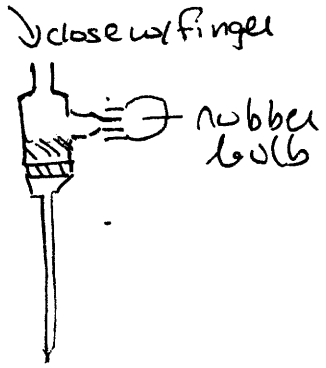
close w/ finger
rubber bulb

11-3-76 Preparation of Adduct for MS and NMR

- Place original ~ 2mg adduct in PS vial and apply into vacuum dessicator.
- place the two MS spls in also (prepared 11-1-76)

11-4-76 - The NMR sample was pumped on until 1:00 PM. It was then taken to Alex Nadzan's Lab where we dissolved it fairly well in d_6-dmso. The insoluble material was filtered off (filter stored in refrig.) The clear filtrate was taken for NMR
- the filtrate turned cloudy over time - this could be reversed by heating - but not permanently.
- The FT-NMR was obtained in 12 min (2 sec pulse interval) relative to tetramethyl-silane. D_2O was then added to see which protons were rapidly exchangable.
- The spectrum generally was interpretable. In fact most of the expected protons were present, and fortunately, the sample's purity probably was good bec. of the integral relationship in the protons observed. The sample appeared to have H_2O, however.

11-4-76 Mass Spectrometry

- take 2 small portions of crystals harvested on 11/3 (Sample 11-3-2).
- dry, add 10 µl pyridine and 10 µl of Verne Reinhold's special silylation mixture (1:1 TMS-Cl : BSTFA)
- heat at 110°C for 1 hr.
- NOTE - sample dissolved almost immediately and then it turned yellow to dark brown — reaction terminated in a few minutes.