

Quantitative Data: 100 μ l of 12-1-1 \rightarrow 10,527 cpm
and there are 2858 cpm of J5-4 Adduct / μ g

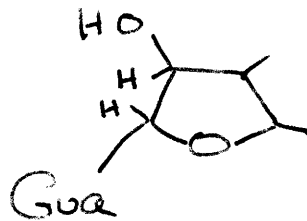
\therefore 368 μ g of Adduct / 10 ml \Rightarrow

$$\frac{36.8 \frac{\mu\text{g}}{\text{ml}}}{479 \frac{\mu\text{g}}{\mu\text{Mole}}} = 0.0768 \frac{\mu\text{mole}}{\text{ml}}$$

$$\boxed{\frac{36.8 \mu\text{g}}{\text{ml}}}$$

$$\downarrow 479 \text{ g/mole}$$

$$0.0768 \frac{\mu\text{mole}}{\text{liter}} = \underline{7.68 \times 10^{-2} \text{ molar}}$$



All samples were frozen for later re-processing. The alkaline sample was first made slightly acid.

11-28-76 Preparation of a Pure, Dry Adduct Sample

Sample 11-3-2 is good-quality adduct and some small portions ~~are~~ have been used in HCl-stability studies by PRD. I want some dry adduct for as a back-up sample for the NMRs to be run in D_2O . DCl.

- put ~ 3/4 of sample into very clean PS vial
- evap. off MeOH under vacuum
- cover w/ foil and dry overnight for ~ 36 hours

Sample: 11-28-1

12-1-76 Preparation of UV Sample

- Scrape crystals of very dry adduct and transfer into 10 ml volumetric
- The particles didn't actually fly around much but they picked up a static charge; it wasn't possible to weigh them, because of a ~~test~~ mechanical problem with the balance.
- dissolve crystals in 0.10 N HCl - crystals slowly went into solution w/o heating.
- this is sample 12-1-1
- obtain UV spectrum
 - ↳ sample was ~ 2x too concentrated, so I dil. 1:1 with 0.1N HCl
- new spectrum: perfect - looked exactly like MeOH/H₂O spectrum
- add 2 drops 10 N NaOH - got very substantial ↓ in all abs (~ factor of 2) - but no bathochromic shift.