/ cold MeOH - air dry  
→ 12.75 g around solids

12.75g into 100 ml MeOH  
+ 5.0 g AgNO<sub>3</sub> (3.2 I have, 2.0 form) →  
5 hr - stand a few days - filter - wash MeOH  
amber fluid -  
2.4g AgNO<sub>3</sub>  
into 40 ml MeOH  
+ NaOH ▽  
2.4g AgNO<sub>3</sub>

+ 5g φSH  
Flesh → 8.97 viscous amber oil. Be trying to dissolve  
into ether - to get into a KR flesh, bubbles, exothermic, and  
eventually 2 phases in ether! NOT spontaneous phase ↑ but  
surely acid & base smelly! ? 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 ↓

140  
over-

2.18g watery fluid  
φSH smell  
↙ 5.33 g amber  
fluid  
out

(82)

*Trichophan* → *trichemice*

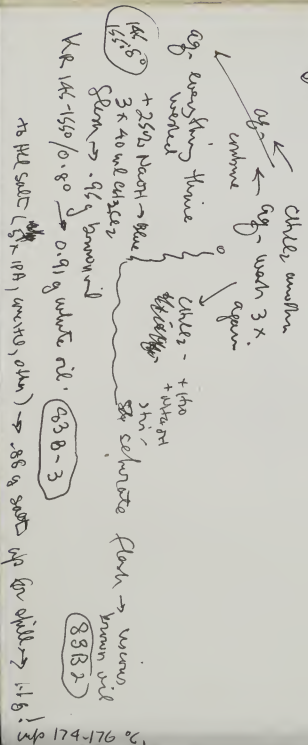
(See) SCS 3993 (1965) ; Russ. Chem. Soc. Japan II (21) (1962)

9:00  
dark  
start

2g dl-trichophan, into  
100g p20 - under H<sub>2</sub>O - Δ c wandle to trail (350°) signs  
of deceleration as time earlier - 9:00 PM at vent - all 4 legs  
totally seen. Pale yellow specimen.  
Let cool - incubate to ambient.

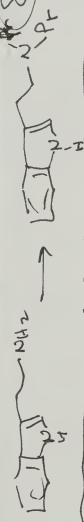
→ here from 83B.

eggs wash 3x chills



174-176 °C

83  
PT



(B)

with  $\text{N}^+$   
1.6g T (10mm)  
20 wt IPA =  
spill 20% (21.6 → 16.7)  
+ 4.1g n-propyl I  
+ 4.1g ( $\gamma$ -N)<sub>2</sub>N Et (4 fold)  
Stir 36 hrs RT  
Strip n.P.G. → ~~RT~~  
4.51g  
granular blank soil

(A)

with NaHCO<sub>3</sub>  
1.6g T (10mm)  
into 20 wt warm IPA A, ▽ clean sulfur  
5.1g n-propyl I (3 fold)  
+ 3.4g NaHCO<sub>3</sub> (4 fold)  
Stir RT 36 hrs.  
add 100 wt H<sub>2</sub>O → pH blue  
+ bleach oil  
xrt 3 x 50 wt chcl<sub>2</sub> - clean  
Solvent very clean - flash  
dichlor → 3.18g fluid brown  
oil - white smell. (83A I) <sup>emulsion</sup>  
Save a bit for master spec - The Fisher  
add 5g Ac<sub>2</sub>O. ▽ SB 10 min  
into 0.15N H<sub>2</sub>SO<sub>4</sub>. (~150 wt)

into chcl<sub>2</sub> + 100 wt H<sub>2</sub>O  
vented - OH 2.6N NDAI  
xrt → powder chcl<sub>2</sub>  
flash → 2.21g brown  
fluid oil

orig.  
xrt 3 x 40 wt chcl<sub>2</sub> → xrt 15  
Keep ag. <sup>Some bleach generated</sup>  
+ emulsion  
ag with 3 x 40 wt  
+ 150 wt H<sub>2</sub>O  
Set OUT  
chcl<sub>2</sub> + emulsion  
Set OUT  
+ 150 wt H<sub>2</sub>O  
Set OUT

83 B I

add 4 wt Ac<sub>2</sub>O.  
A SB 20 min - into chcl<sub>2</sub>  
+ 150 wt 5.2 N H<sub>2</sub>O  
xrt ag. 3 x chcl<sub>2</sub> - pool  
chcl<sub>2</sub> 2.15N H<sub>2</sub>O  
- Swells by Ac<sub>2</sub>O wash 2x pool  
into chcl<sub>2</sub> - add  
NDAI. stir -  
Sep. flash again  
→ crude amide  
fraction

83 A 2

into chcl<sub>2</sub> - flash → 0.41g  
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 0.20g

18 mm  
2.5 120°  
135° 500  
180° T up.  
Av. weight

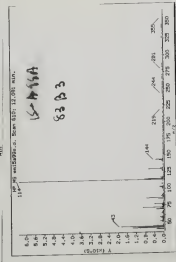
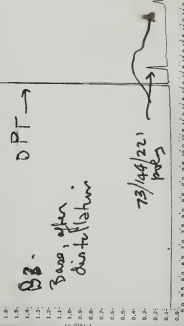
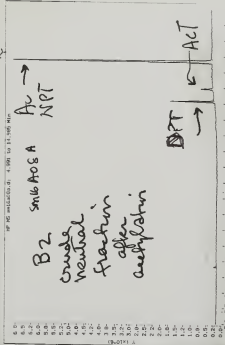
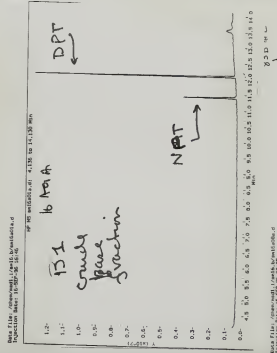
83 B



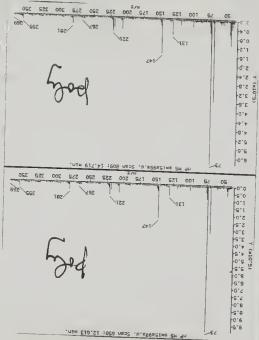
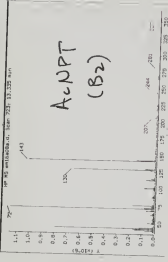
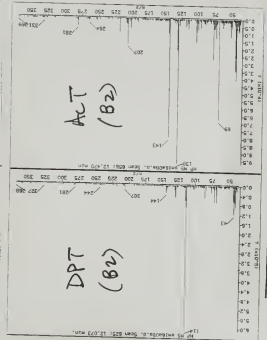
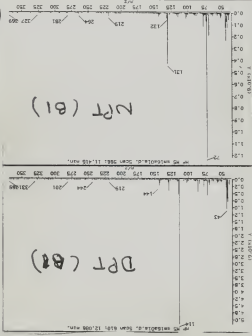


DPT (NPT)

P 183 B



183



501  
bay  
65

180  
180





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

87

1.6g Tryptamine base, into 20 mL hot IPA -  $\rightarrow$  RT, add

5.5 mL diisopropylamine <sup>ethyl</sup> add

3.0 mL (5.1g) iPrI. (30 mm). stir RT.

Sp. 1902

~~add~~ add 1.5 mL more base Friday evening.

strip on Sunday evening  $\rightarrow$  3.2g medium brown oil

add 5 mL Ac<sub>2</sub>O - 5 min.  $\Delta$  to RT.

add 3-5 mL conc. NITROH.  $\rightarrow$  hot - let cool to RT. 7.0 mL

AM - still acid !! - add goods NITROH - let stand 4x

next day - partition 0.5 N H<sub>2</sub>SO<sub>4</sub> (no, stronger!)  
w. 3x 40 CH<sub>2</sub>Cl<sub>2</sub>

Xtract ~~perle~~ perle CH<sub>2</sub>Cl<sub>2</sub>  $\hat{c}$  H<sub>2</sub>SO<sub>4</sub> vs 3x CH<sub>2</sub>Cl<sub>2</sub>  
40 mL

$\swarrow$  ag. prod.

base  $\hat{c}$  ~~NaOH~~ NaOH  
25%

xht 3x 40 mL CH<sub>2</sub>Cl<sub>2</sub> - flash  $\rightarrow$  0.20 g wgh.

KCl: 170-185°/0.05  $\rightarrow$  a couple of droplets of white oil  
slow & hot! OUT

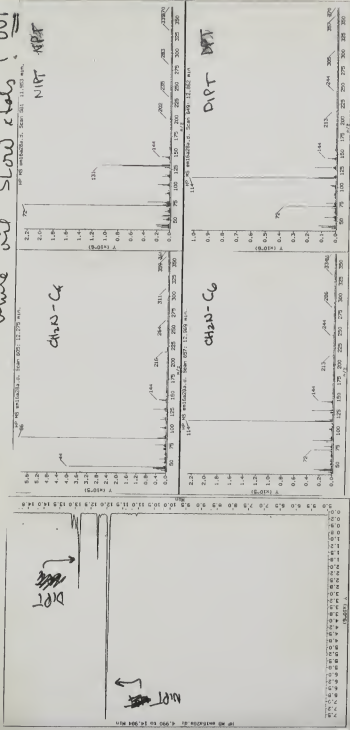
perle  
CH<sub>2</sub>Cl<sub>2</sub>  
stand

~~perle~~

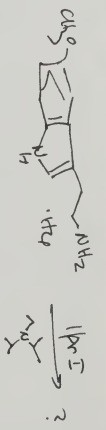
base  $\hat{c}$  ~~NaOH~~ NaOH  
25%

xht 3x 40 mL CH<sub>2</sub>Cl<sub>2</sub> - flash  $\rightarrow$  0.20 g wgh.

KCl: 170-185°/0.05  $\rightarrow$  a couple of droplets of white oil  
slow & hot! OUT



strip  
0.20



10g aniline + HCl MW 226.5 = 4.42mM  
 10 mL IPA

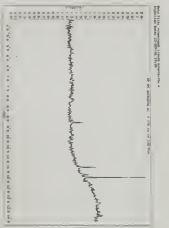
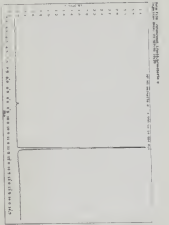
About Aniline MW 124  $\times 5 = "645" \text{ MW}$   
 $\times 4.42 \text{ mM} = 286 \text{ g}$   $\Delta = 742 = 38 \text{ mL}$   $\Delta$  to ethanol

1.33 mL inside  
 1PR I MW 170  $\times 3 = "510" \text{ MW}$   
 $\times 4.42 \text{ mM} = 226 \text{ g}$   $\Delta = 170 = 1.33 \text{ mL}$

stir for ~ 36 hrs RT - stream to surf out a white ppt -  
 filter solids 0.77g dry; benzoyl - only ML to

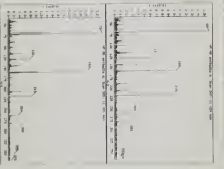
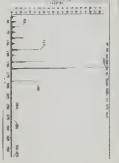
Ad 0.77  
 NL good  
 Swudge

MW 5.440 T (5 MWs g)  
 150 + 30 = 190  
 150 + 72 = 232  
 100 + 114 = 274



White solids (MW 10/1)

Swudge





90

push 88  
again



HP  
I  
?

0.77g white shade (p 88 - into two (S&E) + 600 ml water → pH > 5  
right is 3 x 40 we chills flash, add add p. 88  
sounding?

0.83g fine Det. 2mlp. (Snow x 100s farming)  
try again, will not ce

10 ml 10R  
4 we

1.33 ml iPRi use 1.5 we onto SB 36 km

strip to residue (same list for TLE, MS) (A)

+ 5 we Arzo - gets quite hot. did not put in SB. 10 min -  
5 we come later. hot 10 min. + 100g water -  
water hot - to home - hot 4 x 50 we chills part  
not parked under shadow

1 to 200 we. 500 this set work 2 x 40 w  
chills

1 again 200 we. 500 this set work 2 x 40  
chills

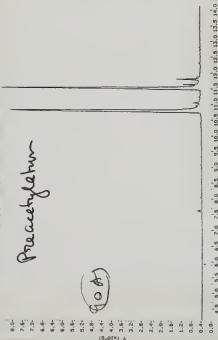
proof, home 2 600 water (Monday. we) hot 2 3 x 50 w  
chills  
Stack → 80g cruds  
KR 175-100 0.10 mm → 0.52 g off-white ash (B)

11.00.15.16  
17.00.15.16  
17.00.15.16  
17.00.15.16



100% DEUTERIUM OXIDE, 300 MHz, 100 °C

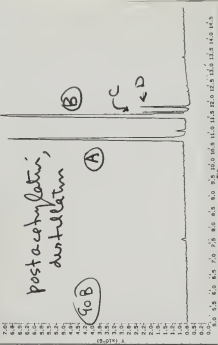
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100% DEUTERIUM OXIDE, 300 MHz, 100 °C

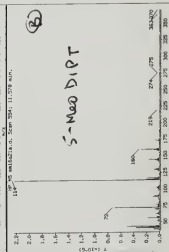
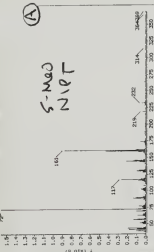
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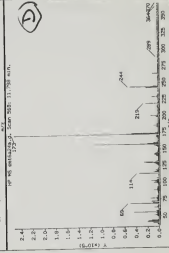
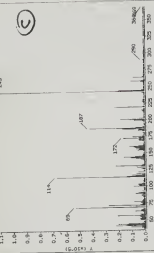
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911

100% DEUTERIUM OXIDE, 300 MHz, 100 °C



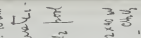
100% DEUTERIUM OXIDE, 300 MHz, 100 °C



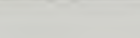
100% DEUTERIUM OXIDE, 300 MHz, 100 °C



100% DEUTERIUM OXIDE, 300 MHz, 100 °C



100% DEUTERIUM OXIDE, 300 MHz, 100 °C

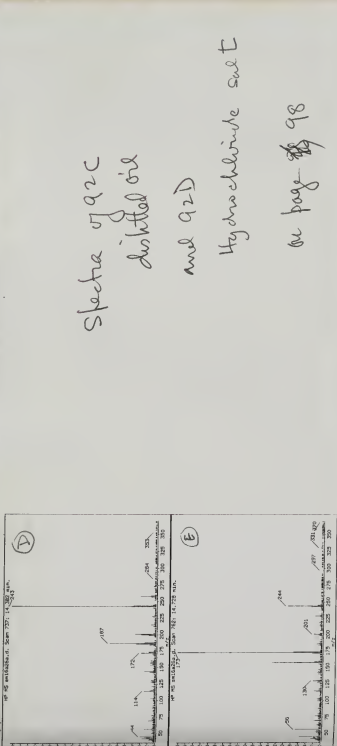
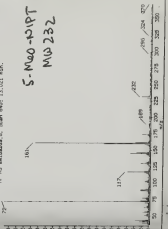
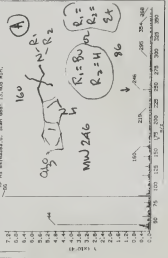
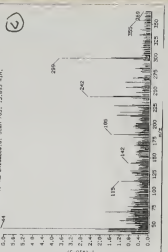
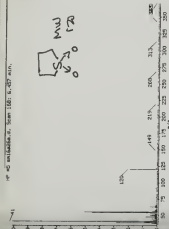
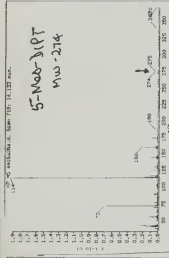
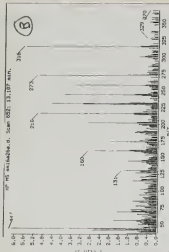
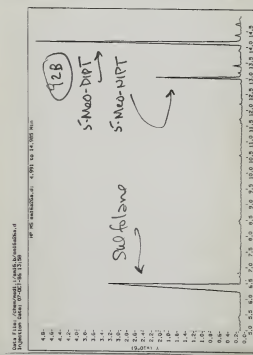
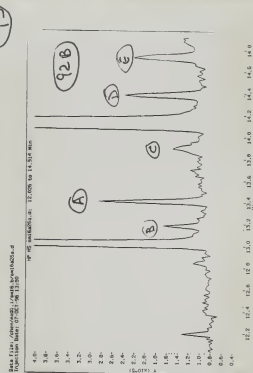


100% DEUTERIUM OXIDE, 300 MHz, 100 °C









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

92B

5-MAO-DIPT

5-MAO-NIPT

Sulfone

92B

5-MAO-DIPT

5-MAO-NIPT

MW 170

92B

5-MAO-DIPT

5-MAO-NIPT

MW 246

92B

92C

92D

92E

92F

92G

92H

92I

92J

92K

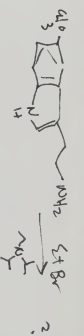
92L

92M

92N

92O

91k



0.95 g  $\text{C}_{10}\text{H}_{18}\text{O}$  - 5.8 mmol. freeze base TCI - benzene sol. - looking stiffer into  
 10 mL 10%  $\Delta$  m. SB in benzene  $\gamma$  RT. add

+ 2.5 mL  $\text{Me}_2\text{C}=\text{CH}_2$  (2.41 = 15 mmol  $\Delta$  d. 742 mmol (29))

+ 1.2 mL (used 1.3) STBr (1.18 = 15 mmol  $\Delta$  = 146 mmol (90))

stir RT 30m. wash.

36 hrs - add more than stir

add more STBr

more than 36 hrs.

stir  $\rightarrow$  (91k) 3.4g same dec. - 2 phases!

add 1.6g Me<sub>2</sub>C=CH<sub>2</sub> (5.1 mmol) over SB. no more. into beaker -  $\rightarrow$  3 mL Me<sub>2</sub>C=CH<sub>2</sub>.

+ 100 mL 0.5% 1H<sub>2</sub>O<sub>2</sub> (11% O<sub>2</sub>) stir 3 x 50 mL cycles

3 x 50 mL cycles x 100 mL 1H<sub>2</sub>O<sub>2</sub>

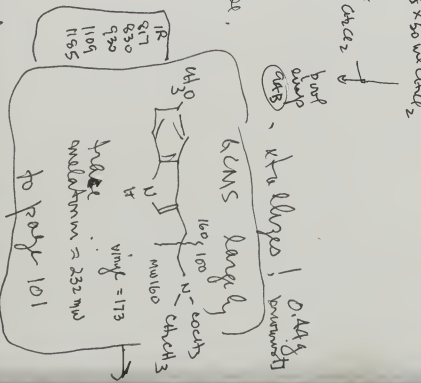
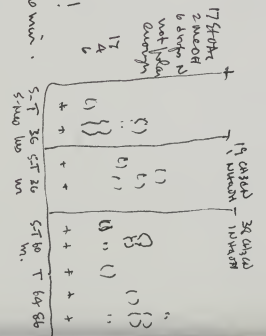
point down (91k) - what happens! D. 4.4g

0.95 g  $\text{C}_{10}\text{H}_{18}\text{O}$  (10.0 mmol) (leaves) flash  
 x 1.3  $\rightarrow$  15 mL water, 0.80 g starch, 0.5 mL 80% Me  
 100% Me 0.45 g white oil  
 100% Me  
 No. 200 yps.

