

Subject Adduct MS

Instructor's Name

## 6-22-76 - Preparation of Two Purified Peak-1 Samples

this can be  
quant. exactly  
by radioactivity

- Take  $\frac{1}{2}$  ml of 6-21 and place into (2) Ps vials
- freeze
- place in vac. desiccator (these are 17% MeOH) with aluminum foil caps with holes (not parafilm).

Observation - these samples concentrated very quickly. They almost completely melted in the vacuum desiccator, but they did not bump.

Sample Nomenclature: Duplicates - 6-22-1, 6-22-2

## 6-23-76 Adduct Mass Spectrometry

6-22-1 Sample was transferred with redist. MeOH into a probe. The probe previously was taken to over  $200^{\circ}\text{C}$  in the MS to make sure it was clean.

6-22-2 An attempt was made to form a volatile derivative of the adduct for MS.

- Sample: described above; 75  $\mu\text{l}$  ~~BSA~~ BSA were added and this required 150  $\mu\text{l}$  acetonitrile for solubilization. I don't know whether the adduct dissolved
- heat  $100^{\circ}$  then cool at room T
- dry in vac. oven several hours; First, crystals formed, but these were volatile and evaporated away.
- Sample transferred with 3 acetonitrile (check with Bobroy) into probe - counts were deposited.