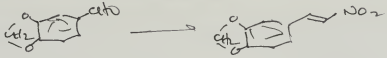
Another try:



(189)

TLC stuff

|| P. p512 2CD
P. p503 2CB

25 g piperonal
75 ml $MeNO_2$ Aldrich 96% pure
Lot 01109 CT 4 liter

endothermic soln.

2.5 g anh. NH_4OAc - not sol.
colorless - on SB 10 AM.

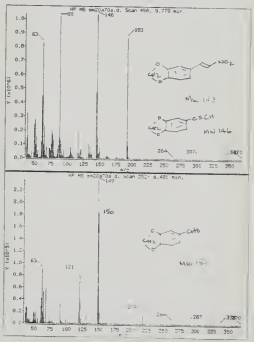
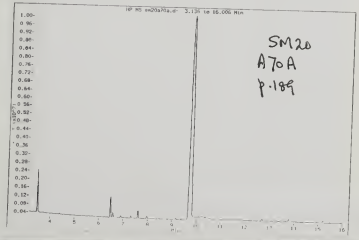
1 hr. turned a dirty yellow - TLC → short xtals
Rf .6 yellow
Rf .5 aldehyde
Rf .4 ? no color

off at 2 1/4 hrs. - into beaker - ▽ to cold water temp → heavy yellow xtals - filter - wash c̄ RT methanol,

air dry → 16.53 g beautiful yellow xtals
(189 A)
small amt. sol
hot HOAc, xtals on ▽,

play c̄ rextal.
small amt 186 A
wash boiling IPA (X)
xtals f. MeOH (Y)
1 1/2 hr pot to RT (Z)

mp stuff.
189 A. 155-8
ex MeOH 158
ex HOAc 161+63

from ML's + washes -
on standing: more xtals vs NS
filter → (189 B)

Piperonyl-NS from HOAc