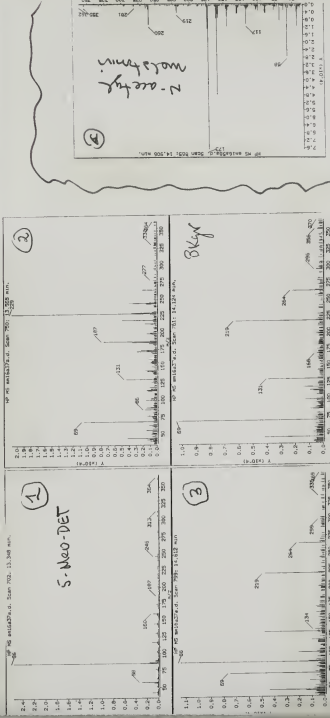
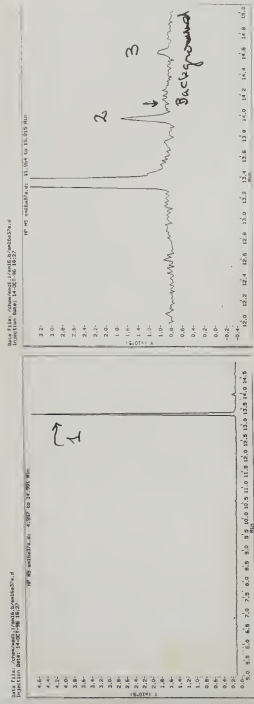
all into 2.5 mL 10% RT + cone like (low temp) dispense in low temp

stir 40 min. slowly to find out 30 mL to ask then

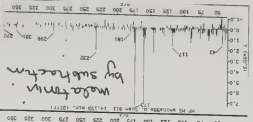
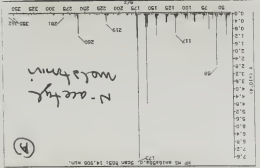
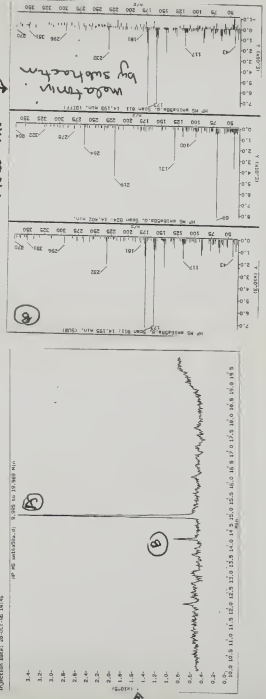
$\rightarrow$  0.52g beautiful white x 1.5. col. 0.50g 34%



56



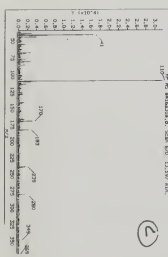
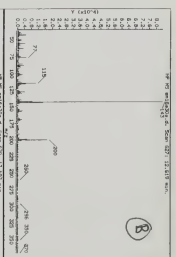
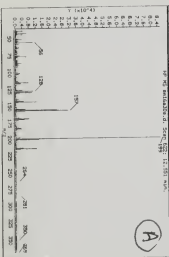
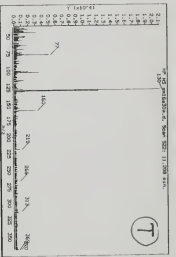
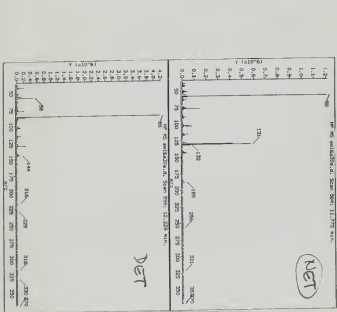
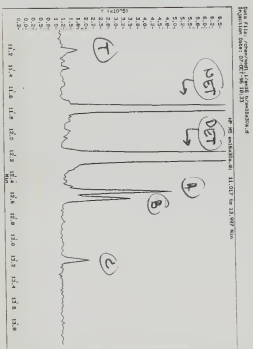
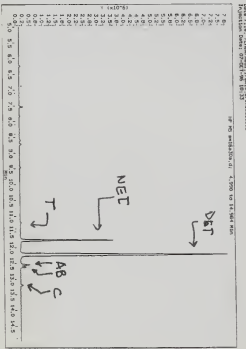
← w-eg. melatonin  
→ melatonin



Handwritten notes and arrows at the bottom of the page, including 'B', '1173', and other markings.

96

Page 86 - proactin tablet



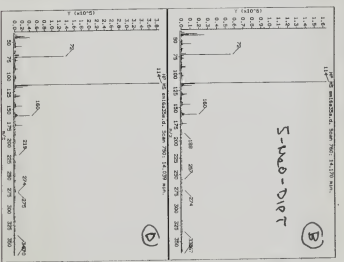
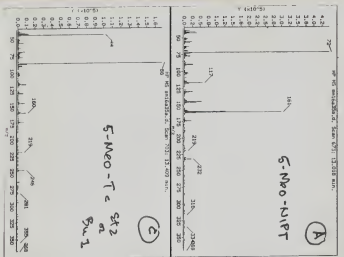
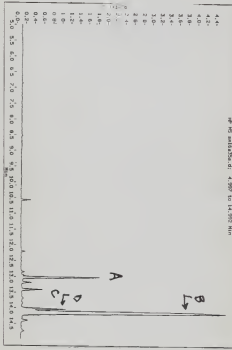




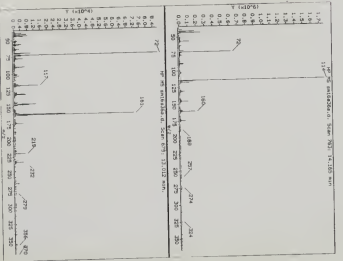
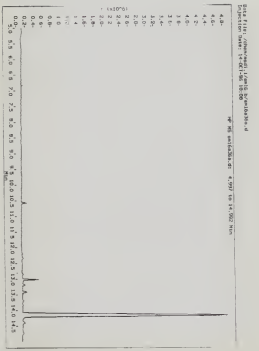


98.

Sample of pure 5-MeO-MeO-DIPT p 423



distilled oil  
 ↓  
 HCl



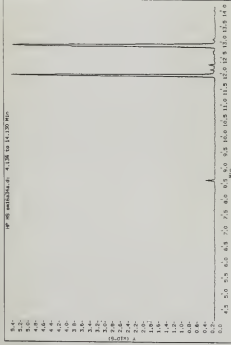
MS  
 1000  
 1000

Work-up of page 89 - distilled, Acidified DIPT

(99)

Mass 89: compound 1 (99) 8.00000000

Retention time: 1.00000000

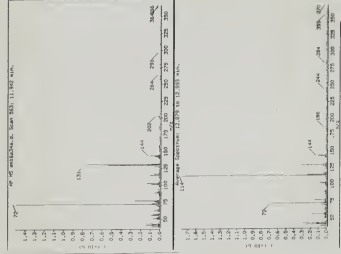


page 89  
 LR'd out  
 → xthls.  
 0.9 g

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

decant  
 into  
 pale yellow  
 soln  
 evap → xthls  
 0.13 g  
 (89E)

Small amt of (H) possibly!  
 Small amt of MeOH - too sl.  
 pentamer  
 MeOH / hexane



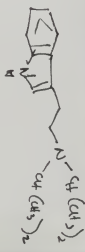
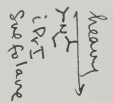
wash  
 sin out  
 hexane  
 0.02 g  
 (89F)

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

1.000

1.000

100



10 ml - was for ring conditions

1.6 g triethylamine

10 g washed sulfonamide (not all dimers)

6.5 g diisopropyl ether

8.5 g isopropyl alcohol 2 phases - shake occasionally

into SB.

12 hrs ester - reaction still 2 phases - off into 100 ml -  
pH is blue, xrt 3 x 30 ml hexane, (and wash 100 ml  
water - wash on R.E.)

2.67 g deep brownish fluid oil  
OH → xrt washes oil -  
because

1.034  
cryst  
white  
dmy.

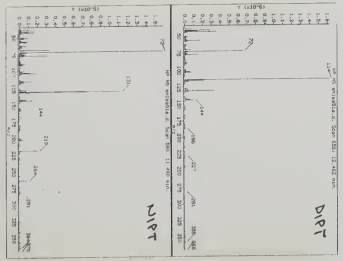
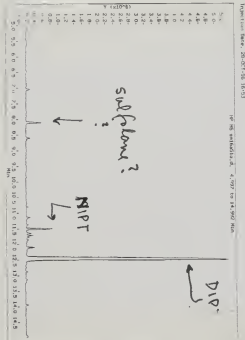
1.37 g 100-70  
0.05 m

mp 69-70  
off white

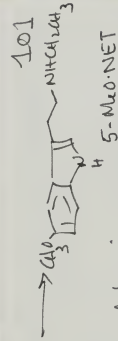
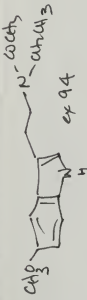
reptology from hexane  
what's the MS gonna be  
C<sub>10</sub>H<sub>15</sub>N - free or salt, NPT probably!

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

Smudges of fluid for  
C<sub>10</sub>H<sub>15</sub>N (solid in vial!)  
good spectrum - integrate  
by peak weight = 95.6%  
small amt NPT -  
trace sulfonamide, water not  
mp 72-74



100  
90  
80  
70  
60  
50  
40  
30  
20  
10  
0



0.44 g unfully good crude N-St. indatomin  
 from page 94

ground under a few ml MeOH → almost white solids - look OK  
 a few milligrams from MeOH → white x-beds.

same for mp  
 rest 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 100 ml CH<sub>2</sub>Cl<sub>2</sub> - (pale-  
 x 100 ml CH<sub>2</sub>Cl<sub>2</sub> - 3 x 50 ml 1 N H<sub>2</sub>SO<sub>4</sub>

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

flashes

→ 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, 1031, 1068  
 1108, 1182, 3119.

work in CH<sub>2</sub>Cl<sub>2</sub>  
 OH - 25% NaOH  
 x 100 ml CH<sub>2</sub>Cl<sub>2</sub>  
 flash  
 → 0.05 g

102

attack



DOWN SCALE

3.2g tryptamine base, into 40 mL IPA,  $\Delta$  to dissolve, add

7.7g dicyclopentylamine 65 mL, add

9.3g ICH<sub>2</sub> 30 mL -  $\Delta$  stir overnight (RB, reflux.)

Am - some darkened, solids in bottom,  $\nabla$  RT  $\rightarrow$  solids  
decant liquid  
solids not solids

$\rightarrow$  25g drying dark

$\leftarrow$  flash  
1.48g trim solids  
dry w/ 50% hexane  
drying in the

into 100 mL H<sub>2</sub>O (neutral)

+ 25% NaOH - xHT  $\bar{c}$  3 x 50 mL

CH<sub>2</sub>Cl<sub>2</sub> - part - xHT  $\bar{c}$  3 x 75 mL

1.2. H<sub>2</sub>O<sub>2</sub>  $\rightarrow$  color in rxn of -

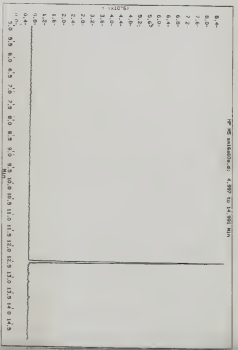
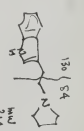
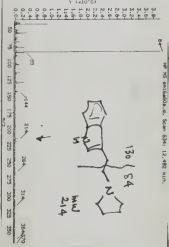
water mix with CH<sub>2</sub>Cl<sub>2</sub>

RT  $\rightarrow$  to pH  $>$  9, xHT 3 x 25 mL

check - clean  $\rightarrow$  0.25g -

KR 0.05 mm 170-180  $\rightarrow$  white

oil react xHT layers - cems  $\rightarrow$   
52 mg



0.25g  
0.28g  
DS mm  
4.710  
195  
shd 170



103

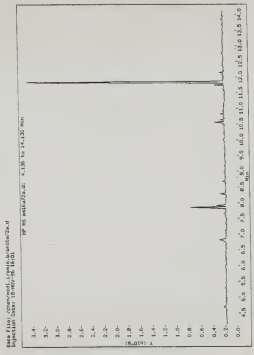
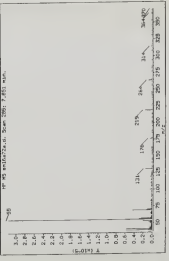
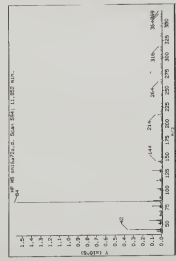


1.4g tryptamine base  
3.6g ICH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>I (10% XS)  
2.6g CC(=O)O

Stir RT - 15 hr work up - there is a 2-phenyl character to it all - into 100 ml H<sub>2</sub>O (is basic) and x 1/2 = 30 ml hexane. (There are globs not soluble in either phase). Strip hexane → 0.25 g sticky white

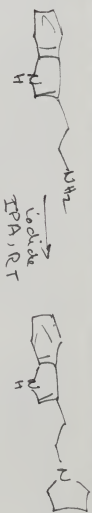
into circles for distil - strip down hard  
↳ 0.05 g - distil → trace product

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





104



1.6g Naphthamine base into 20 mL IPA &  $\Delta$   
 3.6g Dichloroac<sub>2</sub>E  
 2.8g  $\text{NH}_4^+$  shi RT a few days. solvent evaporated  
 stored a couple of weeks.

104A

(1) 9g. Et<sub>2</sub>N + acetone  
 from ft. tin.

hexane  $\rightarrow$  ~1.1g

(2) 10g Et<sub>2</sub>N + acetone  
 Feltin, stink  
 $\rightarrow$  1.47g. ground in tin

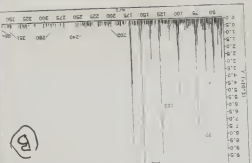
hexane  
 Rock. oil  
 under ch<sub>2</sub> - skin  
 drink.

~1600  
 -0.4 m m  
 130 mVg

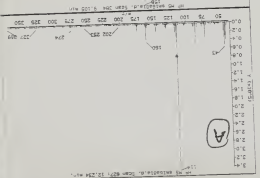
I forgot I used stinked skin

3.2g T<sub>30</sub> wa IPA - not all in skin  
 9.5g under  
 9.9g ICA I shi RT  
 8 PM

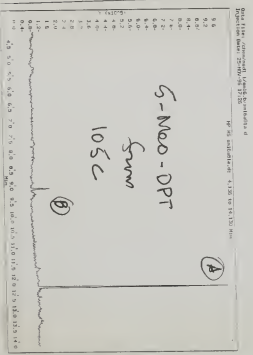
104B



104A



104B



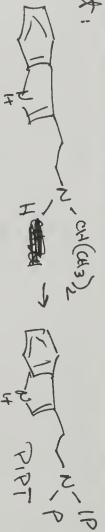
104C

5-Meo-DPT  
 sum  
 105c



106 (attempt):

MW	NPT	202
RT		170
RT		129
RT		238



0.50 g NPT into 5 mL solvent - Δ SB to benzene -

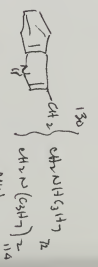
add 0.8 g 6N NaOH - then

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

3.4 g (should be 1.7 g (x2) but some RTI way - get ~~down~~ cleaned up  $\bar{\sigma}$  the  $\text{KOH}$

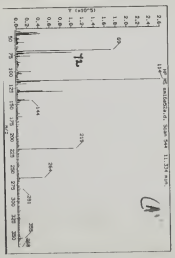
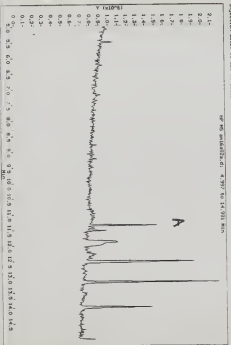
Outo SB worn - off 5:30 PM - ship  $\rightarrow$  black powder -  
 add 100 mL H<sub>2</sub>O - second run - it should have been  
 absent - add 50 mL MeOH as well - now it is kinda  
 in solution. that 3X 30 we became - Pk<sub>2</sub>

$\rightarrow$  Peak at 106 Å  
 0.14  
 1st fraction, 0.10



(MS - it is more  
 sensitivity of MS can find

130° large no weight. OUT -  
 0.05 min  
 150-160° (phenolacetate)  
 0.05 min H<sub>2</sub>  
 0.01 g - one droplet - saw Smudger  
 for CIMS  
 PIRT. 106



AQ2A

separate

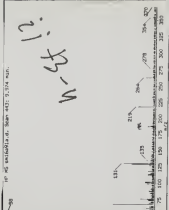
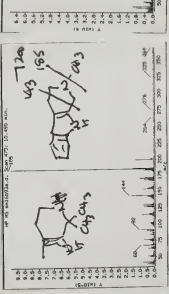
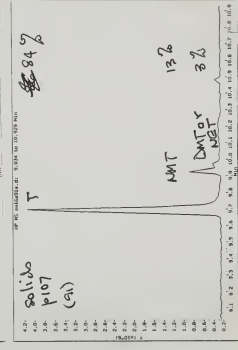
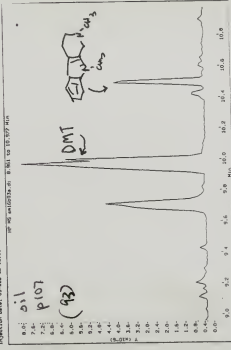


see 66

4.34 suspended in ~10 ml ether

SOLIDS which 1.72g  
 TIC largely T.  
 OIL 1.52g  
 both T, M<sub>2</sub>T  
 other stuff. 1.82  
 (3 samples  
 average  
 x 1.465)

dissolve in 10 ml IPA. conc. ites to red ppt paper. scratch  
 studs (washed  
 w/ ether)



0.45g white solids  
 + no white solids  
 we either turbid.  
 scratch - finally  
 xtals.  
 0.19 g almost  
 of white solids  
 Lots of ether -> oil  
 decant from  
 IPA ether  
 decanting  
 oil. residue  
 (signs of fine  
 xtals on  
 standing)

NO peak -1%

108

24 liter Nix materials from p 92



rotated fraction: skip head 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~~  
0.23 g (Substance?)

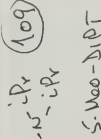
exlone benzyly  
same data for CCNS (108 k)  
wada for nmrts.

is subfrance  
no higher unives  
but MS is not with  
murd nmrts. with

but - out



Repeat.



3.0 g 5-MeO-T into

20 ml sulfone. Δ m sb → clear soln. ▽ RT. add

8.2 g  $\rightarrow$  4 equivalents. add

10.7 g solens PPI - 4 eqs. Δ SB ~ 3 ~~eqs~~ hrs - SLO

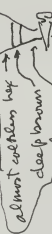
2 phases - stir at RT 16 hrs. Strip to find

thick residue on R.E. (30g). add 100 ml THF →

clear solution !! maybe a wee bit basic? add

a few ml 5% NaOH → very cloudy

Partition between aq. base & 3 x 4 ml hexane.



2  $\frac{1}{2}$  & 3 vol extract - more

solens hexane could be gusted out yellow

of the deep brown inter face

clear hexane xtds pooled - stripped → 176 g almost

white oil - stinks of amine.

Transfer to 14/20 RT - a bit of eth<sub>2</sub> - really stink on

R.E. → 0.99 g pale cream-colored oil - slight amine

smell. To K.R. fogging over at 100° (0.01 min!)   
 ↳ trace (109A)   
 (very faint (109B))

beautiful white oil over   
 at 140-150° (109B)

0.80 g

no residue!

dissolve into 3.5 ml IPA

done like (KRS) 10 ml 15 ml.

no xtds. add 5 drops ether - xtds start V - another 10 drops

scratch

10 minutes filter - wash 2 ~~4~~ 1 IPA/ether - air dry

beautiful xtds white (109C)

0.95 g.

mp 191-192°

MS 114 ~~100%~~ 100% CH<sub>2</sub>N-14

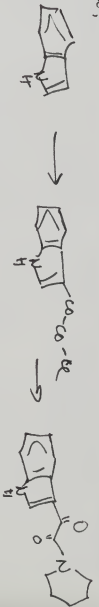
30 to 72 ~~100%~~ 100% CH<sub>2</sub>N-14

100% ~~100%~~ 100% CH<sub>2</sub>N-14

ML 5 OVER   
 109D



(110) ethylene;



transfers  
PIP-T

1.0 g indole 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  $\bar{c}$  cold TBME. - add,  $\bar{c}$  stirring,  
about at a time, to

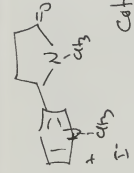
2.0 ml pyridine neat. Add xs 2.0 N HCl  $\rightarrow$   
satur. - filter. air dry. 1.98 g air dry

reprecipitate f. 7 ml hot neat, filter, wash lightly,  
air dry  $\rightarrow$  1.01 g white xbrs. mp 182-183°C  
mp 175-178°C

(111)



col.



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

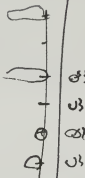
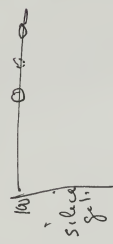
half to vial

half still in flask

into 1/2 ml tho xrt 2 x Et<sub>2</sub>O

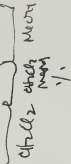
flash eq.

↳ dark only spots.



(A)

quat.



