DATE 9/25/10 E MAENT Acylation of Anisolo (Lehman) OCH 3 + CH3COCCH3 + AIC/3 CH2C/2 Q CCM3 FW 108.2 102.1 133.3 150. 2 1.152 1.082 3,205 (1,592> mmol 11.0 24.0 10.6 1.1 2.2 d 1.082 anhydride Prep calls for of A1C/3 of anisolo Calé amounts? 133. 29(mol = 2,93269 A1C/3 108.2 g/md = 1.082 g anisole 102,1 g/mol = 1,123 g anhydride 1.152 g anisolo/1082 = 10.6 mmol anisolo 10.6 x 2.2 = 23.3 mmd AIC/3 = 3.105y AIC/3 Actual aint weighed 3. 205 g AIC/3

* Actual aint anhydride - 1.0ml

Theor. 4/8 based on anisol 10.6 mmol x FW = 1.5929 product * denisty anhydride 1.082g/ml 1.0 m L X 1.0829 lm L = 1.0829 of anhydride

Procedure med:

Some r.b. flask fitted with claison adapted and condenser and gas trap, stir bar Add anisole in 5 ml solvent to flask Add 2 mc more solvent 7 ml total so far Add AICI3 in comall portions with microspatula over 1-2 minute period Color of mixture was light green at this point With syringe add anhydride dropwise Add is of anhydride caused color to change Remove 120 bath Reflux 30 minutes Pour RXN mixture onto \$ 109 of ice in Add 5 ml CH2C/2 Mixture was pasty + thick on ice extra CH2C/2 helped dissolve paste Transfer to sep finnel and wash with \$ 10ml NaOH Separate layers, wash with brine dry with mg504

Proper organic layer there glass wood plug in to taxed wall a remove so went with Crude mass 1,1259 (710/6)