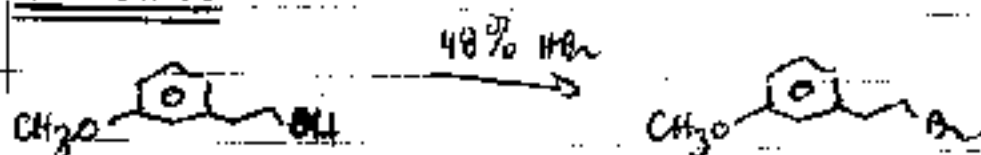


96/7/11

Preparation of $\text{CH}_3\text{O}-\text{C}_6\text{H}_4-\text{CH}_2\text{Br}$ IReactionReagents & Products

MW	Compound	Amount	Mols	Supplier	Description
152.14	3-methoxyphenethyl alcohol	28 g	0.184	ECR 281A	viscous yellow oil
80.92	48% aq. HBr, p1-49	160 mL	1.4	Fisher	water-like, yellow
215.09	3-methoxyphenethyl bromide	40 g ^{theoretical}	0.184		

Reaction

- Set up a 250 mL rbf with 3-methoxyphenethyl alcohol, stirbar and reflux condenser.
- Heated in oil bath until reflux, ^{oil bath @ 90°-100°} ~~refluxed~~ 2 hours.
- Monitored by TLC: $\text{K}_2\text{CO}_3/\text{SiO}_2$, 50% EtOAc/Hex.

- 3-methoxyphenyl acetic acid $R_f: 0.15$ (smear)
- (281A) 3-methoxy β -phenethyl alcohol (crude) $R_f: 0.3$ (one spot)
- (201A) 3-methoxy β -phenethyl bromide (crude) $R_f: 0.5$ (4 spots, color @ 0)

- Rxn over. Let sit for 36 hours.

97/7/13

- Poured into 150 mL H_2O & ice - cautious! Turned white, with brown oily spots.

- Transferred to 1L sep. funnel, 2 layers top white bottom brown.

- Ben flask rinsed 100 mL Et_2O , poured into sep. funnel, shook, 2 layers. Top Et_2O brown. Bottom H_2O light brown.

- Drained off aqueous, organic into clean Erlenmeyer flask.

- Extracted Et_2O aq. 2x 100 mL Et_2O .