1/1

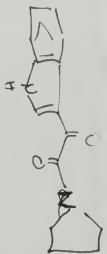
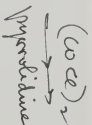
MeOH + 10% D<sub>2</sub>O

no diffcult.

quat immiscibly

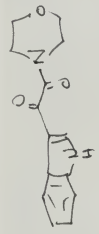
↳ bottle to silica

110



A solution of 1.0 g indole base in 15 ml TBME  
 is stirred well. There is added, dropwise, a  
 solution of 1.1 g ceasocce (Kraus (1.17 g) over 20 minutes  
 (solid appears at 1/2 reaction point)) stir an  
 add in 10 ~~min~~ min. Filter  $\rightarrow$  yellow xrbds - work " "  
 spinning  $\bar{c}$  RT TBME - add as a solid, a bit more  
 at a time to stir 2.1 g pyridine reat.  
 Benzoin,  $\rightarrow$  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 anhydrous at 1.2 g - try 10  
 seconds for xrbds - too insoluble about 1/4 TBME  
 toluene, strike out. CH<sub>3</sub>CO Perfect.  
 Dissolve all in 30 ml boiling CH<sub>3</sub>CO - slow to  
 evaporate  $\bar{c}$  RT white xrbds - color in ml ! (wet)  
 slightly off white, fine xrbds, filter, wash  
 lighter  $\bar{c}$  CH<sub>3</sub>CO - air dry white xrbds dry weight

0.87 g  
 wmp 219-220



(except  
 → morpholine



≡ to page 112 down to "new"

none added to 2.0 ml morpholine - stirred - exothermic - but stays large white. let cool. MeOH added 80 up 1 N tce. filter - wash - this - removes, ground w/ the chunks under fresh water filter → white solids. 1.55g wet but partly ~~air-dry~~. 0.95 when completely dry.  
 pale pink.

reconstitute from 4 ml MeOH at the base - filter - wash extremely sparingly with MeOH - air dry to 0.75  
 in first we get - white 0.75g - crude mp 183-186  
 4 MeOH → mp 187-188°

of 4ml





MET

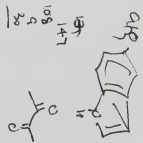
Tryptamine, N-ethyl-N-methyl  
Indole, 3-[2-(ethylmethylamino)ethyl]  
N-Ethyl-N-methyltryptamine  
3-[2-(ethylmethylamino)ethyl]indole

See under MPT

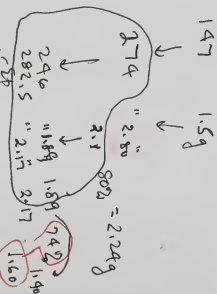
**SYNTHESIS:** Under an inert atmosphere, a solution of 7.0 g N-formyltryptamine (see preparation under the recipe for N-methyltryptamine, NMT) in 50 mL anhydrous THF was added to a well stirred mixture of 7.0 g LAH in 100 mL THF. This was brought to reflux temperature, and held there for 3 h. There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

drop-wise  
stir

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 = 2.114



1.60  
 96  
 246  
 0.43g 145  
 36.5  
 2.5 mL 8 mL 282.5  
 1.1x

246 0.45  
 282.5 51.6  
 " "

50g

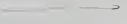


0.95  
 282.5 " " 1.41g  
 0.50g z

Fig

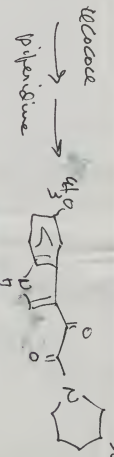
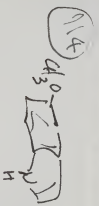


145



5.516

Fig



A solution of 125g 5-methyl-2-oxobicyclo[2.2.1]heptane in 15 ml TBME

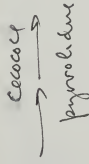
was stirred, and a solution of 11g oxalyldiamide in 15 ml TBME added over 10 min, filter. - work with a little TBME - done here

10 min, filter. - work with a little TBME - done here  
piperidine - stirred - exo Norm & fumes, or some.  
End up adding 1 ml more piperidine to get everything wet & cool triethylamine. to RT + 80 ml 100 the -  
quantity - stain for quite a while - sig not quite -  
get with a few washes off by keeping solution - ground ~~with~~  
under went in 2 ml? solution - filter

aground, filter  
→ 0.01g pink solid out  
wp 158- → 0.65g totally dry  
158  
neat f, 2 g neat  
0.82g vacuum dist  
in hot.  
MS (solid) v.  
→ 0.37 pale  
wp cream  
158- fine x 100  
158

115 continued - filter → wet  
medium colored solids - dry in SF<sub>6</sub> → 1.13g  
brown colored powder  
sample for mp 115 B scale. mp 158-200 (195 area).

Recrystallizing from 15 ml boiling MeOH (decolor)  
from a wet bit of (washed side) clear hot - ▽ RT → solid -  
filter → white solid, x two - another ML  
↓ air dry. pale cream : 75 wet - MeOH.  
65 dry -  
washed - wash i  
acetone - air dry  
158-158. mp 210-214  
115's derivatives → mp 210-214  
reformed 1  
workable



115

Identical to "hove"

deep tomato color  
deeper than 114  
30 min +  
15 min

"hove"  
make a mix 2.1g pyrrolidine & 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. into 2.5g (2 1/2 ml) MeOH - Δ 15B → solids - to it → white solids. filter - .36g product.

59  
wet

58  
wet

brown product 0.16g. xtd a bit from MeOH - not good  
brown to gum sets up solid 0.15g  
brown to olive green solid (well, very) OUT all.

48  
wet

Repeat - drop imitations same scale 125 inside (5 ml TME) 1.1g  
oxalyl chloride (15 ml) - dropping over 20 min - 10 min stir - increase  
wet dark tomato solids. filter. brief air dry - add dab by dab to  
2.0 ml wet by uridine. This time washed all the color into the  
the vial wet - all finally decolorizes to cream color - and  
it sets up to a sorta solid (15 min) y rub - keep homo-  
genous - good portions - add 20 ml 1:1 H<sub>2</sub>O → nice  
solids - rub the chunks in a spatula until all fine

(116)



cecroic acid  
 for work function



MW 259

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 cecroic in 15 ml TBME over 20 min - solids  
 at about 5 min. Really over thin pencil, and get a  
 normal tomato skin - not dark tomato. Stir and run  
 10 min - filter, wash with TBME, as dry (usually) & add  
 back into 2.0 ml neat methylene. Take time making  
 it all homogeneous (15 min) until all tomato is  
 dispersed in the oil, pencil - becomes dark reddish  
 brown. Then add 80 ml 1N HCl → solids - ground  
 up the chunks - no gum! - filter, wash with  
 onto SB at 100° → dry powder - rest red - 1.35g  
 Almost ant. - out of meat OK. cntly.

ALL 1.35 g aside in 10 ml MeOH Δ SB → clean  
 blend red solution - ▽ → xkals. work sparingly  
 MeOH → white - blend red ML

splish -

0.85g - very weak prod.

frags of pink color mp 193-194°

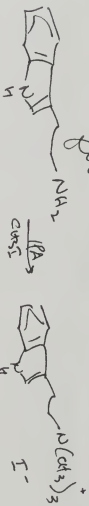
→ get acetone





(118)

ethylene IPA as great solvent.



based on recommendations in schedule 1PA work page 69.

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

at RT. add (stirring) 10 g HCl. - no evolution. 4 PM  
Thinning layer. First wash in 10 min. no over-thinner - 1 hr -  
heavy of solids (shiner stopped - too thick)

mp  
as in 175°  
In PM. - filter → cream colored solids - 2-72g  
works in IPA  
with

dry 1.81 g

xtal tho 203-4°  
wash with IPA 192-4  
xtals clear 210-11

into Toluene

Refract 115 - Surfer

Refract 115 - we

1.25 wet

1.05 cecococ (mats, parts) (fresh)

2.0 ml pyridoline

differences, fresh cecococ, from Actos.

3.42 wet

3 ml acetone

1.42

(wet)

97 air dry.

96 ← 213 deak

dry: mix

cell in P. salt - H<sub>2</sub>O

119

15 MBE

15 MBE

15 MBE

15 MBE

15 MBE

15 MBE

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

Add (scatter, not bunch-wise) to 2.0 ml

pyridoline. The few dark spots rubbed in

5 spatula → dark cream viscous oil. Add

some 12 H<sub>2</sub>O → off-white solids - grind w/

chunks, filter, wash 5 H<sub>2</sub>O → 3.42 g wet solids

grind under 3 ml acetone to almost white paste - it seems

to form as I work with it. Filter - wash - very small

aut. acetone. → 1.42 wet. ON air dry 0.97 g

0.94 g  
47%  
Minerals

0.94 g dry

MP 212-213°.

0.69 g dry

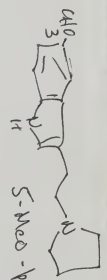
0.52 g wet

p 20

0.94 g into 12 ml boiling Et<sub>2</sub>O - clear to RT  
let stand ~ 1 hr. filter, wash sparingly & checked → 0.72 g wet

(120)

Repeating page 117:



5-MeO-Py-T

25 ml 10 L in THF stir, under Ar.

Δ to boil add

151  
- 1.22 water  
4x

0.67 g (19 dia carbonyl in 25 ml dry THF

add in ~ warm. reflux 20 min. VET - add cat 50/50

THF with water, then 50% DIET until cloudy. (1.6 hrs

wash c 50 50 THF. strip - into 100 (basic oil)

Ykt c ketone (60 mg) xht c ether → 0.37 g - combine

dirty  
↳ 110 mg unknown pale amber oil

MS see below

60 mg  
ketone  
strip

ether  
0.37 g

Crystalline

0.50 mm  
100 g  
100 g  
100 g

cool extending  
(170) 0.20 x ml

110 mg water

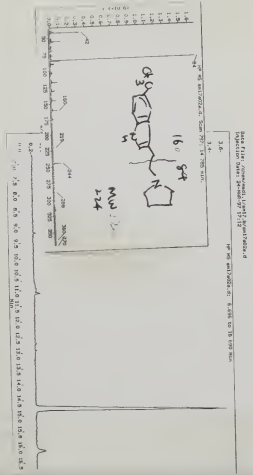
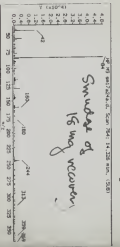
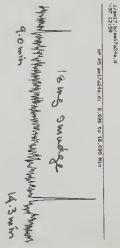
into 10 ml IPA

100 (2 sample)

ether to oil.

strip → black oil

try to make 100% - weak oil - stand sunset  
 morning wash - into 1 ml HO (not completely clear)  
 + 5% DIET → very cloudy - xht ether  
 flash → 30 mg - try KR 0.05 mm/ht. 140 mm  
 take to 220° → 18 mg film



See 109



(121)

2.07g S-NEOT

.15 ml softflame Δ SB → sol ▽

5.5 g  $\rightarrow$  mix

7.2g iPrI (quf'd, used 7.9g, saving)

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

28.5 thick, dark amber

+ 75 ml hex → 1 phase.

XHT 2 x 40 ml 20-60 jet ether - back wash @ 40 ml (H<sub>2</sub>O)

Here 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 softflame

(121A)

jet ether sharp hard

1.47g pale amber oil - amine smell.

0.04 min

120 blush.

145 over steam heat, 1 hr, FB

160 done

1.33g

6 ml IPA

the 22 was 25 yrs #1

starts at 20 already starting.

add 4 ml ether to air fractionator.

wash @ 3/12 IPA/ether

check IR - to impurities add min's to 109 #D

bin dry 1.34g



(122)

Odds & ends.

5-Med-DMT.

Free base - not for <sup>100°C</sup> vacuum left hand

→ water (caution) 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°C? E. Flakes on lab bench in warmth or two,  
underwater. - lots of solids - red - and so wet & ground, flaked,  
strong smell of amine strip → 2.3g redish vial.  
Scrape out solids → 8.1g  
3.47g  
grind under H<sub>2</sub>O, crystals - self crystals

Second crystals lost  
wash & dry N<sub>2</sub>O

Slush crystals, → 3.47g

Flund ~~+~~ + gums. - work & check  
← (wet wt)

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

50-70 lots of white. Dry grind over.  
0.04mm. stop at 90 change flasks.

0.60g  
weighed.

on - 135° wa

by 170°

Some orange  
stuff over -  
not in box.

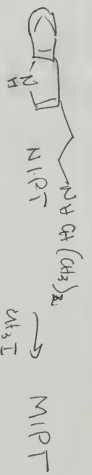
'junkie!'

OUT





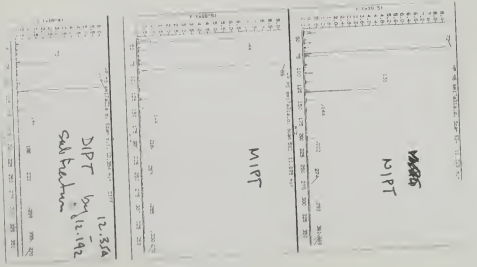
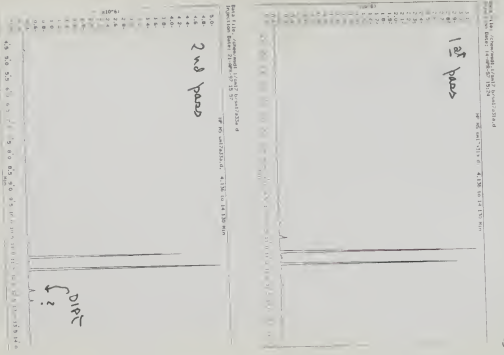
124



0.41 g (2 mM) NIPPT. The  $\text{wt} \approx 50 \text{ mL}$  has,  $52 \text{ mg NIPPT}$  -  
 $\text{XRT} \approx 2 \text{ CH}_2$  (3x40 mL)  $\text{R} \rightarrow$   
 $\text{R} \rightarrow 0.43 \text{ g}$  in  $\text{CH}_2$

$\text{wt} \approx 25 \text{ mL}$  in  $1 \text{ PA}$ .  
 $+ 0.35 \text{ g CH}_2\text{I}$  ( $252 \times 5$ )  $0.28 = \Delta \text{ SR} \approx 3 \text{ hrs}$   
 $\rightarrow \text{dry!} +$   
 TLC same NIPPT -  $2 \times 15 \text{ a}$  NIPPT -  $\text{wt} \approx 10 \text{ mL}$   $1 \text{ PA}$  - +  
 $0.14 \text{ g CH}_2\text{I}$  ( $252 \times 5 \rightarrow 752 \times 5$ ) -  $\Delta 5^\circ$  -  $2 \text{ at strands}$

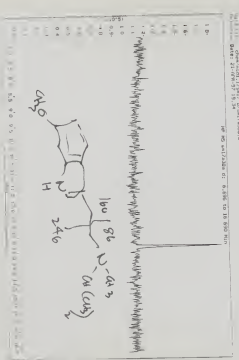
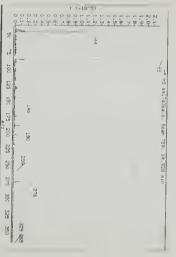
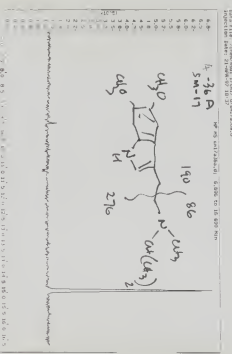
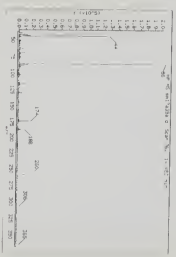
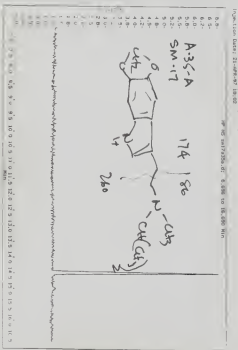
$11.29 \text{ oil}$ ,  $25 \text{ mL PA}$   $0.35 \text{ g CH}_2\text{I}$





(26)

Spectra from here of this





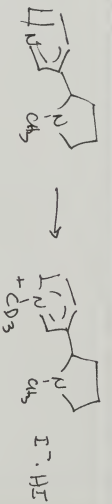






129

130



Wegius.

mixture 162  
 .4HI 290  
 .2HI 418  
 Cotinine 176  
 CD<sub>3</sub>I 145

Fyler **CT**  
 Dec 19, 1979  
 p 70

2.5 g mixture - 30 ml aq. STOH  
 (has ~ 8 g 50% HI)

at ca. 7 g pretty red - add the rest anyway  
 + 20 ml ether, stop, ether brack & for th.

on to EG - hard evaporate  $\rightarrow$  yellow solids.  
 6.48 g yellow solids 130A

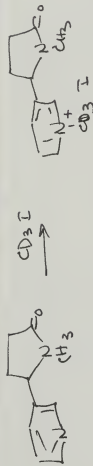


3.58 g Nic. 2HI  
 1.38 g mixture in } onto reflux - 2 hrs ;  $\nabla$   $\rightarrow$  xhls  
 30 ml STOH  
 1.21 g CD<sub>3</sub>I

filter  $\rightarrow$  3.30 g yellow solids  
 (stand a week, minor quantity to 3.5%)  $\rightarrow$  3.18g

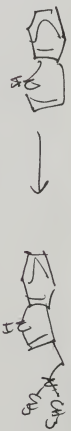
great  
 Nic CD<sub>3</sub><sup>+</sup> I<sup>-</sup> .4HI 130B

131



0.85 g Cofinine - info

1.5 mL MeOH



To 20 g ag. 10H H<sub>2</sub>O H<sub>2</sub> H<sub>2</sub>O (sat soln - K<sub>2</sub>SO<sub>4</sub> + starch) 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 etols - skin (stick to evaporative -  
 skin oil. - in km bump into)

40 g KM in 300 ml H<sub>2</sub>O.

→ yellow foam - GENS of ~~some~~ gym  
 mostly indole + glaucine  
 a bit skatole in this - warrick -  
 a lot later in warrick (1813) -

stand a day → fine white water ← same seed → return get

decant ag → heavy yellow sedimenting oil (sour seed) - wts 100 wt

etcher - separate, xft ag. = etcher

prod. xft 1x200

1x100 ml 10 H<sub>2</sub>O

wash etcher (steel drum)

etcher yellow  
 ag. w/etcher

wash base = 25% base → pH ↑ 10, solids!  
 cloudy! -

xft 3 x 75 ml etcher part fresh

→ 7.7 g almost white solid

skilled some getting 400 away from product etcher's  
 washes white solids looking at "normal yft"

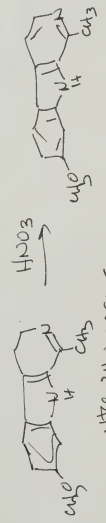
yft a lot of etcher washed  
 all the SO<sub>2</sub> in some

etcher in some  
 water separate stuck in car-bath from oil funnel

yft ~ 11 g.  
 part of from before - bearing white w/ 131-132

(Synthesis, at about (fine) 112-114) (old 5. NADOM 58-61)

133



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

- 0.5g bariumline. 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

Dist 2.12

to this, add

1 S.B. - almost lost - bubble bubble bubble! -  
 30 seconds - off -  $\nabla$   $\bar{c}$  add water  $\rightarrow$  x'tals stand  
 better - work 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 253° dec?

133B

134

11 to purg 400



100 mg P<sub>2</sub>O<sub>5</sub> = 400 mg

into 25 ml the solvent clean

white crystals

add (under nitrogen)

100 mg P<sub>2</sub>O<sub>5</sub> - 500 mg

400 mg P<sub>2</sub>O<sub>5</sub> in 400 ml (3.6 ml) a few drops of a few

20 minutes

P<sub>2</sub>O<sub>5</sub> looks almost colorless (P<sub>2</sub>O<sub>5</sub> in water)

Filter through paper → pale yellow filtrate - water. 1 hr - water bath 2 hours → P<sub>2</sub> white crystals.

MT = 3x50 ml ether

15% white solids x 2 hours

2,3 ml 50.

Flask in P.E. → 0.58 g white crystals

Sample not

Flask residue = 10 ml water

134A.

125 ml 8-12 drops HCl

0.05 g residue

187-9 mp

134B

mp 132-134°

Start RT 1 day filter, work = 19A

0.58 g greenish crystals

15% yield

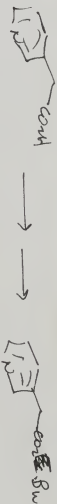
mp 132-134°

mp 187-9 (pure) 134B mp 132-134°





136



#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  
deposits. Add 0.8 wt Brant. - Δ briefly sat.

add 25 wt sat Na2CO3 + dashes of sat Na2CO3 water!

pH → green on paper (40 marks).

XHT 1 x 20 wt ch2z flask → 0.46 g white rd.

pourp run → still 0.46g

after small - no Brant.

#2

as above - no solvent - change still diameter. add 0.8 Brant -  
Kevlar run, lose weight.

15 Isolation of thimble from red-blossomed  
*T. grandiflorus*.

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

cells:  
greenish

xht 2 x 66 ml 10% HCl

cells

Slush

0.38 g  
wily film

137A

0.29 m  
Swedish

GWT

HT

aqueous  
pale green

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

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

↓ to RT.

extract 2x  
~ 25 ml cells

flash

137B-

attention  
function.

0 weight

GCMS

See extract  
in notebook

page 24

137C - same a bit for GCMS  
next (ATMs) - Acid & Vapour

aqueous  
yellowish

neutralize

2 HCl (aq)

Juggle 2

5% NaOH

will want,

+ 60 ml

Sol

Na<sub>2</sub>CO<sub>3</sub>

(pale green  
pH film)

pale yellow  
soln.

Wittmann

fraction

0.03 g

filling  
fluids

137C

See

aqueous

MS

want 2 HCl

PI52 add HCl to

~ 10:11

of HCl

N.M.

show a few  
damp,

Δ SB 2 hrs

RT ON

138



1.8 g aniline (sol in carbon tetrachloride, not in the bottle is a pipette)

into

3 g pyridine - add

1.2 g Ac<sub>2</sub>O (1.02 Ac<sub>2</sub>O)

give warm, wait until it cools, into a 50 ml flask  
 quickly in the hot ether (2 x 5 ml)  
 part ~~added~~ is sat NaHCO<sub>3</sub> - warm -  
 clean - work in the -  
 shade,

fresh ether (acid washed, base washed  
 the warm)

1.72 g pale yellow oil.

GCMS sample

138A

add 1.7 g polyphosphoric acid.

Δ 50 - 1/2 hr.

+ 3 ml (HO - 1/2 hr more,

ref in 2 x 50 ml ether

I have forgotten what I did. ether into base?  
 of base, into ether?

from ether → 1.41 g brown oil  
 NaOH.

138B

(139)

from (138)

Final product  $\rightarrow$  1.14g Amber oil.  
distill. 0.2 mg/45 hrs 600  
130

150  $\rightarrow$  170  $\rightarrow$  0.1 min

160  $\rightarrow$  170  $\rightarrow$  0.70 oil white  
xtals.

