

8-4-76 Sample Preparation for MS -

Sample 8-4-1 (425 μ l original volume)

8-4-1-MS take ¹⁵⁰~~200~~ μ l } place in capillary
 8-4-2-MS " " " }

adduct conc = 22.7 ng/ μ l \Rightarrow 3.41 μ g
454 μ g submitted
 to MS in each sample

Purity Check -

The re-purified adduct was submitted to HPLC (5 μ l).
 It was pure with respect to early-eluting material

8-4-76 Search for Missing Counts:

The sample lost \sim 3/4 of the counts between the time it was at the 8.7 ml level and this morning - possibly lost in evaporation? or not soluble in the 10% MeOH used to dissolve the sample after rotary evaporation. (supported by the fact that a residue was observed after removing the liquid from the flask). I then added 400 μ l of MeOH (pure) to the flask, and 4.6×10^4 counts were there (\sim 10% of original sample; therefore not enough to account for the large loss).

- loss could be attrib. to ppt out of adduct as an insoluble salt, or it could have ppt with another contaminating salt.