1,3-Diketones from Acid Chlorides: A Rapid and General One-Pot Synthesis of Pyrazoles

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Supporting Information

Table of Contents

General Procedure S3
General Experimental Information S3

Compound Data:
5, 6a,b S4
7, 8, 9 S5
10, 12, 13 S6
14, 15, 16 S7
17, 19, 20 S8
21a,b, 22 S9
23, 24, 25 S10

NMR spectra:
7 \( ^1\text{H} \) S12
7 \( ^1\text{H} \) \( ^{13}\text{C} \) S13
8 \( ^1\text{H} \) S14
8 \( ^1\text{H} \) \( ^{13}\text{C} \) S15
9 \( ^1\text{H} \) S16
9 \( ^1\text{H} \) \( ^{13}\text{C} \) S17
10 \( ^1\text{H} \) S18
10 \( ^1\text{H} \) \( ^{13}\text{C} \) S19
5 \( ^1\text{H} \) S20
5 \( ^1\text{H} \) \( ^{13}\text{C} \) S21
<table>
<thead>
<tr>
<th>Carbon</th>
<th>Hydrogen</th>
<th>Isotopes</th>
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**General Procedure:** Ketone (2 mmol) was dissolved in 5 mL dry toluene in a screw cap vial (with septum) and then the solution was cooled to 0°C under nitrogen. LiHMDS (2.1 mL, 1.0 M in THF, 2.1 mmol) was added quickly via syringe with agitation stirring and the formed anion was allowed to sit for approximately 1 minute before the addition via syringe\(^1\) of acid chloride (1 mmol) in one portion with stirring. The vial was then removed from the ice bath and allowed to stand for 1 minute, then 2 mL AcOH was added with stirring. 10 mL EtOH and 5 mL THF was added to form a homogeneous mixture, and then hydrazine hydrate (2 mL, 1.1 g, 34.3 mmol) or substituted hydrazine (5 mmol) was added and the mixture was allowed to auto-reflux and was held at that temperature for 5 minutes, when LCMS showed that all diketone had reacted. The resulting solution was added to 1.0 M NaOH solution and extracted with EtOAc. The organic fraction was then washed with brine, dried over Na\(_2\)SO\(_4\), and evaporated under reduced pressure. See specific compounds for purification details.

**General Comments:** All reagents were purchased from Aldrich and were used as received. LiHMDS solutions were rigorously kept under a nitrogen atmosphere after opening. Dry toluene and THF were supplied by Acros. All other solvents were provided by Fisher Scientific. All chemistry was performed under a nitrogen atmosphere using standard techniques. NMR spectra were taken with a Varian Inova (400 MHz or 500 MHz). LC/MS were obtained using a Agilent 1100 series LC and Micromass Zq spectrometer. Column chromatography was performed using a Biotage Horizon system utilizing standard pre-packed silica gel cartridges (40g).

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\(^1\) Solid acid chlorides were dissolved in 2 mL toluene and the minimum amount of THF required to make a solution.
**Spectroscopic Notes:** All spectra calibrated with TMS, or in the case of $^{13}$C experiments, the solvent peak (CDCl$_3$ = 77.0 ppm, d$_6$-DMSO = 39.5 ppm). The room temperature $^1$H NMR spectra of 11 and 20 showed two distinct tautomers. In both cases, the relevant peaks converged substantially. Reported data is taken from experiments run at 70°C. This effect was also observed in the $^{13}$C NMR for 9, for which the reported data corresponds to the major tautomer. In a few cases quaternary carbons were not detected in $^{13}$C NMR experiments.

![5-(4-bromophenyl)-3-(4-methoxyphenyl)-1H-pyrazole (5)](image)

5-(4-bromophenyl)-3-(4-methoxyphenyl)-1H-pyrazole (5). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, d$_6$-DMSO) $\delta$ 13.30 (s, 1H), 7.80 (d, $J$ = 7.6, 2H), 7.75 (d, $J$ = 8 Hz, 2H), 7.64 (d, $J$ = 7.6 Hz, 2H), 7.11 (s, 1H), 7.03 (d, $J$ = 8 Hz, 2H), 3.80 (s, 3H); $^{13}$C NMR (500 MHz, d$_6$-DMSO) $\delta$ 159.1, 131.7, 127.0, 126.5, 120.7, 114.3, 99.0, 55.1; HRMS calcd for C$_{16}$H$_{14}$BrN$_2$O, 331.0271; found, 331.0280.

![3-(4-bromophenyl)-5-(4-methoxyphenyl)-1-methylpyrazole (6a)](image)

3-(4-bromophenyl)-5-(4-methoxyphenyl)-1-methylpyrazole (6a). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.73 (d, $J$ = 8.8 Hz, 2H), 7.55 (d, $J$ = 8.4 Hz, 2H), 7.27 (d, $J$ = 8.8 Hz, 2H), 6.92 (d, $J$ = 8.4 Hz, 2H), 6.49 (s, 1H), 3.83 (s, 3H), 3.78 (s, 3H); MS calcd for C$_{17}$H$_{15}$BrN$_2$O, 343.04; found, 343.06.