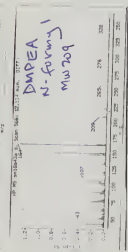
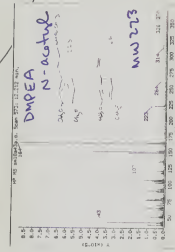
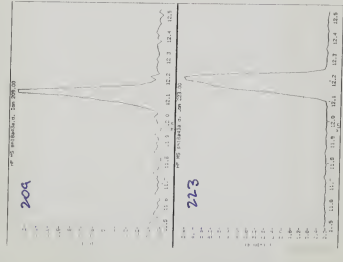
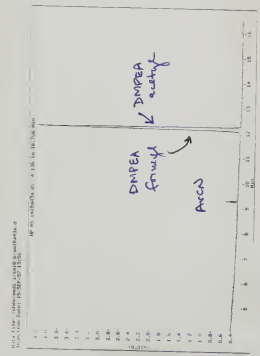


This is the amide

Residue 0.64 | 1140  
Residue 0.74 hex

(139)

See page (41)  
Hexane used.  
cyclohex  
in soil.  
need to eat.

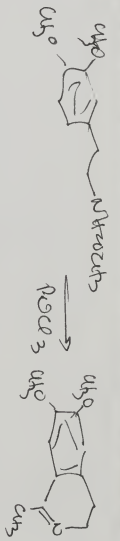


12.212 min

12.114 min  
difference

9.251 min

(140)



Dist g white dim killed solid (p 138) . 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 warm  
 $\Sigma$  5% DMSO  
 xmt 2 x 25 ml  
 chloro

repeat  
 page  
 144  
 flash

0.49 g  
 pale off - g  
 white solid -  
 warm!

(140 B)

Save 100 mg  
 the rest  
 reduce.

Chloro  
 went to the brown color

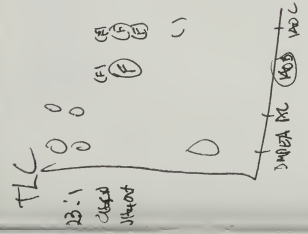
0.11 g black oil  
 140A  
 OOT  
 sunrise

into dilute 0.39 g  
 Hg

+ 5 x 5 lbs Hg  
 shiny yellow  $\rightarrow$  pale yellow  
 OOT - rest - clear -

$\rightarrow$  0.35 g @ 180-140 / 0.5 microns  
 140C.





Repeat - wayland & wash dead. DMPEA → acetamide p 138

3.6g amine (+ carbamate) n 20 ml  
6g pyridine  
2.2g pero → onto SiO<sub>2</sub> ~ 10 minutes. ▽ + 100 ml tbo -

make strongly basic ~ 25% NaOH - x 1 + 3 + 25 ml CH<sub>2</sub>Cl<sub>2</sub>  
color into ether (amber) - wash 2 x tba (boil. H<sub>2</sub>O)  
drown → 3.44 g amber oil

→ 2.46 g white oil 170°-190° / 0.2 mm/Hg x tbo  
all to page 144.

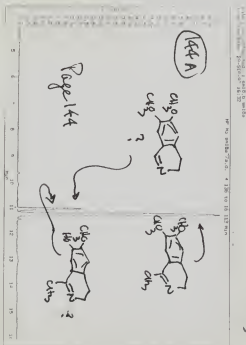
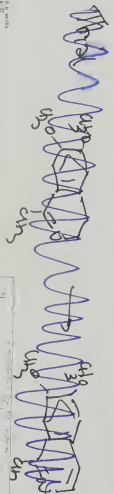
See CM15  
page 139

150° 0.2 no.  
170° white oil  
180° considerable (and  
190° still considerable) pot off

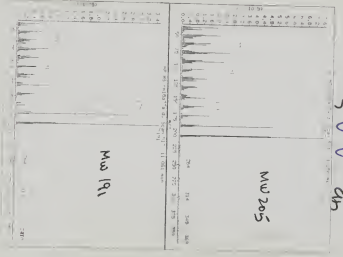
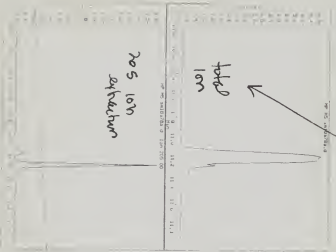
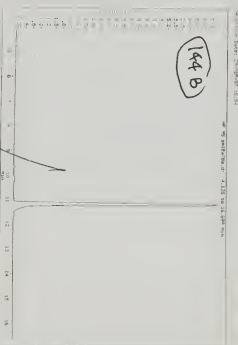
an. side - characterizing thin  
amide 0.13 g from p 139  
drown in 0.13 g boiling EtOH.  
let cool → insignificant x tbo.  
feller (p. plate) - work  
liquor = EtOH.

mp 97-98°  
Van 18,  
Sun 16/6/51.

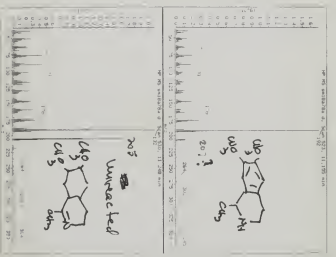
142



144 B



Page 144 A  
 Page 144 B

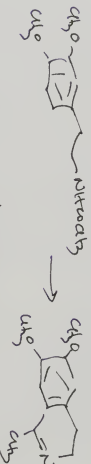


No  
 side  
 chain  
 N.B.!!  
 See  
 page  
 144  
 also



(144) extract of page 140

See page 141



2 g of amide (24141) (all volatile) add  
 10 g PdCl<sub>3</sub> - onto sp. 1 hr. Reheat with water 'no  
 snow (no heat) & heat a little w/ it with the  
 lab bench

1.10. work acidic ag.  $\bar{c}$  ch<sub>2</sub>s, OH  $\bar{c}$  95% NaOH - xlt  $\bar{c}$   
 2 x 50 ml ch<sub>2</sub>l<sub>2</sub> - flask → 1.10g residue that is  
 a fine tan solid.

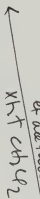
save a small (144) for cons. (144A)

vert 1.085 T into water 10k to ~ 2.5 ml  $\bar{c}$  NaBH<sub>4</sub>  
 on small volume. - old HPLC no record for deep Pt  
 acetic 5.75 - low yields at 8.6 g/hr. - this

should be another specimen xlt - accept on such - work  
 $\bar{c}$  2 x 5 ml H<sub>2</sub>O.

add NaBH<sub>4</sub> spm by stream - H<sub>2</sub>O to so needed to keep  
 acidic.

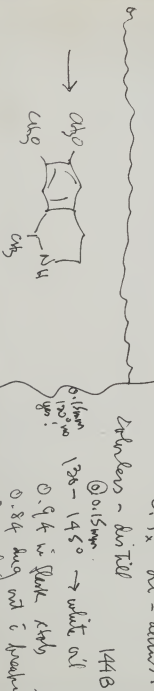
after ~ 2 gram in two of NaBH<sub>4</sub> - oxygen sl. acidic.



the water phase  
 will be used

or about  
 xlt  $\bar{c}$  ch<sub>2</sub>l<sub>2</sub>

yellow ch<sub>2</sub>l<sub>2</sub>  
 0.06 g oil -  
 run in all the carbon column



to page 141



(145)



NITRO CH 3

↳ MW 200 = 5 mM

1.0g MDREA. 4% anisole reference sample Box 1A15!

# (to 141)

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

1.5g pyridine

0.6g Ar<sub>2</sub>O Δ 50 → 10 min ▷ RT add v.40 ml H<sub>2</sub>O + 0.1  
couple of ml 25% base. \* xht 2 CHCl<sub>3</sub>

\* oil change  
save a bit

wash pooled CHCl<sub>3</sub> 2 x 25 ml 10 H<sub>2</sub>O  
2 x 25 ml

flesh → off. white solids 0.54g.

GENS One fresh - good structure  
see page 150

neutralize a bit  
from Stettin. alkyd-styrene

reheat - see 209, 215

POCl<sub>3</sub>



0.52 (about) xht 45 min at 100°C → 1.5g POCl<sub>3</sub>

onto SB 45 min at 100°C → 7 to RT. looks

like xhls coming out. Is strongly acidic -

wash 2 x 25 ml CHCl<sub>3</sub> - CH<sub>2</sub> 25% NaOH - very

cloudy - xht 2 x 25 ml

CHCl<sub>3</sub> - flesh →

oil that sets up to

fine, almost white xhls

0.61g

GCMS peak at P150

trace of 78-15mm

trace of aromatic ID.

0.70

leaves → quite a

bit of double emulsion

(amide?) 0.17g

under STRAPE gum.

2 acetone - flesh

solids. OUT

See repeat page 216



(140)



0.55 g m.p. into 20 ml. h<sub>2</sub>o + 10 ml. sulphuric  
acid into solution.

an equal weight of Br<sub>2</sub> was added, with periodic  
analysis of H<sub>2</sub>SO<sub>4</sub> added to main from ~ unchanged to pH  
pH ~ 2. Two. dilute to 50 ml, pH = 2.2, 100 ml  
oil - to K.R. 0.04 mm / 145° → 125° white oil.  
advent no ppt. 0.40 g absolute  
white!

for the records.

uncovered old flow sheet  
to 30 ml. 84° T

14.2 g Br<sub>2</sub>  
13 g h<sub>2</sub>o

All to flow 2 in 80s  
white with everywhere  
more, not clear, fresh,  
+ 9 g h<sub>2</sub>o + 5 g pyridine  
1500 20 min + distill h<sub>2</sub>o +  
2500 more - clear - xrc  
2 (1) the - ~~to~~ to oil acid  
24 ml. h<sub>2</sub>o - xrc - flash  
→ 1.19 g. K.R. 0.05 mm  
130° - 145° - white oil  
0.40 g. - xrc.  
0.35 g. white.



(147)

isolation from yellow-blossomed *T. grandiflorus*.

140 g green ground-up centers - add  
140 ml 25% NaOH - stir 1 month. (2 days - virtually  
all wt solution - some except some centers) add = vol the  
extract 50 ml  $\text{CH}_2\text{Cl}_2$  - leave to centrifuge to separate  
phases -

eg. →

Chloro

pale yellow - flesh -  
weight mil.

into small vol (2 ml)  $\text{CH}_2\text{Cl}_2$

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

other half.

+ a couple of drops pyridine  
and a couple of drops  $\text{H}_2\text{SO}_4$

GCMS (147B)

no DHT, gramine ~~etc~~  
obvious.

148

To another red-bellied T. grandiflora. Red #2

Run one for photos, for overall slides.  
one banana-skipper caught 2:44 p.m. - always so piercing, they damage grape vine w/out do it! From all spines with the red

4.2 g spines  
+ shoot + dirt

229 g uronema group. add 400 mg 1.5 N H<sub>2</sub>O  
into stream water ~ 3 P.M.

Red Do yet another (I think it is red)  $\in$  wild and (149)

#3 One volume by large  $\sim 8$  cm dia,  $\sim 25$  cm long cactus

472g, worked up to slimy pulp - add = volume (470ml)

2.5% citric acid solution.

Red

#2

144

144

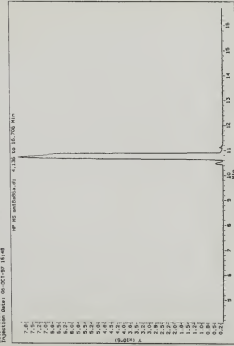
144



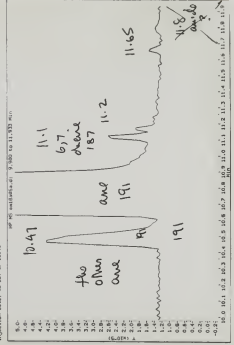
From 146 6,7 MDO MetH H4

151

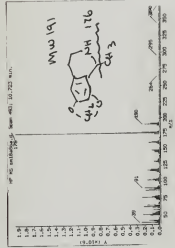
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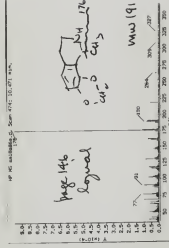
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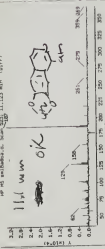
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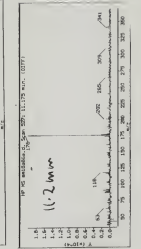
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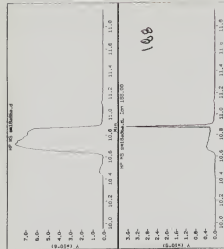
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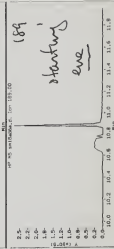
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MSD 6100 GC/MS/MS (10/21/11) 187.00000000000000



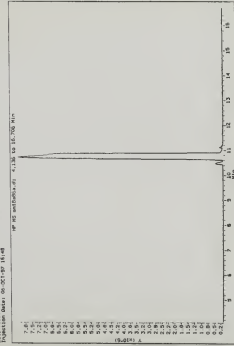
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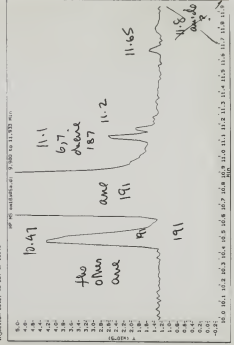
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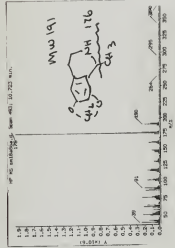
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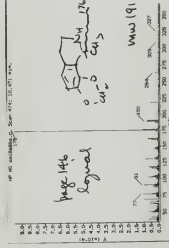
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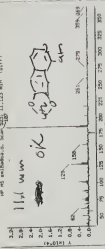
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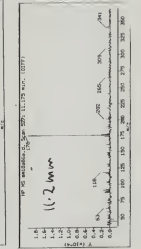
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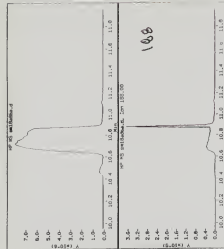
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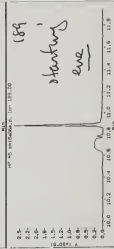
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MSD 6100 GC/MS/MS (10/21/11) 187.00000000000000



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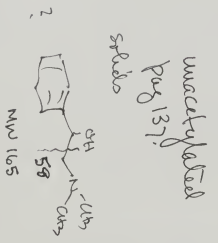
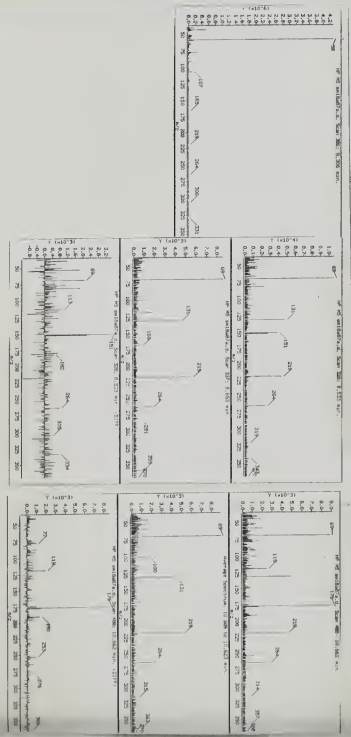
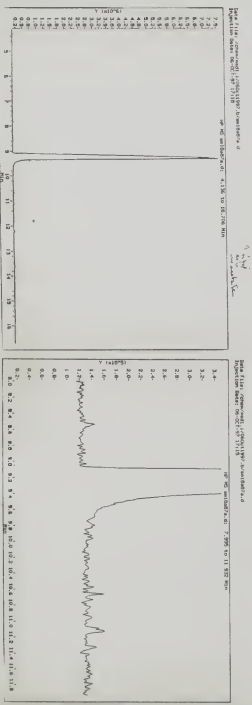


MSD 6100 GC/MS/MS (10/21/11) 187.00000000000000



(152)

PH 9 fraction from Rad #1 page 137





Try!



153



Initially - 5g 5-HT-170 to free base, alkylate, o-sulfate - there you are!

5.16g serotonin. 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. - brown EtOAc

Add 10 ml MeOH - soluble! - flesh → 0.4 g fluffy film

4g Sulfurane - largely dissolves in Δ.

1.3g diisobutyl-ethylamine

1.6g isopropyl iodide

Δ stir occasional swirling. 3 hrs.

Sample removed - Ac2O (B) → separate for GCMS

Post + ~~2.7g~~ 25% NaOH

~~2.7g~~ Et Br - stir ON.

5g a few minutes in SB - stand on.

both sample (A) and sample (B)

pretty much unrecoverable

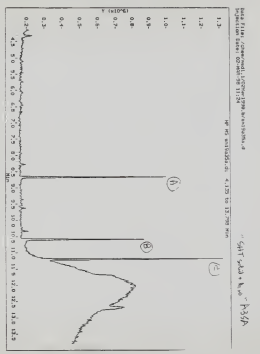
See next page



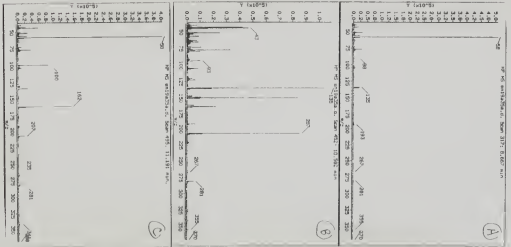
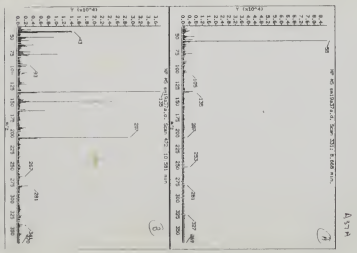
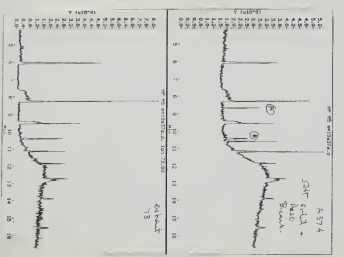
gum + Ac2O (B)

154

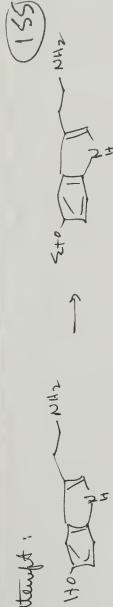
Sample A ('son trin' A Arzo)  
page 153



Sample B (after PCT)  
page 153



Outscript:



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 water, for good luck

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

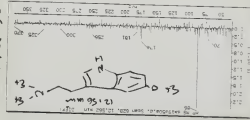
stir RT ~ 2 hrs under nitrogen - not much  
discoloration

xtbt chx<sub>2</sub> (color stamp in ag.) (open to  
air - rapid darkening).

flash xtbt  $\rightarrow$  almost nothing

run in 90/10 anyway  
major product -

Sm19a 50a

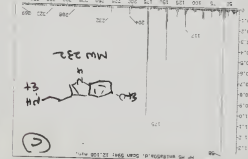
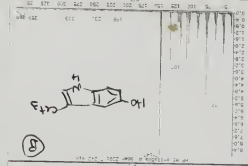
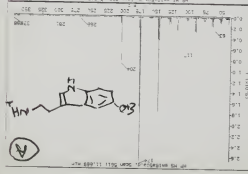


5-EtO-I 11.67 min

5-EtO-NET 12.11 min

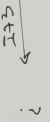
5-EtO-DET found by  
single run 86

12.56 min



155

(156) again - try aush!



2.12 g zero-tonic 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 dihydroxyethylamine  
 Lenses a bit turbid at  
 end of addition - then clear  
 = 3.504 mL  
 dm. 0.742

20g (1 mL) STI - stir. + 5x 100 clean

(160)

(300)

Sample out at 0.5 hr. against. + 5x 100 clean.  
 + drop of H<sub>2</sub>O (don't!) xrt 90/10 Sun 19 AS1A.  
 covered by triup, H<sub>2</sub>S ⇒ major peak 105  
 trace of 5-thio-DET?

→ write up - slightly warm from stirrer. Into 100 ml  
 tho (in al. waste) - extract quickly as is with  
 3x 40 ml ether (Sun color light xrt) - part-  
 exhaust 2x 50 ml die H<sub>2</sub>O, water aq. waste  
 ammonium by the side - xrt 2x 50 ml ether  
 part - wash → check off quickly - ~~some~~ wash  
 residue (if resist?) off more slowly →  
 final residue of ~ 1 g - no number! so is OK  
 ACHS in 90/10 Sun 19 AS1A. FULL One heavy  
 peak - no spectrum - search for 58 & 66 → nothing.

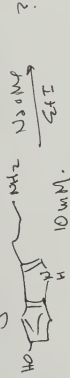


R=H (141)  
 R=H (58)  
 R=st (174)  
 R=st (86)

∴ Mu's to look for  
 H H 204  
 2+H {  
 H 24 232  
 2+ 24 260

100  
 100  
 100

Again - try MeOH aurb.



(157)

2.12 g serotonin HCl - into MeOH 10 mL sol<sup>1</sup>!!

1.1 g N2O Me (20 + mM) then 1.08 - in MeOH 10 mL sol<sup>1</sup> with acetone

under aurb - add MeOH soln to serotonin & swirling - white solid out which do not redissolve (N2O?). Strip well on rotary. residue - brittle - heavy oily stuff - some solids around the precipitate. not nice. Add 0.81 mL (1.56g) EtI - in 10 mL EtOAc

Comm. on swirling → goes sublimably (surface) white x tabs (N2O?) scrape, scratch - more and more x tabs. Dramatized. Let stand under xenon for a couple of hours. White solid!

