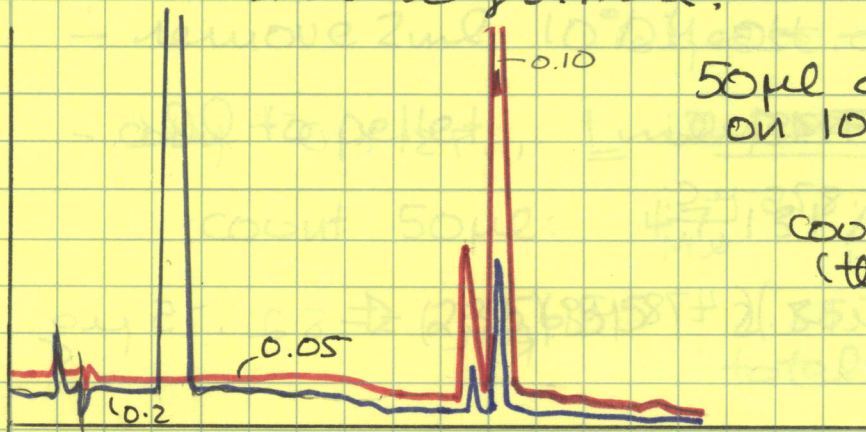


10-27-76 Adduct Purification:

Sample: 10-26-2 - this is the mother liquor from which the crystalline material formed.



50  $\mu$ l of 10-26-2 (sampled on 10-27-76)

counts:  $5034/4 = 1258.5 \text{ cpm}$   
(the vol. of spl. = 20.4  $\mu$ l)

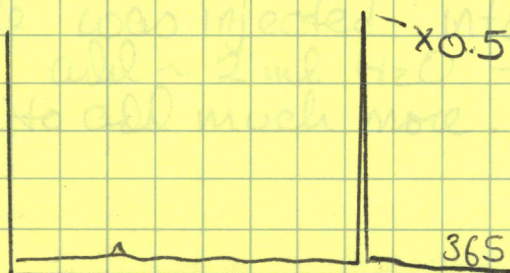
10-27-76 Sample 10-27-1

Dissolving of Pellet of ? Adduct -

- A few crystals (actually quite a bit after centrifugation) were taken from the washings that had been left behind after rinsing flasks containing prep. column effluents.
- add  $\sim$  2  $\mu$ l  $\text{H}_2\text{O}$  - no apparent dissolving, even w/ heating
- $\sim$  3  $\mu$ l  $\text{H}_2\text{O}$  - same
- 0.1 ml MeOH - same
- bring to 1 ml w/ 10% MeOH - sample now is  $\sim$  10% MeOH - heat, centrifuge

LC:

Count: 3613 cpm  
 $\Rightarrow$  36,130 total cpm in sample



sample looks quite pure by 365 and 254

10-27-4

50  $\mu$ l  $\rightarrow$  11,783 cpm

$$\left( \frac{11,783 \text{ cpm}}{.05} \right) (1) = 2.357 \times 10^5 \text{ cpm} \rightarrow 7.141 \times 10^5 \text{ dpm}$$

$$\rightarrow 0.325 \mu\text{Ci}$$

$$\frac{0.325 \mu\text{Ci}}{1.875 \mu\text{Ci}} = 1.731 \times 10^{-1} \mu\text{M}$$

$$1.875 \mu\text{Ci}$$

$$\mu\text{M}$$

$$(1.731 \times 10^{-1} \mu\text{M}) (478 \mu\text{g}(\mu\text{M})) = 82.75 \mu\text{g}$$

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10-27-4a

$$\left( \frac{637 \text{ cpm}}{0.1 \text{ ml}} \right) (1.70 \text{ ml}) \rightarrow 1.083 \times 10^4 \text{ cpm in whole sample}$$

NOTE: this compares very favorably with the 11,783 cpm injected (for a change).

10-27-76 Subject

Instructor's Name

- add another 1 ml of 10% MeOH, reheat  
 count 100  $\mu$ l (of  $\approx$  2 ml)  
 $: 17,180 / 4 = 4295 \text{ cpm} \rightarrow 85,900$   
 cpm in sample  
 (much better than at 1 ml level)

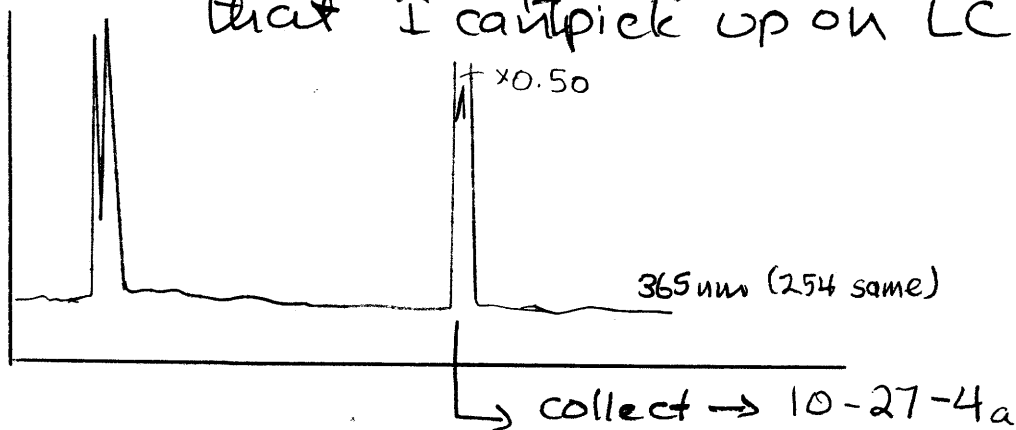
- remove 2 ml 10% MeOH - call this 10-27-3

- add to pellet, 1 ml DMF - heat  $\rightarrow$  DISSOLVED!

count 50  $\mu$ l:  $47,131 / 4 = 11,782.75 \text{ cpm}$

$\Rightarrow 235,655 = 2.357 \times 10^5$  counts in  
 total sample

this gives 82.75  $\mu$ g  $\rightarrow$  note: It looked  
 like much more than this - ? impurity  
 that I can pick up on LC?



100  $\mu$ l  $\rightarrow 2,548 / 4 = 637 \text{ cpm}$

After counting, remainder of sample was  
 pooled with 'adduct stock'.

- the sample was injected into LC several times
- afterwards, add  $\sim$  2 ml H<sub>2</sub>O - no ppt occurred -  
 may have to add much more.