

11-7-76

(actually 11-8-76 - all samples labelled 11-7 are really 11-8)

1. Preparation of Field Desorption M.S. Sample

- SAMPLE 11-7-2-J**
- Sample: the crystals obtained from 11-3-2
 - Transfer most of the crystals into PS vial (tared)
 - evap. off MeOH w/ vacuum
weights dropped from 4.20 mg \rightarrow 3.78 mg \rightarrow 2.96 mg
 - sample given to Al Neffan

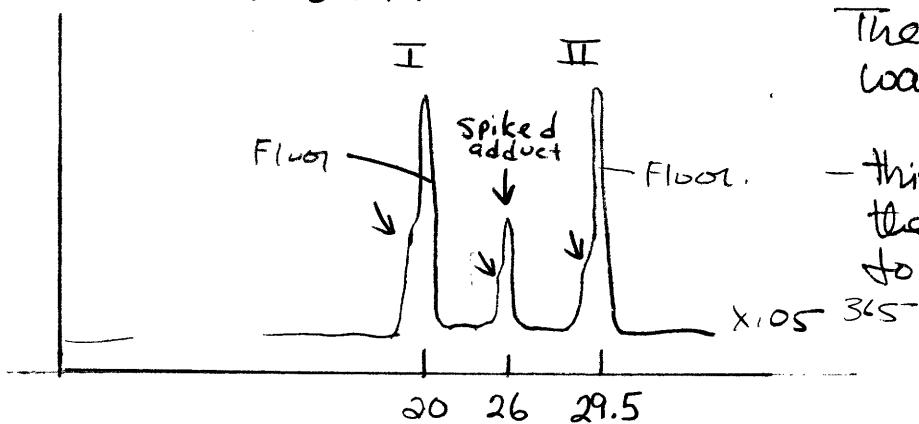
Sample 11-7-1-J

The rest of the crystals were separated into two parts. A few were dissolved in DMF and injected into LC - looked good - only one late-eluting peak at exactly t_R of adduct.

** (Standard used for this was some 10-5-1 which gave nice peak $\times 0.5$ at 26 min)

2. Analysis of Acetylated Reaction Mixture - This sample was described on the previous page.

- Inject 10 μl of MeOH - recon. sample.
- Results



The late-eluting peak was collected!

- this was repeatable - and the adduct was confirmed to be absent from the ctgm.

- probably caused by pyridine