Suspended in 100 mL MeOH (pH 11.0) acidify &

dil (10) HCl → red. wash 2 x 50 mL EtOAc - brown 2 x 50 mL MeOH - x HCl 2 x 50 mL EtOAc - some little bit of the brown color goes into the diethyl. - pour - flash → small amt - viscous oil.

GCMS page 158

n = aurb  
 5-EtO-T  
 5-EtO-NET  
 5-EtO-DIET

treat i acetic anhydride  
 Kill N2O - x HCl 2 x 25  
 x HCl 2 x 25

neurotoxic -  
 EtOAc wash  
 EtOAc flash  
 → 80 mg  
 neurotoxic

aq. water basin  
 3 x HCl 2 x 25 mL EtOAc  
 flash → 80 mg

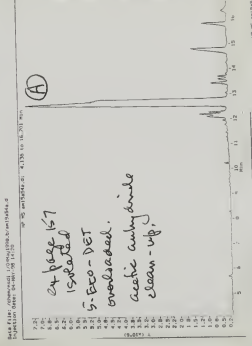
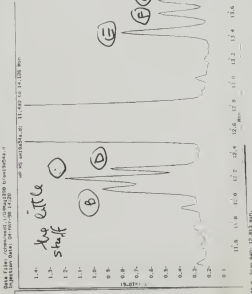
test amine fraction (10% HCl other in 100 mL)  
 spectra 159

spectra page 160



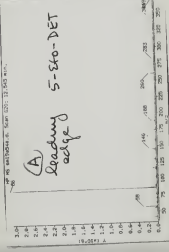




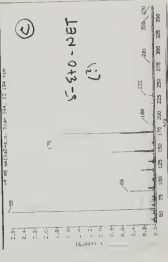
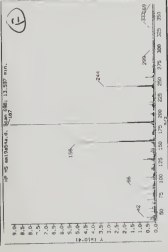
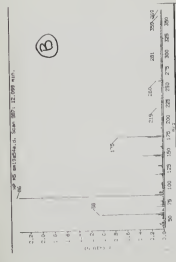
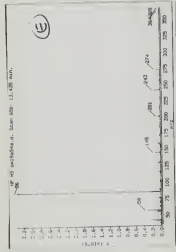


4 pages 157  
 158  
 5-Eto-DET  
 overlaid.  
 acetic anhydride  
 clean up.

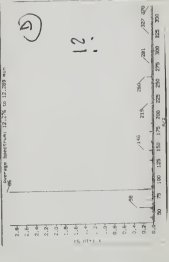
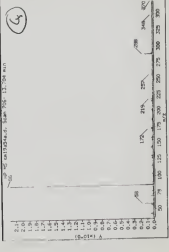
(A) trailing edge.



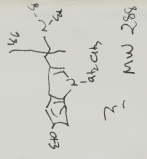
(A)  
 leading  
 edge  
 5-Eto-DET



5-Eto-DET  
 (1)

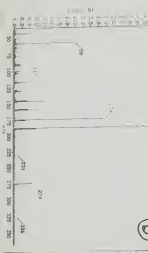
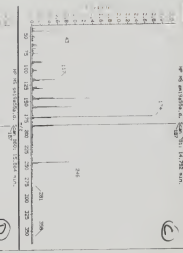
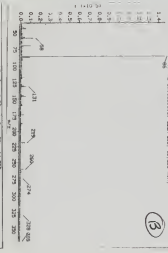
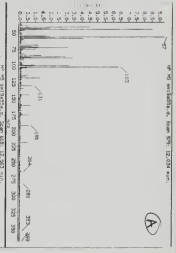
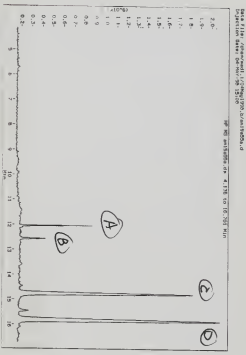


12

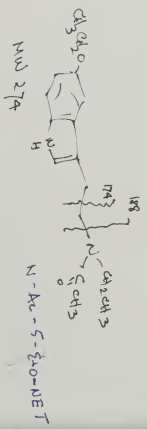
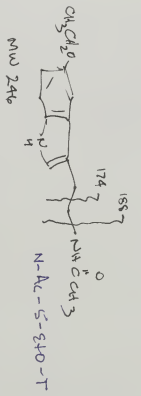


Also

acetylated neutrals - see page 157



5-Ethyl-DET



Small scale experiment



CH<sub>3</sub>I



NaBH<sub>4</sub>



a few mg H<sub>2</sub>O<sub>2</sub> - into water → clear yellow soln.  
add a few drops N<sub>2</sub>O<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>3</sub>I - never really dissolves but  
gets immediately yellow - should have added hindered  
base? let stand ~3 hrs. CH<sub>3</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  
→ cloudy that on spatula scraping → yellow solids!

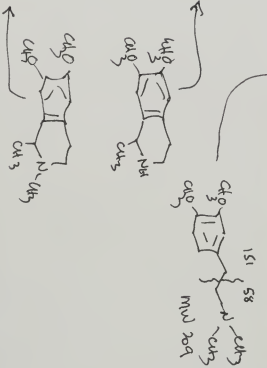
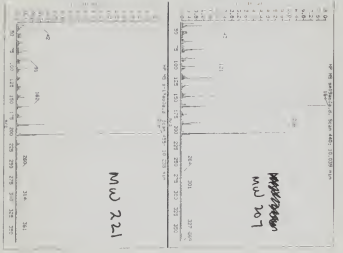
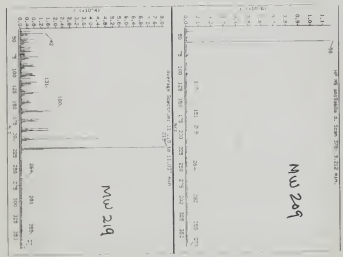
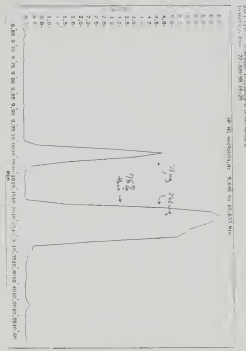
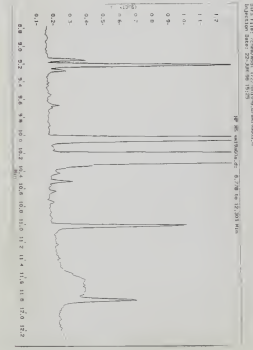
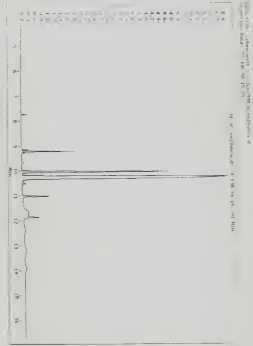
see page 164 for barium/bromine resolution

Sample TLC  
(C)  
SM19  
A64A

161

162

from page 143 incomplete DMM



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



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

(163)

164 Let's straighten up the Sigma Humaline/lanuine when the next sigma humaline the lot 123N 3717 appears to be lanuine by ACSIS page 161

Ref. lanuine

Humaine the TCI lot A501 I break seal, 10 mg into  
~1 ml H<sub>2</sub>O (sol) + 5% NaOH - check  
evap → solid. (A) 5M1A A15A A73

Humaine base, p133b - base washed, by digestion of humaline  
H66A

Humaine the, Radio - lot 229 into 1 ml <sup>(B)</sup> H<sub>2</sub>O - + 5% NaOH  
→ slowly - slt 2 H<sub>2</sub>O<sub>2</sub> - evap →

Ref. Humaline

Humaline (base?) Sigma lot 28C-0433 - into check -  
evap → pale yellow film. (D) A68A

Humaline (base?) Aldrich, lot # 01321 AV I break the  
seal; into check (sol) and evap →  
yellow (pale) film solid. (E) A69A

Humaline (base?) Aldrich, lot # 11226 PX - into check -

Humaline the Sigma lot # 123H 3717 - this is the  
Peak → film of solid. (F) A70A  
SUBJECT sample that participated this  
white thing, sol in 1 ml H<sub>2</sub>O - base:  
5% NaOH (G) A71A

Ref. Tetrahydrolanuine

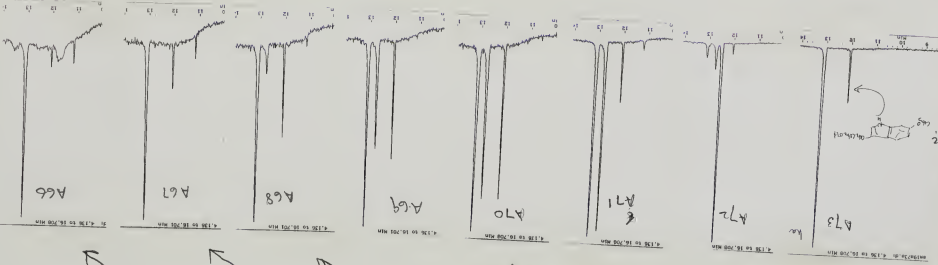
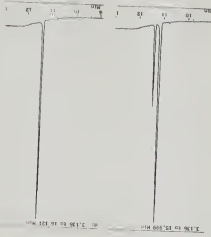
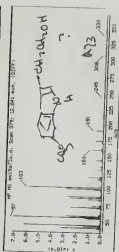
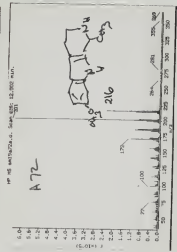
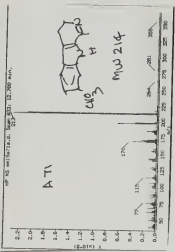
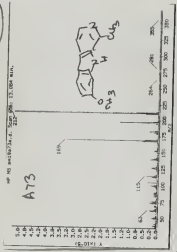
ligno lanuine (base?) Radio lot # 12L 549 Pl-70  
into check - sample, solid film. (H) A72A

A-73A - violet of A15A -

A-74A - yellow w/ol from lanuine

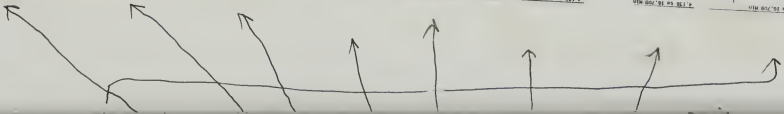


165



code  
127  
analogous  
code  
239  
analogous

same type samples  
clean column



analogous  
clean

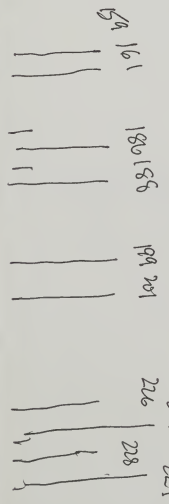
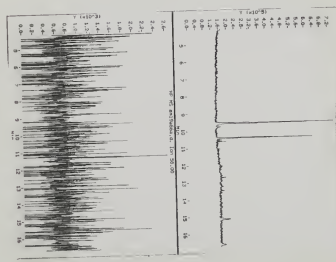
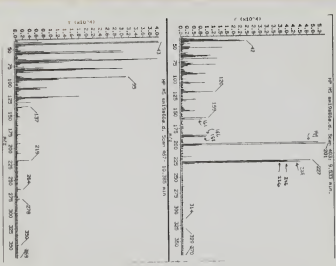
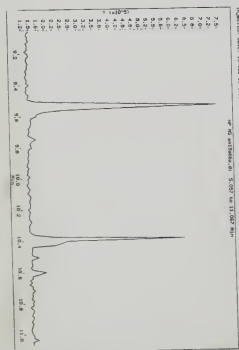
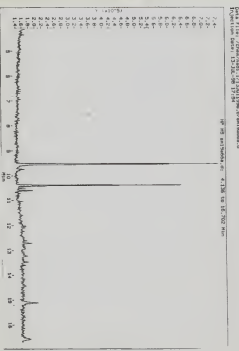
A73  
A72  
A71  
A70  
A69  
A68  
A67  
A66

160

Extract.

1189 aerial part

S and HO - thymophy swatch + square in Nant -  
Kht c 1/2 was ch. d. - pin → identified green  
solution - reformed - plain - with 1/4 was 90/10



160  
1189  
15/1

(167)



S Neo-DIPT

11 p 171

2.46 g TCI amine - dissolve in  
 18 g warm (SB) Sulfolane; 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  $\phi$ ! when broken  
 to strip on R.E. 2

add 150 metho  $\rightarrow$  2  $\phi$  (heavy oil, red)  
 xft  $\bar{c}$  35-60 pet ether 3 x 50 wt

heavy strip  $\rightarrow$  1.16 g deep amber oil. slight brown  
 transfer  $\bar{c}$  ether to KR flask 0.92!?

-182.47  
 -39.77

no smell!  
 (then ugly)

KR!.

0.2 mm  
 95°/40  $\mu$ m cloudy.  
 flame - flash into  
 sunbale. -

up Temp.  $\bar{c}$  155/40  $\mu$ m. white oil. trivial black pt.

0.57g.

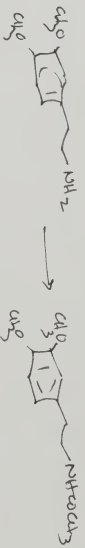
0.57? more? balance crafting

into 2.6 wt IPA

+ same like  $\rightarrow$  red = 23 drops!! xlab  
 + another wt IPA (still xlab)  
 + diethyl ether 2.0 wt fine xlab

Soliter - wash  $\bar{c}$  10 wt 3/2 IPA/ether  $\rightarrow$  white xlab  
 0.82 dry?  
 ml out.

(106)



11 p 135

9.0 g fresh - clean amine from TCI add  
15 ml pyridine add

6 ml Ac<sub>2</sub>O - quite hot - let cool to RT over 1 hr  
+ 150 ml H<sub>2</sub>O - some HCl  $\rightarrow$  used (a few ml)  
Xfct  $\approx 3 \times 75$  ml H<sub>2</sub>O

prod - wash 1 time  $\approx$  50 ml 10% NaOH.

11.45 g carbon  
K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> every culture - throw away  $\leftarrow$  I don't know the  
to K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>  $\approx$  10.58 g solids. Volacra

0.15 min RT NR.

200 gr 100° washing.

150 gr 140° washing.

150 gr 150° up water.

165° over - white beautiful.

175° 1/2 over - cool  $\approx$  clear

180° 10% aq.  $\rightarrow$   $\approx$  clear

185° done - vacuum clear

Gurukul Evida

wf (p. 141) 47-48°C at 300K

MS (p. 134) 164 (100%, styrene) 150 (49%)

140 (23%) 225 7% present

9.73 in presence - don't burn  
9.63 Mps. cold  
recovered.

fade yellow solids -  
pale yellow almost white.

9.50 clear almost white  
to right  
weight

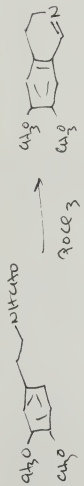
0.50 g same  
9.00 g to p. 104

MS  
100%  
100%  
100%









8.5 g formamide (CH<sub>3</sub>CO<sub>2</sub>H) to remove amide from KR  
 voluines - strip n R.E. - add

42 g POCl<sub>3</sub> - swirl - evaporation & pretty strong  
 bubbling - outside! - when quiet (and still  
 quite warm) onto S.B. 7 PM off - 1 hr. Stand  
 O/N - into n 300 ml H<sub>2</sub>O - controllable -

forgot to wash c CHCl<sub>3</sub> - basic  
 c 25% N<sub>2</sub>O<sub>4</sub> let cool. - what c 3x75 ml  
 CHCl<sub>3</sub> - prod - wash 1 time c 50 ml H<sub>2</sub>O -

Steam -> 6.95 g amber oil - sundry in an  
 x hot dish in lab. AM - some xstals (solid), not  
 can I think) + some minor xstals in flask -  
 (not wash - nice! - same bath.) 6.95 g on  
 gravimable balance. Transfer to KR pot -

40 μ in hand setup  
 140 μ. setup  
 Δ → n 70°  
 150 μ.  
 200 μ 190° over

white  
 V. antiferd,  
 140° a 400 flit  
 150° over  
 say 13.5-145/200 μ.

6.64 g ← .05 + 6.59 g white oil c  
 → some in 2nd receiver,  
 50 μg.

GCMS  
 solvent entials  
 DMTHT-2  
 some DUREA  
 see page  
 173 for  
 spectra

try seed, 171A  
 distinct H<sub>2</sub>O. GCMS  
 a little Vit - into IPA  
 + H<sub>2</sub>O → yellow xstals.  
 wavy.

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

(171)

Play with C1=CC=C(C=C1)C

Just under 7g of distilled product - colorless when distilled - 2 deep dists - water very abundant. Dist 20 wt IPA. - add em. the drugmine - checking acid (1 pt paper) - water really red - no dark acid - plus straw (yellow). shi - add - 10 wt ether - heavy mass of straw - (yellowish).

soln.

Mixtures

good quantity of soln. when 15 wt 2/1 IPA/Ether. IC is dissolving!!! Swale say (My guess is? w). into poly film n > 3g (172A) sun ems 2.225

add mix's → flesh. → seasoned with heavy oil.

oil into 100ml water & swirl. that 4x 75ml dist till

ether, flesh → 50mg of sound

waste basin: 25% N/AH that 3 x 50 ether - flesh

(172B) for acns - vent out. N. (see page)

See page

M3 191 (100%) 176 (82%) 190 (32%) 133 (25%) p-2

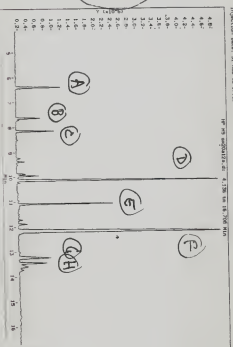
(172C) (172D)

clean up - add water 1 wt per 20 lmk, because water + 20 wt ether - dissolve total 150 wt ether, 1 x 3 x 50 wt 100 wt - wash x ether - 8th - 25g 5000 lmk 7 x 50 ether, flesh → 2.57 g water oil K2SA

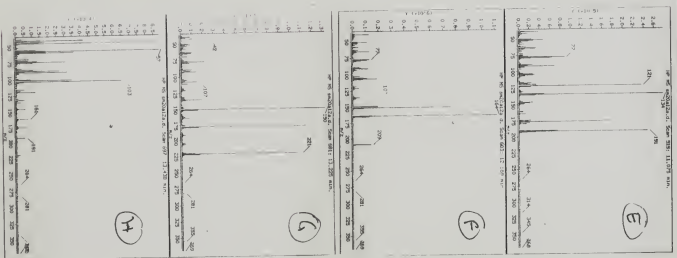
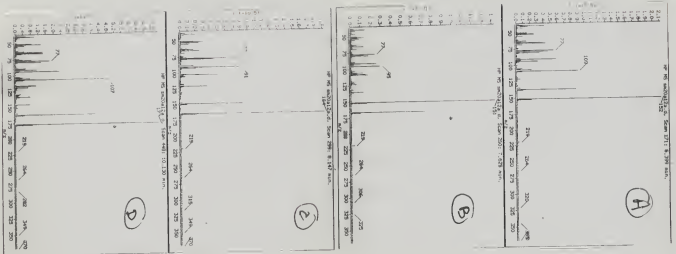


174

from  
172B  
sample



partial or  
acid fraction  
→



(175)

(F) is definitely C#N formamide

(D) is maybe O=C1N=CN=C1 a zwitterion, neutral

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

Make extremely strongly basic (hold D as the anion?)  
that i-C4H9Li (all color goes in by sig - too bad!)  
take aqueous (25% base) + HCl until pH ved.  
let stand - ~~2~~ 10M - some color has xtds -  
white N2c! OUT

---

176



DMHH-4

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

some H<sub>2</sub>O containing roughly 1 ml HCl. Stir - add  
~ 2 g NaOH (1/2 in - re-run warm - water - add  
3/4 in add 1/2 ml more HCl - add rest - color  
remains nearly feature!

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

150 μl  
200 μl 110° w

125-140 (with 135) 150 μl white oil  
135-145 @ 150 μl 1.80 g - add sets  
to white solid.

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

Try vertel DMTH-4.

feature too  
10% toluene in try. no.

176-X  
KACAMS

0.5 g into 10A 5 ml hot - almost all in.  
I more ml still not all. → - no

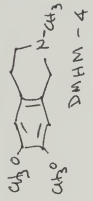
stirring - a few 10A & not enough -  
100 mg 10A runs - short. xrt 5  
immediately - filter - 10A with.  
oil dry → 0.45 g of acetone  
white xrt 176-B mp 25+ 25°

Keep trace  
run ACMS  
run 10A  
run mp.

176-A  
mp 115-127

spectra  
page 173-170





1.18 g (way for more?)  
MW 143

DMHM-4  
MW 143

his - calc. based.

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 37%

(~6 mM)

add 1.5 g N<sub>2</sub>B<sub>4</sub> exo Mercuri  
freeze dry  
very clean.

