

6-2-76 Isolation of Large Quantity of Peak 1
 ↳ enz. hydrolysatc

Sample: 100 ml of JE-1 (contains ethanol)

Procedure:

- The sample was rotovapped to ~ 5 ml (T=50°C)
- The contents were then washed into a 15 ml centrifuge tube with 10% MeOH and the total sample (~ 7.5 ml) was centrifuged at high speed in clinical cent. to spin down particles (of what may have been protein)
- The supernatant (not completely clear) was pumped onto the precolumn in two separate runs (~ 3 1/2 and 4 ml, respectively)
- Peak 1 was isolated and the amount was ~ 14 µg!

The 365 went ~ 80% and 70% of full scale on 1AUFs.

- Sample stored in refrigerator

SAMPLE NOMENCLATURE = 6-2-1

- 6-3-76
- place ~ 1 1/2 ml of 6-2-1 into PS vial and reduce pressure
 sample conc. to ~ 50 µg w/o problem.
 - Put the rest of the sample in ~~the~~ PS vial -

- ACCIDENT - ~ 1/3 of sample bumped out of the vial. The liquid was collected with a pasteur pipette and replaced in the sample.
- ~~rotovap~~ sample to near dryness (~ 1/2 - 3/4 ml)
 - transfer with 10% MeOH into cent. tube - prepare sample for precolumn clean-up.

Results: Large adduct peak - ~ 80% on X1.0 (30mm)
 This is less than I expected - it probably represents about 8 µg.