

11-5-76 Acetylation of Adduct.

wally 11-6-76

## Sample 11-5-1 (Control)

NOTE  
some  
ctgms on  
acetylation  
are filed under  
11-9-76

- Take adduct isolated for MS studies on 9-21-76 - one of the small PS vials - add 50  $\mu$ l dry pyridine (dist. from Barium oxide + stored over 4A ~~dry~~ mol-sieve)
- dissolve by heating

## Sample 11-5-2 (acetyl-derivative)

- harvest w. 1 mg<sup>+</sup> of new dry adduct (11-3-2 batch)
  - add 75  $\mu$ l dry pyridine, heat - didn't dissolve well
  - add 75  $\mu$ l dry acetic anhydride - heat
- The entire sample didn't go into solution, but I think some of it did.  
t: 9:00 PM - let stand in dark at room temperature overnight.

1. Inject 11-5-2  $\rightarrow$  w/o removing pyridine (Ac. Anh. a problem devel. w/ Waters pump - but it looked like a huge peak x.2 before the adducts t<sub>R</sub> and another x.1 after. Both were strongly fluorescent.
2. Inject 11-5-2  $\rightarrow$  take 50  $\mu$ l, evap. solvent away and recon with 100  $\mu$ l MeOH. Inject 25  $\mu$ l.  $\rightarrow$  see same type of ctgm as above.