(~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 N<sub>2</sub>B<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 N<sub>2</sub>B<sub>4</sub> good exo Mercuri. (B<sub>4</sub>H<sub>4</sub><sup>-</sup> in sol. wet)

Let come to RT. + 300 mL H<sub>2</sub>O, MeOH 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 manif.!

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

Wrong, wrong, wrong! IR shows it to  
be a SWEET, but one that distills!  
0.94g  
← 177A

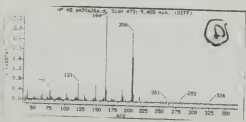
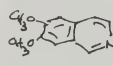
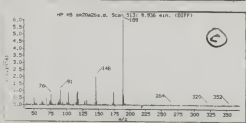
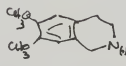
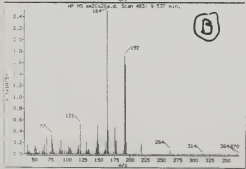
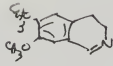
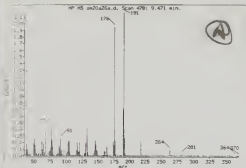
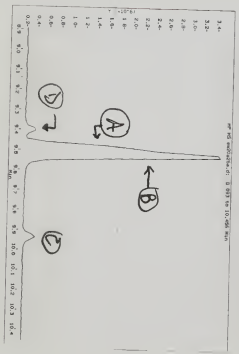
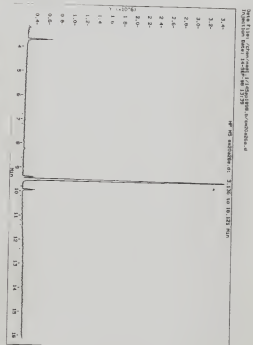


?

178

Spectra from page 176 DMFH-4

176A

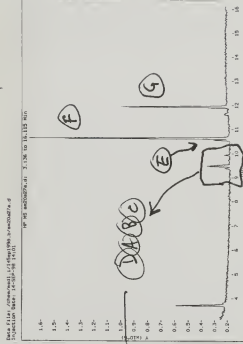


MW 207

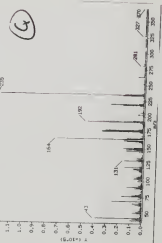
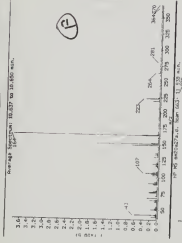
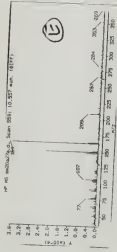
spectra from page 176

179

ve KR  
176x



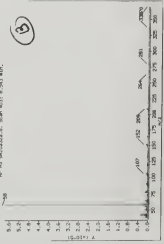
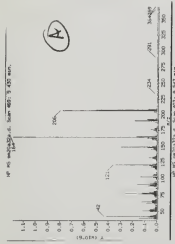
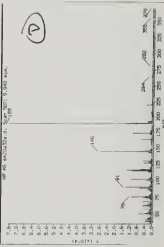
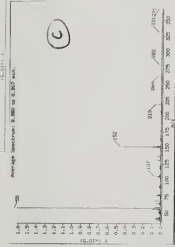
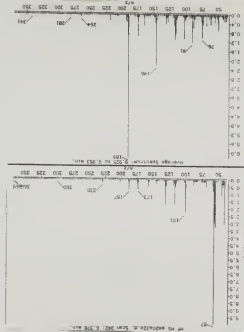
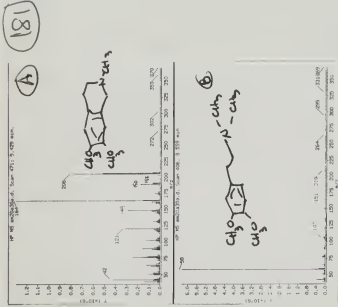
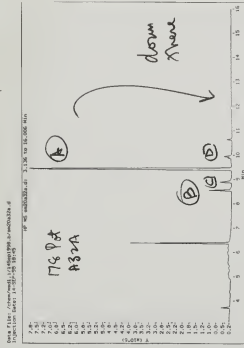
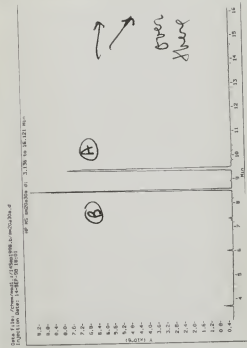
see they  
= spectra



150

Attempt to write the "Pensive" (part 179).

177A C = 178x !!!



142

### mono acetylphenol of 5-HT

→ 2.12 g serotonin.HCl

1.5g

1.5g

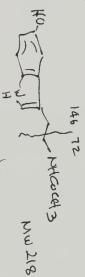
→ 1.52 ml pyridine - stirred slowly as it goes to a rather solid cake,

→ 1.04 ml Ac2O added evenly, drying, 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 - a small amount of pyridine - back into ether, wash in 1.5 ml H<sub>2</sub>O - flash → 0.9g white oil almost no smell

serotonin 176

N-monoacetyl



3,0-diacetyl



triacetyl



take all 0.8g. Δ in rotavap (50°) ~ a few mL conc HCl - add water - mix - check. → 0.3g film.





(84)

### Diacetylmorphine of Seos formin

2.12 g SAT.HCl

3.12 g .Aceto

50

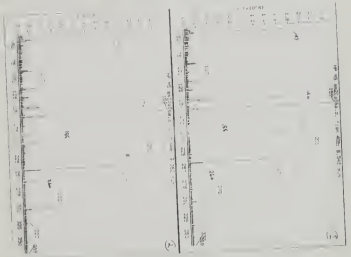
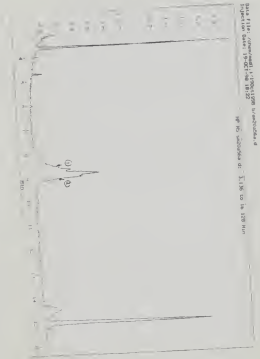
I don't remember

few drops - into sol.

15 we slowly

not kinetic not in phase

XRT  $\hat{c}$  50 we check (not not sol, in oil in solvent  
sol check anyway  $\rightarrow$  2.0 g viscous white oil.



Triacetyl Seos formin.

(581)

(186)  
 pink  
 p. 4416



SMO A61A

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

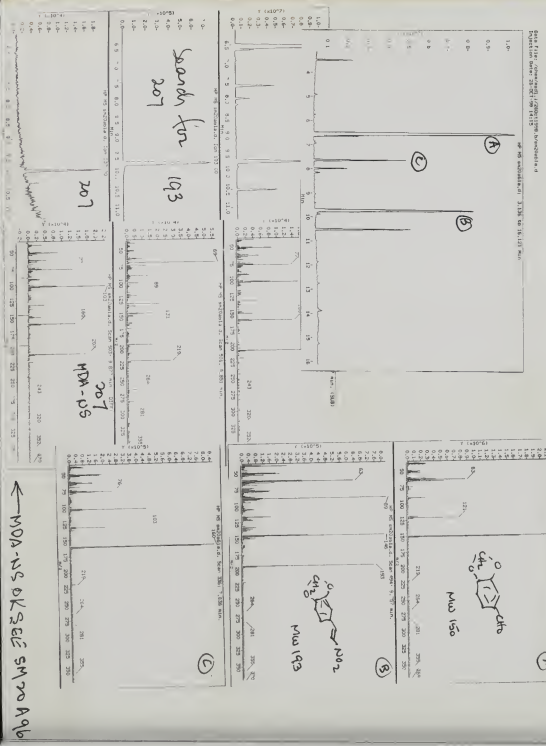
20ml chloro  
 to wd cyclohexanone (blank know!)

on 90 ~ 5 pm - off at 16.0 - observed scratching  $\rightarrow$  yellow xtds

$\nabla$  RT. - rather heavy yellow xtds.

fil. kn. wash  $\bar{c}$  H<sub>2</sub>O - such rather dry -  
 crude wet weight 8.15 g

Mls stand ON  $\rightarrow$  some more light yellow xtds. (186B) out  
 Mls + water to turbid - stand  $\rightarrow$  3.7g wet (186C) out weight (189)



$\leftarrow$  MDA-MS DK 2 E15 SM 20 A 96



4.8 g K1 foil (n. 1 foot - 1" squares) +  
 0.12 g  $\text{HgCl}_2$  in 150 ml  $\text{H}_2\text{O}$  - let amalgamate -  
 already ready in 30 min  
 decant off  $\text{H}_2\text{O}$  - wash 2 x 200 ml  $\text{H}_2\text{O}$   
 1 x 200 ml IPA.

add 100 mL IPA (RT)  
 10 ml  $\text{H}_2\text{O}$

add 2g wet NS page 186 in 20 ml hot IPA.

stir - stand on - AM - gray sludge

filter - wash 150 ml  $\text{H}_2\text{O}$   
 150 ml  $\text{H}_2\text{O}$   
 20 ml IPA  
 100 ml  $\text{H}_2\text{O}$

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

~~add 200 ml + ~5 ml conc  $\text{H}_2\text{SO}_4$~~   
 add 200 ml + ~5 ml conc  $\text{H}_2\text{SO}_4$  - add yellow into org-  
 strip.

make basic c 5% then 15%  $\text{NaOH}$   
 strip c 75 ml  $\text{CH}_2\text{Cl}_2$  (no color).

strip - SMA64A

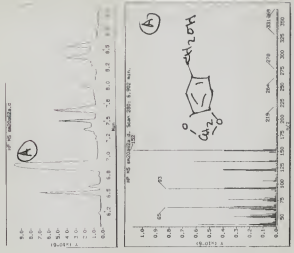
0.40g viscous white oil.

0.52 g sharp solid  
 (H<sub>2</sub>O?)  
 visc yellow oil.  
 SMA62A

see next page

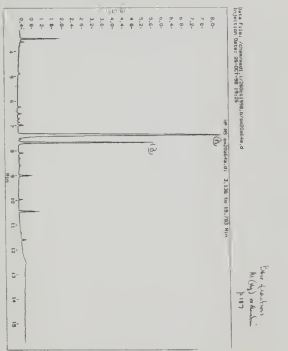
this is pretty  
 good time to run  
 to page 195

neutrals



(188)

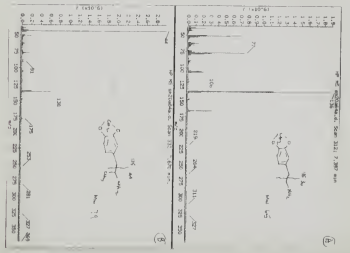
Gene fraction from Ad(117) red. of NS  
(Mass 187)  
SM 180 Ac4A.



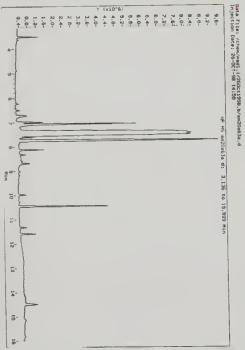
Ad4A

↓ diluted x 10

↓ overlaid



Ac4A



Ad4A

Ad4A

Ad4A



Another try:



11. p. 512 2c5  
P. p. 503 2c8

25 g polyanimal

75 ml Me<sub>2</sub>SO - which 90% pure  
Let 0.110 g CT A filter

orthoformic soln.

2.5 g wh. Me<sub>2</sub>SO - not sol.  
soln less - on SP 10 hr.

1 hr. turned a dirty yellow - TLC → spots stable  
at 5.6 yellow

at 5 absorbance  
at 4.4 ? no color

off at 2 1/4 hrs. - into beaker - D to

cool water temp → heavy yellow  
stabs - filter - wash in water, wash

in dry → 16.53 g (189A) beautiful yellow stabs

Small amt. of sp

not H<sub>2</sub>O, stabs in D,

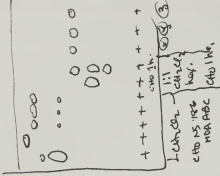
Spin milk + washes -

on standing: more stabs in MS

filter (189B)

(189)

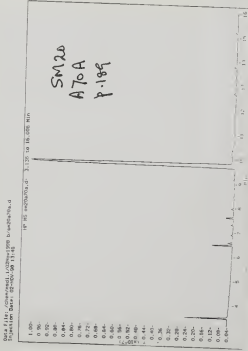
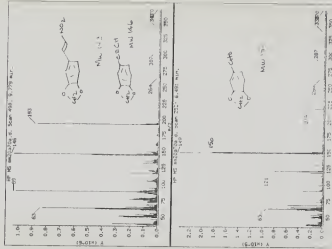
TLC stuff



play = ext. anal.  
Small amt 186 A  
wash boiling IPA (X)  
stabs f. Me<sub>2</sub>O (X)  
1 1/2 hr. pot. 6 (Z)  
RT

Wp stuff:  
189A. 151-8  
on Me<sub>2</sub>O 1158  
see H<sub>2</sub>O 161+13

P. 189 - MS from water



190  
190

Pyrogallin:



Start @ good 125 p 189

16.3 g good 125, 189A into 200ml glue, 4th re  
 1/2 hr on steam bath we had a  
 clean spec.

40 g of Al foil - 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 -  
 drain well - add:

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

125 in hot H<sub>2</sub>O. add @ pretty good hard stirring -

1st grey - immediate yellow dyeing (hot to cold? H<sub>2</sub>O to  
 18A @?) add the rest over 5 minutes. stand 10 min.

AM - clean the amalgam has been killed - let on RT -  
 wash color still, replace w/ hot to push - just work strip

Follow all steps - orange color water - wash @ @ 3 x let on

18A - continue - Reak → heavy yellow - real old  
 holds in it, w/so chels - xlt 3 x 100 ml dilute HCl - part -  
 wash one @ chels - pale yellow

9H<sub>2</sub>O @ 125 hr - xlt @

3 x 50 ml chels - Reak → 2nd  
 of poor fraction. To K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>

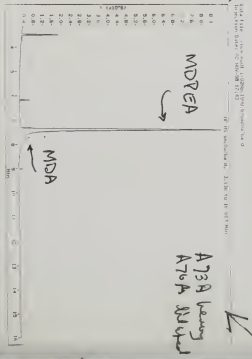
0.2 hr  
 0.15 hr 80° wash  
 90° - 100° / 0.1 hr  
 2.35 white oil  
 Mar yellow

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

simulac

w/so to formula water - page 191

decolorable MDA - 5400 in the Wand 2



See page 190

Attempt:

11 to 170



2.3 g freshly distilled hexamethylamine, rinse out of KR flask =

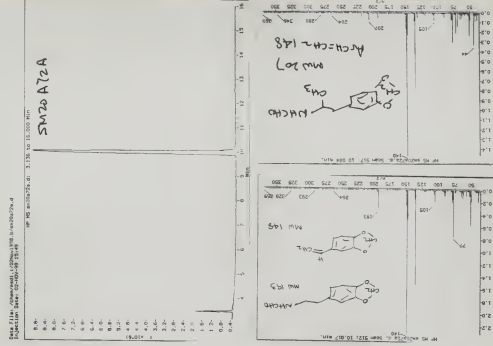
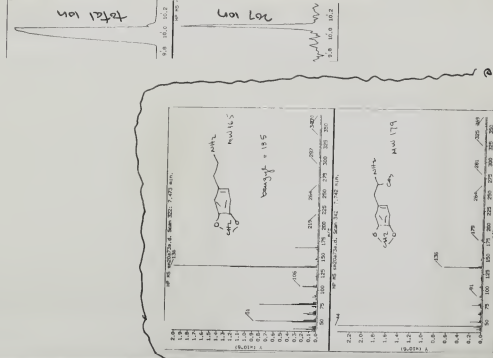
7.0 ml isobutyl formate - Immediate solids !! Must be a. bit of carbonate - very quickly - rest of formate gives no more - i. not immediate Rx - no formate smell in formate. Onto SB 5:30 - Off - 1.35 hrs

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

3.44 (smelly) 18 formate 60 min 110° 40p 136° 2.45

flask → 7.44 g almost colorless oil - smells of 100% 100% to 140 - 0.05 min immediately ↓ white solids in pot. A call 145-155 (higher) at 40p → white oil that solidifies - 2.45

2.45 isolated sample p(19) 2.30 on to DIA



192

See page 190

The deep red solution phase (the extracted) on the R.F. → red crystalline substance

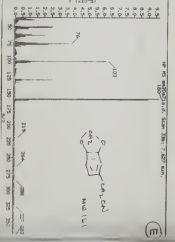
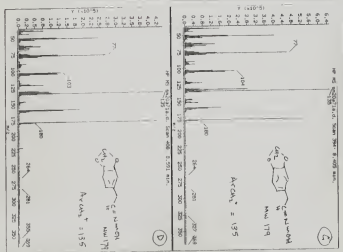
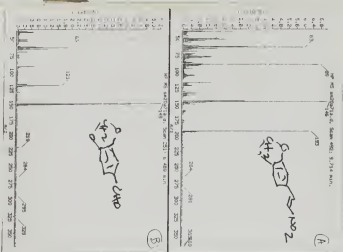
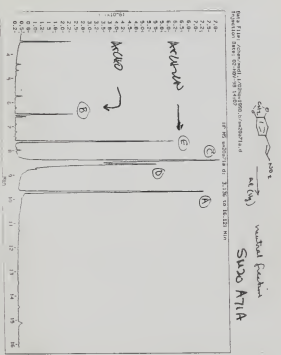



taken to dryness

(192A) 10.36 g

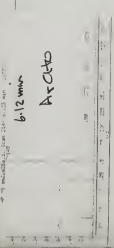
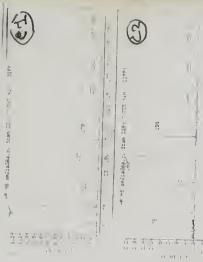
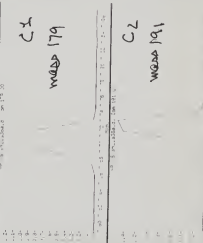
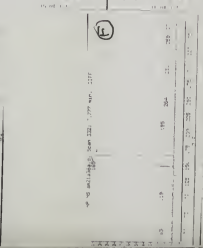
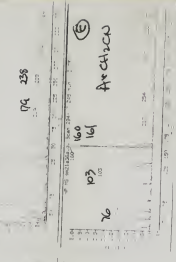
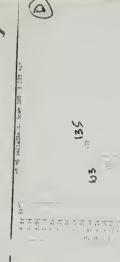
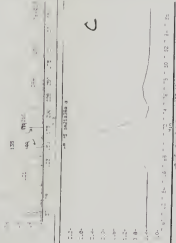
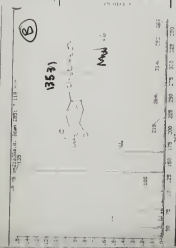
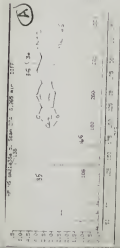
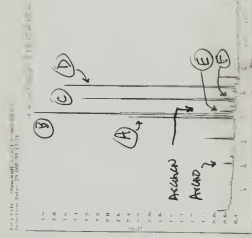
5 crystals out 8.7 g

Same in  
homoverbonylamine  
crystal



numbers from  no. 2 → 66 (H<sub>2</sub>)  
 Sep 14, 2010

193







from page 187.



195

0.40g base - now broken - into  
[ 2 wt H<sub>2</sub>O + 8 wt MeOH ] to 2.5 wt stirrer add

2 wt 37% H<sub>2</sub>O -  
1.5 g Na B<sub>10</sub> - splash. wine - over 20 min. - feeling over - add  
spritz 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> -  
work 2 x 50 wt each 2

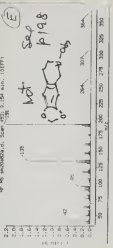
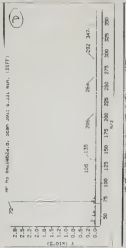
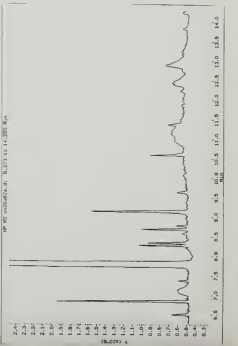
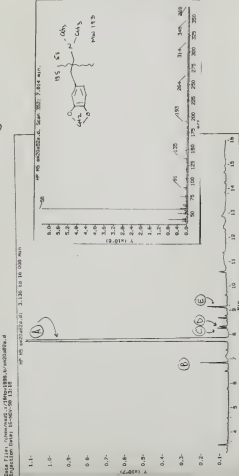
+ 25% Na<sub>2</sub>SO<sub>4</sub>  
X<sub>2</sub>H<sub>2</sub>O < 1 wt

spin both times.  
0.25 g pale amber oil  
IR 0.02 μm 96-105 ~ 0.22g

into 1 wt H<sub>2</sub>O + conc HCl  
2 wt enough 4 drops too much -  
stir 30 min + 2 wt ether - drain  
plates 1.75 wt white x<sub>2</sub>H<sub>2</sub>O  
+ 15 wt ether

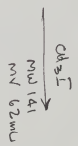
not quite white oil - 10-15% white!

GCMS 195  
GCMS 20 A B2 A



Not sure P198

1916



0.6 g DHA (19A) into

1.0 ml MeOH - Δ → still solid; +

0.3 ml CH<sub>2</sub>I<sub>2</sub> (150% xs) immediately yellow color - Δ on Rot. Evap -

Δ - nothing - Δ again Δ RT → fine white - evaporate more and more - add some solid - add ~ 3-4 ml MeOH - solid disappears but not all dissolve. Stand. RT a few hours - yellow solids.

