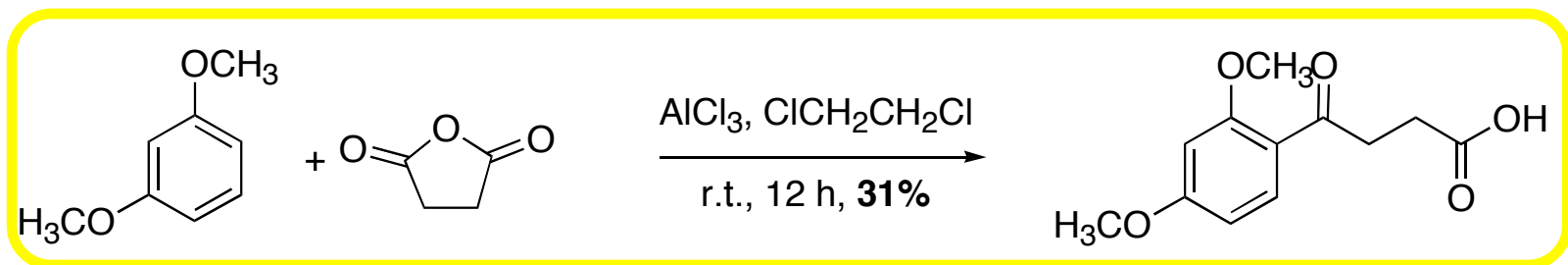


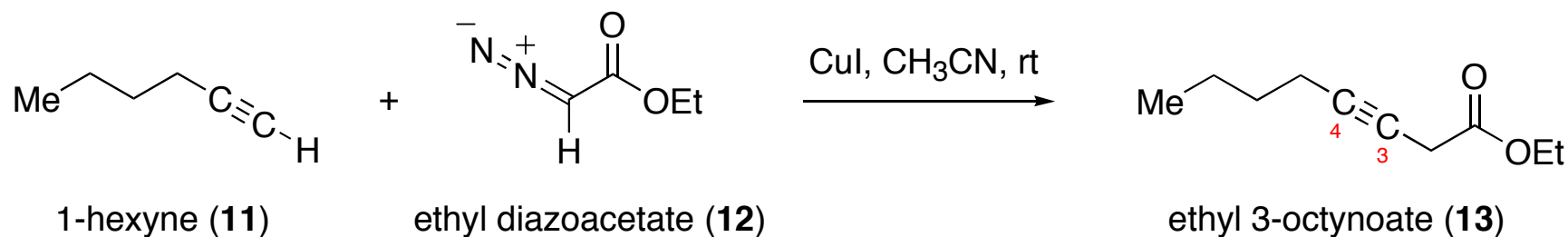
# Interpreting an Experimental Procedure

## 4-(2,4-Dimethoxyphenyl)-4-oxo-butyric acid



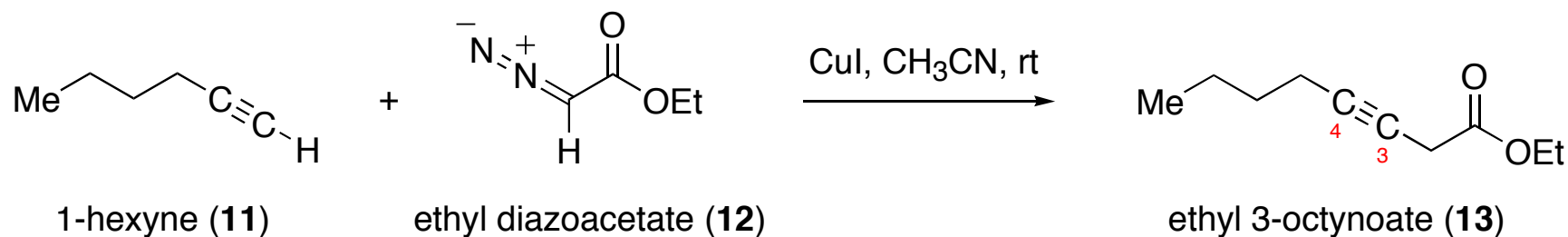
To a solution of  $\text{AlCl}_3$  (6.63 g, 49.2 mmol, 1.30 eq) in 1,2-dichloroethane (200 mL) at 0 °C was added succinic anhydride (3.90 g, 37.8 mmol, 1.0 eq) under an atmosphere  $\text{N}_2$ . 1,3-dimethoxy benzene (5.0 mL, 37.8 mmol, 1.0 eq) was added via syringe, the reaction mixture stirred at 0 °C for 1 h then allowed to warm to r.t. and stirred an additional 12 h. The reaction mixture was poured into ice, the aqueous and organic layers separated and the aqueous layer extracted with  $\text{Et}_2\text{O}$  (3 x 50 mL). The combined organic extracts were dried ( $\text{Na}_2\text{SO}_4$ ), filtered and concentrated in vacuo then recrystallized (MeOH) to provide **4-(2,4-Dimethoxy-phenyl)-4-oxo-butyric acid** (2.85 g, 31%) as a yellow oil:  $R_f$  0.10 (1:1, EtOAc/hexanes);  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ) 7.89 (d,  $J = 8.8$  Hz, 1 H), 6.54 (dd,  $J = 8.3, 2.3$  Hz, 1 H), 6.46 (d,  $J = 2.3$  Hz, 1 H), 3.90 (s, 3 H), 3.86 (s, 3 H), 3.30 (t,  $J = 6.6$  Hz, 2 H), 2.73 (t,  $J = 6.6$  Hz, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 197.8, 178.7, 165.0, 161.5, 133.2, 120.2, 105.5, 98.5, 55.8, 55.7, 38.7, 28.9; FTIR (film)  $\bar{\nu}_{\text{max}}$  3437, 1707, 1655, 1606, 1420  $\text{cm}^{-1}$ .

# Constructing Your Reaction Table



compound	M.W. (g/mol)	d (g/mL) or M (mmol/mL)	Rxn weight or volume (include unit)	<b>You calculate!</b>	<b>From lit. procedure</b>
				mmol or M	equivalents (or concentration)
CuI (catalyst)	190.45	—	g		0.05
CH <sub>3</sub> CN (acetonitrile)	41.05	d = 0.786	mL		0.10 M
1-hexyne (11) <i>only 97%!</i>	82.14	d = 0.715	mL		1.05 eq
ethyl diazoacetate (12) <i>only 85%!</i>	114.10	d = 1.085	2.35 mL		1.00 eq (limiting rgt)

# Constructing Your Reaction Table



compound	M.W. (g/mol)	d (g/mL) or M (mmol/mL)	Rxn weight or volume (include unit)	You calculate!	You calculate!
				mmol or M	equivalents (or concentration)
CuI (catalyst)	190.45	–	0.250 g		
CH <sub>3</sub> CN (acetonitrile)	41.05	d = 0.786	30 mL		
1-hexyne (11) only 97%!	82.14	d = 0.715	5.0 mL		
ethyl diazoacetate (12) only 85%!	114.10	d = 1.085	4.0 mL		