Filter - wash catch on filter - wet weight (yellow + hb) 0.55 g.

0.42 g dry

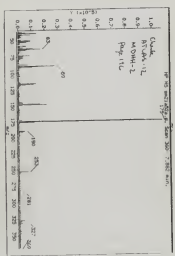
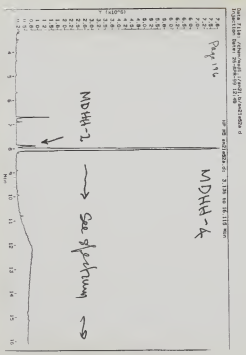
0.35 g on to solution

Same 0.04 g ref. (1916)

M15 → yellow crystals - onto plate. 1916A

Calcat - on page 222 from M1071 (19A too weak)

Wash up 0.149 g (197A) - now turned slightly of white. Absorptive id 3 we but 19A (1/2 not enough) add 12/14 and zinc try to strip, off white. Filter without ether - wash 2 small cuts 19A - then filter. 2 small cuts ether → 0.43 g off-white MDAH-t<sub>4</sub>. 192 (197B). SW21A52A





MDH-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

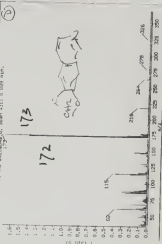
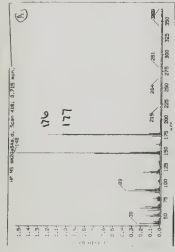
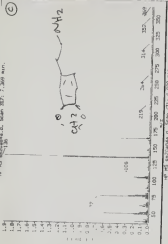
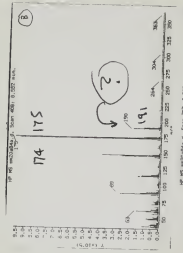
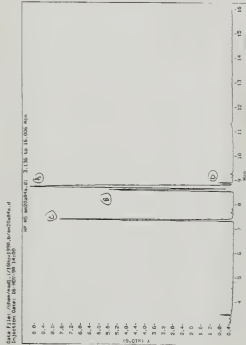
20 ml H<sub>2</sub>O - not much change - add several cc 1 N HCl. some foaming - solution largely clears, and no more response to HCl.

strongly acid - shut 2 x 40 ml ether → vents  
 OH = 50% NaOH (get new pH paper!) - shut

3 x 40 ml ether - flush → 0.56 g white crystalline solid. to IR.  $\star$  fused white x-hob

over 90-100° 60  $\mu$ . → 0.49. fused white x-hob into dist. vial. (197) small sample for GCMS 197A SM 20 ABGA

50  $\mu$  50°  
 60  $\mu$  90° oven





0.38 g yellow solids, 10 wt thd - w/ completed solids - add  
 some 10 wt thd - no better - add

0.39 g NBH<sub>4</sub> from etc. add over thd - finally quiet - when no longer  
 sample has gone. + thd + the - xht 2 x 5 wt thd +

0.11 g S<sub>2</sub>, from 25% bar → white slurry - hot 3 x 40  
 wt thd

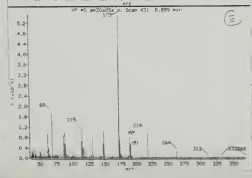
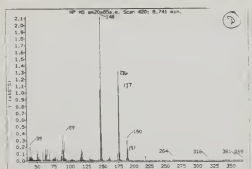
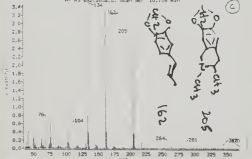
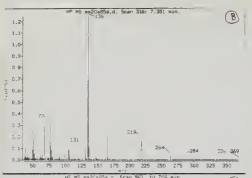
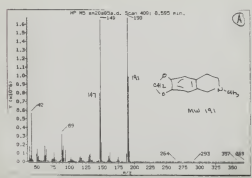
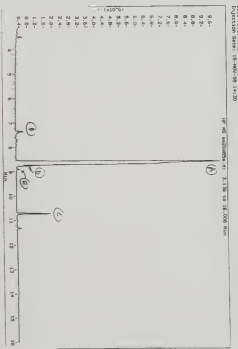
0.16 g bar  
 in P.E.

K.P. → white rd 95-95/50  
 immediately hot & vapor

150 wt white rd.

small sample 2 dry ice → xht - tubes.  
 small sample cins SM20ASSA

0.16  
 Cuside  
 wave  
 95-95/50 yr.  
 cins  
 white rd 1  
 w/ 2 kg.  
 part T.  
 to 1 yr - still part rd.  
 nothing wave.



Defect,

See 170  
171



199

10 g DMPEA, substituted i carbamate - the ancient bottle of Pedicis char. stuff - all that would drain - carbamate now solid in bottle.

25 g isobutyl formate → cloudy - Δ 50 ± hr → brown soln. - add water c 5% D2O - feed → 10.85 g ~~viscous~~ oil - ~~viscous~~ diesel crude

KR 15-160°/120 μm → 7.52 g vacuum oil pale amber. into 45 ml POCl<sub>2</sub> - spill - nasty - into 58 ml (1.5 hrs actually.) - off - cool OP. Into 400 ml cold water - exotherm of course - Δ t too - xht c 2 x 50 ml CH<sub>2</sub>Cl<sub>2</sub> OH c 25% N2OH to pH 13 - Δ → solids, H<sub>2</sub>O to 1300 ml to dissolve - xht in 1/2 batches c 3 x 50 ml CH<sub>2</sub>Cl<sub>2</sub> each - pond. work c 1 hr - flask → 4.24 g vacuum brown oil. KR.

200 μ 130-140°

273 g. Save ~ 0.2 g to see how the oil discolours colour.

250 g to page 200

neutralized

→ this all from memory - lost the paper c the notes and weights. Flash of CH<sub>2</sub>Cl<sub>2</sub> → a lot of material - 2-3g that tends to go solid. TLC a bit checking against reference sample of formamide. solvent 80/20 hexane/ethyl. Amide ~ 0.2 Rf. thin stuff only in. Could the salt of DMHT-2 be extractable into CH<sub>2</sub>Cl<sub>2</sub>? Partition ~~of~~ in OH:H<sub>2</sub>O c CH<sub>2</sub>Cl<sub>2</sub> - ethyls to diguon ~ 1.5? to KR. So, so vacuum (N 200 μ?) and pump 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.

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

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





NW 207

4.0g gold-orange xstals f. p. 200, into 100 ml the  
 squirt of N the - not quite clear, but very yellow, solution  
 1g NaBH<sub>4</sub> added a bit at a time, with occasional  
 squirts of N the. Color dissipated with the first  
 bit of NaBH<sub>4</sub> - add 5%, then 25% NaBH<sub>4</sub> to pH > 10 - heavy  
 gel of may be sodium borate? Dilute to ~ 600 ml -  
 the which pretty much clears it  
 that 2 x 3 x 50 ml conc<sub>2</sub> - absorbance colorless! poor, clear  
 → 2.44g crude white oil, xstals in flask - KBr →  
 11.5 276g white oil that dithiols at 120-130°C at 300  
 reweigh when dry out of the receiver. Beautiful GCMS.

Picked out the rest  
 1.84g

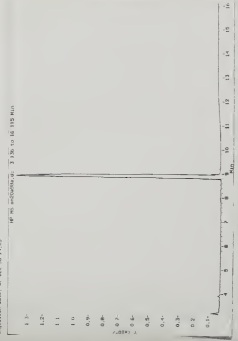
1.84g to  
 dissolve in  
 9 ml IPA.  
 Δ needed.

+ one the (when it is  
 dark?) used, 25% solid  
 + a 6 ml ether  
 heavy white wax - for be  
 wash → after + IPA, then allow  
 white air - stable solids,  
 empty etcher - for →  
 washed heavily, no stir.  
 washed IPA. few →  
 crude w/

0.10g 201 contains  
 GCMS - if same as  
 this, combine &  
 have no reference

0.1g white xstals.  
 I analyzed, same I  
 started pick it out  
 when in the similarity  
 occurred  
 to GCMS as probably

GCMS 201



A. R.

Index

A. R.  
in the  
spring

Albert  
and his

Amey

Some  
times

All the

Amey

From the  
of the

## 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, unrecognised.

Some 46 pages of Drug Code Numbers assigned to drugs, solvents, vitamins, food stuffs, and even things unrecognised.

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

	35.519	
	35.566	
new →	33,800	1.766
	35.519	1.719 weight ↑
	35.466	1.666
	35.455	1.655

page 198  
 0.38g 10 wt% w/v w/ 100 ml  
 + 100 ml H<sub>2</sub>O

0.35g N<sub>2</sub>O<sub>4</sub> when water completely  
 gone

page 219

1.09 g quartz  
 (MSM-2) 40 wt% H<sub>2</sub>O

+ then 3.5 ml H<sub>2</sub>O  
 add 4g in latex + 2g quartz N<sub>2</sub>O<sub>4</sub>

new 1.165 50 wt% H<sub>2</sub>O  
 .6g O<sub>2</sub> ~ 5 ml N<sub>2</sub>O<sub>4</sub>

1000

1000

1000

1000

1000

202

accumulate  soon

1891 B

5.73 g crude -

crude M<sup>o</sup> of!

Δ 98 = 75 me ~~1820~~

AcOH

Δ SB 1/2 in.

fill to just - newing

some wood's!

□ OP RT

3.34 g

25 g polymeral

75 me cis 202

2 1/2 g NH<sub>4</sub>Cl

Δ SB

24 me.

24 me

an. Bng.

- into beak - ▽ cold lab

fillth - wash = a little more



203

Arguing into the "Pharmaparc" available in Pharyngue  
S. NeoDNT piece

[H] pill.

S-NeoDNT piece

H<sup>2</sup> pill.  
dirt yellow

235 mg

lost the quan-  
spray or  
from

white left?

30 mg white solid

to Karmelwin - 444

Signa lot 12343717  
75%

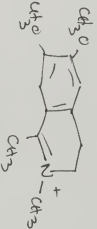
to Hrimad - 444

~~10002~~ lot A J 01

204



DMNH-2



DMNH-2<sup>+</sup> I<sup>-</sup> MW 347

2.70 g DMNH-2 base (69-C - dissolved in

5 mL MeOH - all goes in - off white solution - add

1.2 mL CH<sub>3</sub>I - Δ on edge of SB 15 min → yellow cen-

ter not too deep - faint to deep - off - and white quite

warm scratch with glass rod - eventually → soft - car

takes RT - pale cream xrb - sets up pretty -

sp. - add B the acetone - wash stirring -

↓

3.56 g (shown) pale yellow xrb an active

it looks like it save 100 mg for mp, etc.

has some active solidities - M's + active was too →

wash xrb's - 60 mg

+ 200 mg MeOH

200 mg refolding form U. low MeOH

0.10 mL MeOH

in hot - clean pale yellow xrb.

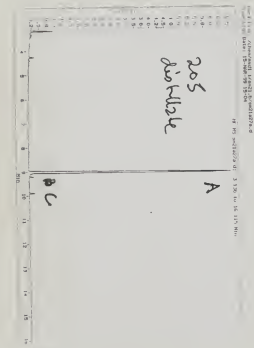
↓

in cold water → in white xrb

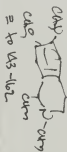
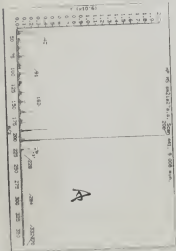
200 → 104 mg white xrb's

save for mp, NMR

204 B



205  
available



≡ to US-462



206



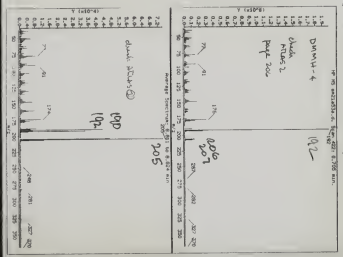
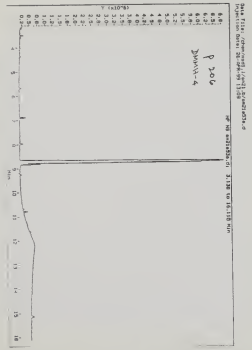
1205

2.6g DMMH-2 (page 165 C) was set in 20 mL water  
 100 mL H<sub>2</sub>O (IT IS WATER SOLUBLE!!!), acidify with  
 1 M HCl (~ 12 mL)  $\rightarrow$  red pH (brown)  
 1 g NaOH. add a bit of lime (good stirring) + 2 hrs  
 as needed to keep the solution acidic. No exoNomin.  
 work  $\bar{c}$  50 mL CH<sub>2</sub>Cl<sub>2</sub>, boil  $\bar{c}$  5% NaOH - extract 2x  
 50 mL CH<sub>2</sub>Cl<sub>2</sub> - stir on R.E.  $\rightarrow$  "1.17g" crude - normally  
 1 mL water the volume, see below - KR 110-120/140g  $\rightarrow$   
 2.13g free base - white oil - on start of redness

picking up CO<sub>2</sub>? GCMS gave A31A (SM2)  
 Same as list of free base A33A  
 base solids - contains CO<sub>2</sub> (206A)

Partition between CH<sub>2</sub>Cl<sub>2</sub>

and 5% NaOH - 3 times -  
 2.24 white oil - brownish 8 mL IPA - 30-32 drops H<sub>2</sub>O  $\rightarrow$   
 on red. no xtds - add ether to turbid - no xtds - repeat 3x on  
 ethanolic - seed - some takes - I feel I am picking too thin?  
 (hydroxide) were after  $\rightarrow$  wash of solids - 10 mL - wash  $\bar{c}$  ether -  
 an dry  $\rightarrow$  2.70 g apocadin white solid - after start to dry, 10 mL;  
 206B active solids!! but the substrate in water!





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 hydride a bit at a time, keeping the Rx mix acidic  $\bar{c}$  5 drops of H<sub>2</sub>O. Work  $\bar{c}$  1x30 ml EtOH, extract  $\bar{c}$  2x40 ml EtOH. Wash basic  $\bar{c}$  5% NaOH, extract  $\bar{c}$  2x40 ml EtOH - flask on R. coat  $\bar{c}$  residue that crystallizes - Distill at KR 110-130 100 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  $\bar{c}$  100 ml H<sub>2</sub>O snow soluble - add 5% base  $\bar{c}$  pH blue - ext 3x40 ml EtOH - flask  $\bar{c}$

0.86 g oil that sets to white xtal - dissolve in 2 ml hot IPA into beaker - vapor  $\bar{c}$  2 ml IPA - clear 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)

814

H.W.

814

H.W.





12

13

14

15

16

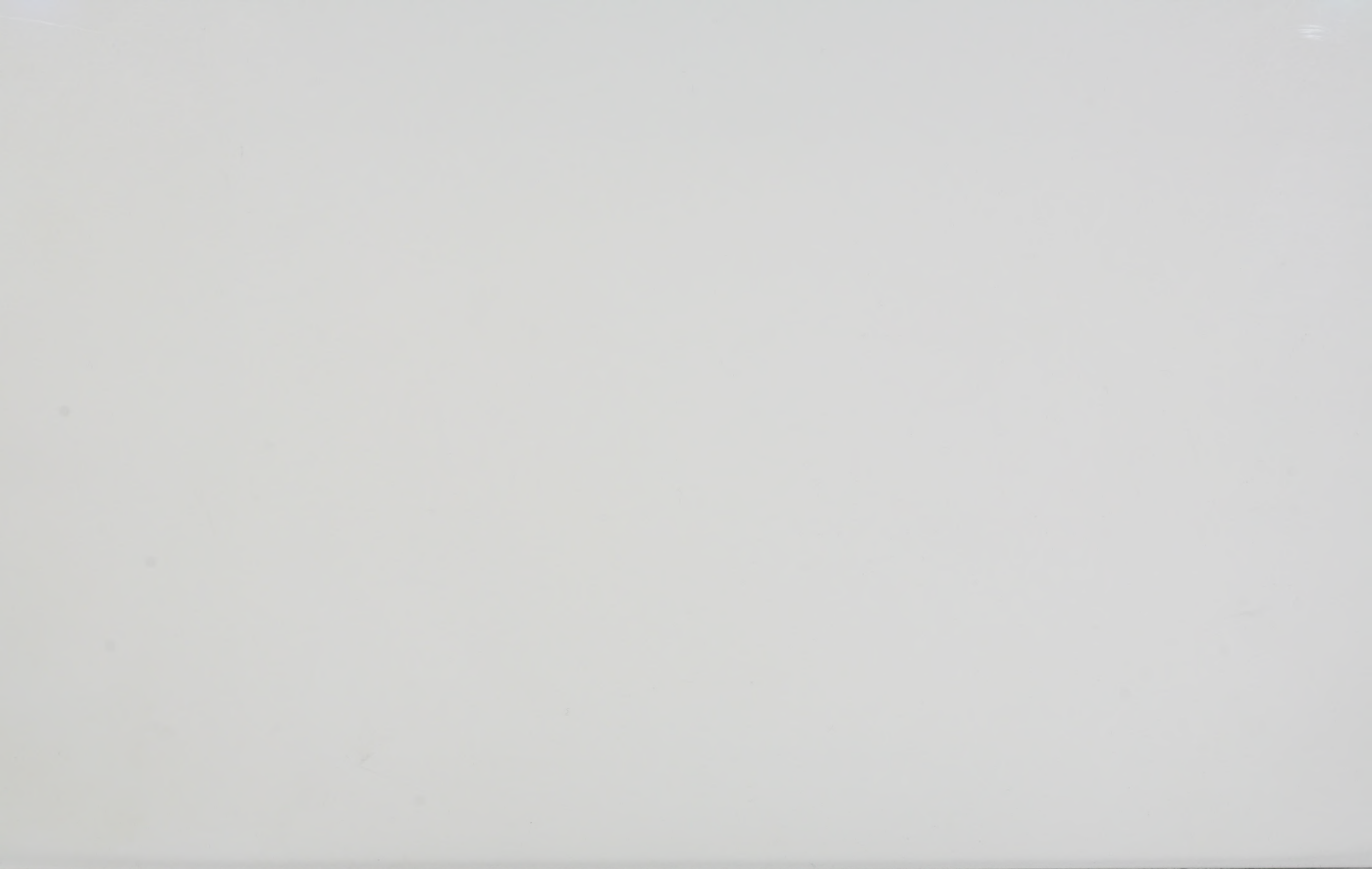
17

18

19

20

21



Handwritten text on a lined notebook page, partially visible at the bottom of the image. The text is written in blue ink and appears to be a list or notes, but it is mostly illegible due to blurring and low contrast. Some faint characters like '1/2', '1/3', and '1/4' are visible.

208



8.2 g Al<sup>1</sup> Squamous (n2 ft fold) - add solution of  
 2.2 g HCl<sub>2</sub> in 300 ml. H<sub>2</sub>O, at RT n 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 207) NS. in 40 ml MeOH  
 40 ml IPA Δ 5h - not in solution.

4th round extraction - 2 ext. H<sub>2</sub>O, 20 ml each, adjust in Al left.  
 add over the white res in PH paper. Try extracting 2 cubes

(addition top phase!!) separate 2 protein pellet - GCMS on  
 these markers see page 193 add 2 cubes 2 cubes volume H<sub>2</sub>O  
 lots water - did not on bottom - extract again → memory

orig. brown 2 25% MeOH - x 4 3 x 75 ml cubes (done in red water)  
 these batches - did not and one after extraction - (pelt - flesh)

in vft. examp. → 1.5 g known oil

90° 0.04

white oil

distill at 40° 90-100° white  
 oil 0.95 g pot left, ~~was~~ even  
 up to 140° - flesh  
 5hr GCMS (208B) → OOT

all - white logs

Synthesized → white solids.  
 Carbazone (208)

nut # 1

1.54 white oil  
 # 2 crude 1.46 g  
 # 3 crude 1.57 g  
 3.33 g

washed for 215  
 washing for 1, 2, 3

used for extraction 215  
 K<sub>2</sub>O 9<sup>10</sup>° → white oil 2.54 g all to ferromide page 226  
 50r  
 pot residue  
 peaks to  
 185°  
 shell pot  
 number

all to ferromide  
 wash vft. at  
 145° C. (208D)  
 carbamate

see page 261

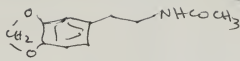
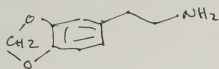
flesh?

2.32 g

same wash

see p 193

(208A)



(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-bromanisole

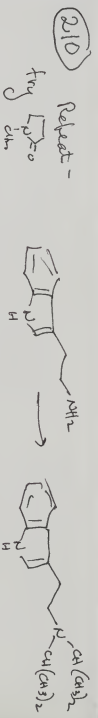
filter - wash w/ H<sub>2</sub>O - kinda dry → 0.92 g off-white solids  
 ag. H<sub>2</sub>O - 2% xht ether - wash acid (1M HCl) f/flash → 0.19 g

distl at KB 165-180° at 0.05 μ. white oil over.

Say 165-175°! shrt. ctals.

0.93 white crystals (209)

0.150 μ.



1100g propylamine  
 10g N-walking pyridone solids

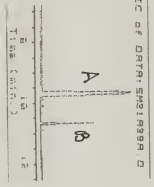
4-5g diisobutyl ethylamine 2 years  
 8.5g: Re I - one phase again (could be sub Glend)

ortho steamover & Rn. n.e. PM darkened - 2 phases?  
 off stream = 19 hrs. very dark - 2 years? K. Add 400 for the 200 wt work -  
~~start~~ in strongly basic - xrt = 3 x 50 wt chcl<sub>2</sub> - all xrt into org.

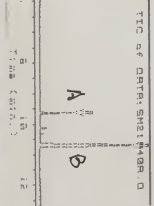
xrt = 2 Dil. HCl 3 x 50 wt (color change in chcl<sub>2</sub>) - pale y.  
 wash 1x = 50 wt chcl<sub>2</sub>, wash eq. waste (color) = 25% Na OH.  
 clarity. xrt 3 x 50 wt water - amber colored - flash. At 80° / 0.05 ju -  
 all the xrt in gone → black heavy oil in pot. At 90° →  
 notice some colorless oil in the receiver - some it, there at  
 90°? - type probably - lots up for Swirls - no small - want br  
 0.4 μm  
 or use P-one 1.2 gr. and more. New receiver.  
**210B** CEMS

140-150° over + 0.76 g pale amber - pot black! out with 4 wt IPA  
 110° standing?  
 distillates slowly - OK + 7 hr little 10 hr wash. darkening  
 came HCl. Spent. xrt + a little 5470. nice white  
 xrt - wash = 2 than / each one. see dry → 0.52 g xrt, 219g yield! wgh

**210 C** #39

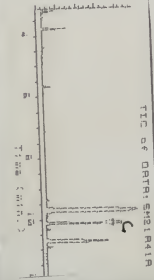


**210 D** #40



ML's 2<sup>nd</sup> crop  
 0.13g white xrt  
**210C**  
**210B** #4

TIC of DBT-1-SM21-R11.R

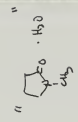
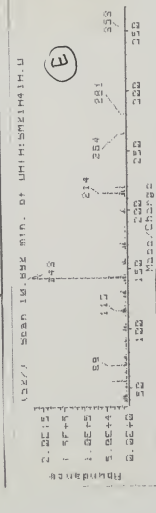
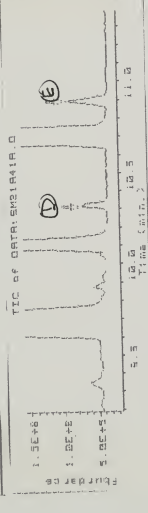
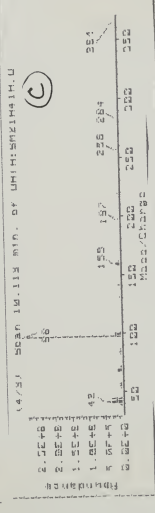
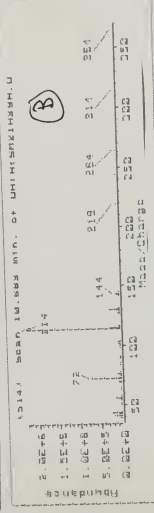
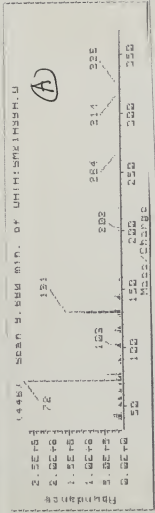




(211)

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Handwritten notes and scribbles at the bottom of the page, including the number 10 and other illegible markings.

SAMPLE FILE

SM-21

REV. 3-2 12-MAY-68

Sample table file name: PROBIOGENESIS

22 Mar 69 4:55 pm

Index, Sample Identification

- 1 DRY 4 FOC C06
- 2 DRY 4 PAGE 047
- 3 #31 DILUTED 05W
- 4 #32 DILUTED 05W
- 5 DIRECTION CONTROL, "CONTROL" 1000/0W
- 6 BIOPROGEN EXTRACT-DRYAL, ETHERALITE 1000/0W
- 7 CONTROL 1800/0W
- 8 SRI 5, PHYLERYTHIN BETA/1
- 9 #301 DRY 4 WITH CERTAINTY
- 10 #304 DRY 4 WITH HIGH PROBABILITY
- 11 CROCK EXTRACT OF BIOPROGENE NUMBER 09 WHITE SEED 200
- 12 #305 DRY 4
- 13 1A DRY
- 14 SEED BAIT
- 15 SUSPECT 5-REP-DHT
- 16 SUSPECT 1-REP-DHC
- 17 PHENYL-SYMBOL-1000
- 18 W-14-100 PAR-02, FISHER
- 19 HELIOMAL HEATING
- 20 RECTONIC 10000/100
- 21 SUSPECT 10000/100 (SEE 16)
- 22 724 EXTRACT, IN 99/19
- 23 506 EXTRACT, IN 99/19
- 24 026 EXTRACT, IN 99/19
- 25 STEM EXTRACT
- 26 TOP EXTRACT
- 27 #302 DRY 4 HELIOMALIS (FROM 1000/1)
- 28 FOC 143 DRY 4 OLD CONTROLS (FROM DRY 4 WITH CROCK)
- 29 16" OF SRIAL-100 3/15/79
- 30 16" OF SRIAL-098 3/15/79
- 31 DRY 4 FOC C06
- 32 DRY 4 PAGE 047
- 33 #31 DILUTED 10W
- 34 #32 DILUTED 10W

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

SM-20

Rev. 3.2 12-May-08

## SAMPLE TABLE

Sample table file name: 20080505005

2 Nov 98 5:40 pm

Index	Sample Identification
1	MECTOL-10
2	MECTOL-14
3	MECTHANE-00
4	MECTHANE-02
5	MECTHANE-03
6	MECTHANE-04
7	MECTHANE-05
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- 51 PICHONOL + CHON  
 52 PHTHALOYL + PHENOL DACTIS  
 53 URETHANE + NBS, + PHTHALOYL, Pd  
 54 1:1:1 CONC. EXTRACT OF UROLOGICAL SKULL CIP  
 55 MONOMETHYLATION OF SEROTONIN H  
 56 DIACETYLATION OF SEROTONIN B  
 57 PHTHALOYL + CHON, C<sub>6</sub>H<sub>5</sub>, C<sub>6</sub>H<sub>5</sub>, 24 HR  
 58 PHTHALOYL + CHON, C<sub>6</sub>H<sub>5</sub>, 24 HR  
 59 MLI + C<sub>6</sub>H<sub>5</sub> + C<sub>6</sub>H<sub>5</sub> + DIMETHYLHYDRAZONE  
 60 MLI + C<sub>6</sub>H<sub>5</sub> + C<sub>6</sub>H<sub>5</sub> + DIMETHYLHYDRAZONE, CH<sub>3</sub>, 3 DAYS  
 61 PHTHALOYL NG WITH NITROBENZENE P 106  
 62 AL (HG) ACETIC ACID REDUCTION NEUTRALS P 187  
 63 AL (HG) ACETIC ACID REDUCTION BASE FRACTION P 187  
 64 6:3 DILUTED 10X  
 65 HYDROLYZED SEROTONIN TREATED WITH CONC. AMMONIAC  
 66 TON ACETATE, NBS, PHTHALOYL, Pd  
 67 TON ACETATE, NBS  
 68 TON ACETATE  
 69 TON ACETATE  
 70 Zn(OAc)<sub>2</sub> REDUCTION  
 71 HYDROLYZED SEROTONIN EX. ACUM - page 189  
 72 HYDROLYZED SEROTONIN AS RECOVERED  
 73 HYDROLYZED SEROTONIN FUMARATE  
 74 PYRROLIDINE + B-URETHANE (Pd)  
 75 Ph-TON  
 76 7:5 DILUTED TEN FOLD  
 77 BENZYL BIRMANE CHON  
 78 MeC<sub>6</sub>H<sub>4</sub>O<sub>2</sub> + HYDRAZONE, 3 DAYS  
 79 MeC<sub>6</sub>H<sub>4</sub>O<sub>2</sub> + HYDRAZONE, 3 DAYS, NABHA/HCHO  
 80 URETHANE/NBS/NABHA/HCHO  
 81 URETHANE/NBS/PAGE 12/Pd  
 82 N,N-DIMETHYL-NOPEA PAGE 195  
 83 N,N-H-2 BASE PAGE 194  
 84 N,N-H-4 BASE PAGE 197  
 85 N,N-H-4 BASE PAGE 198  
 86 NEUTRALS FROM FUMARATE AND POOL: PAGE 194A  
 87 Ph-TON, ACETYLATE  
 88 9:1 HG 0:15 FACITROL  
 89 9:1 HG 0:15 FACITROL  
 90 MeC<sub>6</sub>H<sub>4</sub>O<sub>2</sub> + HYDRAZONE, EXCESS C<sub>6</sub>H<sub>5</sub>, ADDITIONAL CHON

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95 NEUTRALS, P-199 EXTRACT AND BASE AND HR 6.0850

96 EMMA-4 REDUCTION OF 4MHT -- P-201

97 NITROSE REDUCTION, INITIAL

100 NITROSE REDUCTION, OVERNIGHT

91 MeC<sub>6</sub>H<sub>4</sub>OH, ACETATE

92 meta-HYDRAZONE, HCl, EXTS, H<sub>2</sub>O/MeOH

93 5:1:1:1 MeC<sub>6</sub>H<sub>4</sub>O<sub>2</sub> PHN

94 1:1:1 MeC<sub>6</sub>H<sub>4</sub>O<sub>2</sub> PHN

95 HYDROLYZED/SEROTONIN C101

96 NEUTRALS, P-199 EXTRACT AND BASE AND HR 6.0850

97 NEUTRALS, P-199 EXTRACT AND BASE AND HR 6.0850

98 EMMA-4 REDUCTION OF 4MHT -- P-201

99 NITROSE REDUCTION, INITIAL

100 NITROSE REDUCTION, OVERNIGHT

## S A M P L E T A B L E

9419

Rev. 3-2 12-14-69-88

Sample table file name: XXXXXXXXXX

7 Aug 78 6:51 pm

## Index Sample Identification

5

- 1 YELLOW SOLID, TEST FROSTING
  - 2 YELLOW SOLID, BSE FRICTION, ACETIC AMMONIUM PIRIDINE
  - 3 RED SOLID #2, P409 FRICTION IN TOLUENE
  - 4 RED SOLID #3, P409 FRICTION IN 90/10
  - 5 RED SOLID, TEST FROSTING
  - 6 RED SOLID #4, P409 FRICTION ACETIC AMMONIUM
  - 7 REFERENCE HPLS/STACINE, ACETIC AMMONIUM
  - 8 REF. 15/40/10 FROM C. STROBILICUS
  - 9 REF. 15/40/10 FROM C. STROBILICUS
  - 10 1PHIDEN ONE BLOSSOM EXTRACTED 90/10
  - 11 1PHIDEN ONE BULB LEAF, GROUND UP, SPUN, EXTR WITH 90/10
  - 12 MENTHOL FROM DIMETHYL METHOXYPER
  - 13 SPEC FROM DIMETHYL METHOXYPER
  - 14 DIMETHYL METHOXYPER FROM CLOAK AND HERB
- 16 Name Info: DIMETHYLMETHOXYPER 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 DICHLOR  
 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 SRK
- 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, REMAKING OF THE PERCHLORATE -- 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  
 37 Name Info: 35 WASHED WITH AQ. BICARB •  
 38 Name Info: P. HARMALIA SAMPLE B EXTRACT  
 39 Name Info: #39 SAMPLE B DILUTED SEVERAL TIMES  
 40 Name Info: P. HARMALIA SAMPLE A EXTRACT  
 41 Name Info: #41 SAMPLE A DILUTED SEVERAL TIMES  
 43 Name Info: KENNET FROM BARK OF VIROLA THEODORA  
 44 Name Info: 90/10 EXTRACT OF SERUM TUBE #1  
 48 Name Info: 43 AGAIN -- METHANOL AND BASE  
 46 Name Info: 44 DILUTED MUCHO  
 47 Name Info: 90/10 EXTRACT OF SERUM TUBE 2  
 48 Name Info: D-0-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-ETO-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: FU X-87 3,7,5-DIBROMOTRIDEUTEROPYRIDINE (LOOK FOR Br, Cl)  
58 Name Info: FU XII-83 3,7,5-DIBROMOTRIDEUTEROPYRIDINE DIRTY (L.F.Br, Cl)  
59 Name Info: 57 A LOT MORE DILUTE  
60 Name Info: 0.43 G (0.34 G?) SAMPLE FROM KR
- 61 101163 CITRATE 05 10  
62 101163 CITRATE -- BICARBONÄTE  
63 HZ BASE  
64 HZ BASE PHO B44  
65 101163H 1C1 LOT 1001  
66 101163H P-1336  
67 101163H REGIS LOT-239  
68 101163H SIGMA LOT-284-0443  
69 101163H FLURICH LOT 013210W  
70 101163H FLURICH LOT 112269A  
71 101163H SIGMA LOT 1238717  
72 ~~101163H SIGMA LOT 1238717~~

## S A M P L E T A B L E

SW18

Rev. 3.2 12-May-88

Sample table file name: sg00001825

13 Oct 97 7:46 pm

Index Sample Identification  
 1 ACTIVATION OF NERAC B2  
 2 OUFEN/ROSS/SAL/DIENE  
 3 SALS/DIENE DISTILLED  
 4 2-METHYL-5M SOL/DIENE CRUDE  
 5 D-CYCLOHEXYLMACSOH 1 HC/ 4 HC  
 6 Pd40/MASS/VAH 24h  
 7 2-CYMPHOP/DIENE  
 8 NITROTERRIDE - SYNTHETIC  
 9 NITROTERRIDE - TCC12  
 10 NITROTERPENE 1 TRK-5-Ha  
 11 NITRODA FOR RETRIEVAL 07/30/97  
 12 TROD/ETHL VIA TCU/Py/MAI  
 13 TCU  
 14 # B-PROLV-HOHM NEW REAGENT  
 15 # B-PROLV-HOHM OLD REAGENT  
 16 1FD TRODL NHOH OLD REAGENT RECONST AFTER EMP  
 17 # B-PROLV-HOHM OLD REAGENT REBOOT 1 UO/V/L  
 18 # B-PROLV-HOHM OLD REAGENT REBOOT 100 W/V/L  
 19 # B-PROLV-HOHM OLD REAGENT REBOOT 1R W/V/L  
 20 1FD TRODL NHOH OLD REAGENT REBOOT 150 W/V/L EMP/RECONST  
 21 # B-PROLV-HOHM OLD REAGENT REBOOT 1R W/V/L EMP/RECONST  
 22 # B-PROLV-HOHM 1 UO/V/L NARLOS + ET3N 2 HR VORTEX RT  
 23 # B-PROLV-HOHM 1 UO/V/L NARLOS + ET3N 2 HR VORTEX RT  
 24 1FD TRODL NHOH 1 UO/V/L NARLOS + ET3N 2 HR VORTEX RT  
 25 # B-PROLV-HOHM 1 UO/V/L NARLOS + DMAP 2 HR VORTEX RT  
 26 # B-PROLV-HOHM 1 UO/V/L NARLOS + ET3N 2 HR VORTEX RT  
 27 # B-PROLV-HOHM 1 UO/V/L ET3N 2 HR VORTEX RT  
 28 1FD TRODL NHOH 1 UO/V/L PYRIDINE 2 HR VORTEX RT  
 29 # B-PROLV-HOHM 1 UO/V/L DMAP 2 HR VORTEX RT  
 30 1000 + PYRRO/DIENE  
 31 SUALL CIP RECTRL 09/78  
 32 NIFT BR  
 33 PYRIDINERETIC ACID BUTYL ESTER  
 34 SUALL CIP PLANT DICH/OP 09/78  
 35 IODACETYL, CROMS  
 36 PIRIDINEACETIC ACID METHYL ESTER  
 37 PIRIDINE RETRININE (HYBE)  
 38 BASIC ALKALOID EXTRACTION METHYLANA NHOH(SOHL)  
 39 BASIC ALKALOID EXTRACTION TROD/ETHL COND/CL/CL/CL/CL  
 40 BASIC ALKALOID EXTRACTION NITROTERRIDE ALKALOID ex. R08 M.  
 41 NITROTERRIDE  
 42 Pd-CYMPHOP/VAH  
 43 PEGSOLIC COL/SOL/DIENE (SYNTHETIC)  
 44 METHYLATED 1 FOR RETENTION TIMES  
 45 1378 REENT  
 46 RECTOACIC GORMINE FOR TIME  
 47 2-B-1-1000ETHL NR  
 48 2-1-1000ETHL  
 49 Pd40/MASS/VAH  
 50 D-CYCLOHEXYLMACSOH 1 HC/ 4 HC

- 51 Pyrid/mes/NaH, 24h  
 52 3-CYANOPYRIDINE  
 53 NICOTINAMIDE, SYNTHETIC  
 54 NICOTINAMIDE + SOC12  
 55 NICOTINAMIDE + SOC12 SIP  
 56 METHANOL  
 57 METABOLIC NICOTINAMIDE FROM KCL  
 58 METABOLIC NICOTINAMIDE FROM NICOTINIC ACID  
 59 NARLUTININE STANDARD  
 60 NORNICOTINE-D6 SUBSTRATE FROM TLC, KCL  
 61 METABOLIC NICOTINAMIDE FROM KCL  
 62 NICOTINAMIDE STANDARD  
 63 DIBENZOISOLQUINOLINE  
 64 SALSOLINE  
 65 CRISIC EXTRACT OF P.p.  
 66 METHYL NICOTINATE  
 67 ON COLUMN METYLATION OF N.A.  
 68 SALSOLINE VIA ACETIC ACID AND NBBH4  
 69 180CETYL(7),18-D<sub>2</sub>  
 70 PYRINE-AP-S  
 71 NORNICOTINE-D6  
 72 PH-EPoxide/OLYOL VIA NBBH RYN  
 73 PYRINE/GLYCOL VIA NBBH RYN  
 74 PH-EPoxide/OLYOL VIA NBBH RYN  
 75 NORNIC-D2 SYNTH  
 76 METYLATION OF NORNIC D2  
 77 DIBENZOISOLQUINOLINE  
 78 SALSOLINE DISTILLED  
 79 N-METHYLSALSOLINE OXIDE  
 80 NICOTINOLS (P-S PYRINE  
 81 AP BASE AP-S-PYRINE  
 82 TIMING -- P.p.  
 83 TIMING -- N-METHYLSALSOLINE  
 84 HD-DIC #1450  
 85 AP-ENE #1458  
 86 HD-PNE #1468  
 87 ZMITTERUN FRACTION 137C UNDEPIVATIZED  
 88 Z FRACTION 137C WITH ACETIC ANHYDRIDE/PYRIDINE  
 89 SALSUS FROM ACETIC ANHYDRIDE/PYRIDINE  
 90 AP-S tert-PYRINE  
 91 TOLMETHEPHEN : NBBH4  
 92 HUNSON FOR NBBH RYN  
 93 AP-S-NOC  
 94 AP-S-NOC ?  
 95 (P-SOC-NOC ?  
 96 #143 6,7-DIMETHOXY-1,2-DIMETHYL-THIA  
 97 CRUDE HARDOINE  
 98 95 #143 DILUTED 10X  
 99 1. NO EXCH NICOTINIC AND PYRIDINE ACETIC ACIDS, 50:50  
 100

the  
 Page 145A



Chemical Final Recipes: - in red - types up! in green - Nova 4 NMR samples

6,7 Meo 1 Me	6,7-Meo 1-H	6,7 Meo 1 Me	6,7 Meo 1 H
DMPE Acetamide (141) (138)	forward (170) (166)	MDPEA - acetate (209) (145) (215)	MDPEA NS (183) (186) (208) MDPEA (187) (190) (171) DM-MDPEA (195) (208) <del>DM-MDPEA</del> (MDPEA) MDPEA forward (191) (220)
BMMH-2 (140)	DMHH-2 (172) -173, 174, 175, 190,	MDMH-2 (145) (210)	MDHH-2 (194)
DMMM-2+I (204)	DMHH-4 (176)	MDMH-4 (146) (218)	MDHH-4 (197)
DMMM-4 (205)	DMHM-2 (200)	MDMM-2 (211)	MDHM-2 (196)
DMMH-2 (169) (168)	DMHM-4 (201) 171 140		MDHM-4 (198) (23)
DMMH-4 (206)			
of active Sedgeli done (225)			

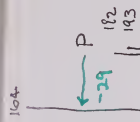
(155)  
(156)



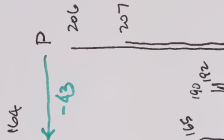


di me moxy

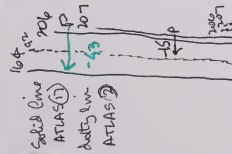
I-H  
1780  
MW 193



I-M 180-201  
MW 207 143



M-I 162 (162) (144)  
Loc MW

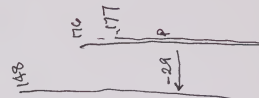


M-M 162-204  
MW 221 143

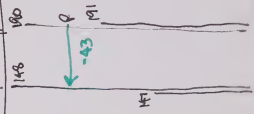


wetly case dioxy

I-H  
MW 177



I-M 198  
MW 191



M-H  
MW 191



M-M  
MW 205