![5-(4-bromophenyl)-3-(4-methoxyphenyl)-1-methylpyrazole (6b)](image)

5-(4-bromophenyl)-3-(4-methoxyphenyl)-1-methylpyrazole (6b). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.68 (d, $J$ = 8.8 Hz, 2H), 7.49 (d, $J$ = 8.4 Hz, 2H), 7.34 (d, $J$ = 8.8 Hz, 2H), 6.96 (d, $J$ = 8.4 Hz, 2H), 6.49 (s, 1H), 3.84 (s, 3H), 3.81 (s, 3H); C$_{17}$H$_{15}$BrN$_2$O, 343.04; found, 343.08.
3-(4-bromophenyl)-5-pentyl-1H-pyrazole (7). Crystallized from 2-propanol/water. 

\(^{1}\text{H NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 7.59 (d, \(J = 8.4\) Hz, 2H), 7.47 (d, \(J = 8.4\)), 6.31 (s, 1H), 2.59-2.53 (m 2H), 1.61-1.58 (m, 2H), 1.30-1.28 (m, 4H), 0.88 (t, 3H); \(^{13}\text{C NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 131.7, 127.2, 121.6, 100.9, 31.4, 28.8, 26.1, 22.4, 13.9; \(\text{HRMS}\) calcd for C\(_{14}\)H\(_{18}\)BrN\(_2\), 295.0634; found, 295.0644.

3-(4-bromophenyl)-5-(5-chloropentyl)-1H-pyrazole (8). Crystallized from 2-propanol/water. 

\(^{1}\text{H NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 7.59 (d, \(J = 8.4\) Hz, 2H), 7.49 (d, \(J = 8.4\)), 6.33 (s, 1H), 3.51 (t, 2H), 2.63-2.60 (m 2H), 1.80-1.73 (quint, 2H), 1.70-1.62 (quint, 2H), 1.50-1.44 (m, 2H); \(^{13}\text{C NMR}\) (500 MHz, CDCl\(_3\)) \(\delta\) 131.7, 127.2, 121.8, 101.1, 44.8, 32.2, 28.4, 26.4, 26.0; \(\text{HRMS}\) calcd for C\(_{14}\)H\(_{17}\)BrClN\(_2\), 329.0242; found, 329.0240.

5-(4-bromophenyl)-3-(4-cyanophenyl)-1H-pyrazole (9). Recrystallized from 2-propanol/water. 

\(^{1}\text{H NMR}\) (400 MHz, d\(_6\)-DMSO) \(\delta\) 13.71 (s, 1H), 8.04-7.92 (m, 4H), 7.79-7.70 (m, 4H), 7.42 (s, 1H); \(^{13}\text{C NMR}\) (500 MHz, d\(_6\)-DMSO) \(\delta\) 149.8, 142.8, 137.9,
132.7, 132.0, 128.2, 127.1, 125.6, 121.4, 119.0, 109.8, 101.1; **HRMS** calcd for C$_{16}$H$_{11}$BrN$_3$, 326.0117; found, 326.0107

![Chemical structure of 5-(2-methylphenyl)-3-(4-cyanophenyl)-1H-pyrazole (10).](image1)

**5-(2-methylphenyl)-3-(4-cyanophenyl)-1H-pyrazole (10).** Crystallized from 2-propanol/water. **$^1$H NMR** (400 MHz, d$_6$-DMSO, T = 70°C ) δ 12.97 (s, 1H), 7.88-7.75 (b, 2H), 7.67-7.58 (b, 2H), 7.46 (b, 1H), 7.36-7.20 (b, 3H), (bd, 6.92-6.84, 1H), 2.41 (s, 3H); **$^{13}$C NMR** (400 MHz, d$_6$-DMSO) δ 149.5, 142.9, 135.5, 133.0, 131.9, 131.5, 130.8, 129.4, 128.7, 128.4, 127.1, 126.0, 120.3, 20.5; **HRMS** calcd for C$_{16}$H$_{14}$BrN$_2$, 315.0321; found, 315.0324.

![Chemical structure of 3-(4-((N,N-dimethylamino)phenyl)-5-(4-methoxyphenyl)-1H-pyrazole (12).](image2)

**3-(4-(N,N-dimethylamino)phenyl)-5-(4-methoxyphenyl)-1H-pyrazole (12).** Crystallized from 2-propanol/water. **$^1$H NMR** (400 MHz, d$_6$-DMSO) δ 12.92 (s, 1H), 7.75-7.62 (m, 4H), 6.99 (d, 2H), 6.86 (s, 1H), 6.77 (d, $J$ = 8 Hz, 2H), 3.79 (s, 3H), 2.94 (s, 6H); **$^{13}$C NMR** (500 MHz, d$_6$-DMSO) δ 158.8, 150.0, 126.4, 126.0, 114.1, 112.3, 97.3, 55.1, 40.0; **HRMS** calcd for C$_{18}$H$_{20}$BrN$_3$O, 294.1606; found, 294.1606.

![Chemical structure of 3-(4-cyanophenyl)-5-(4-methoxyphenyl)-1H-pyrazole (13).](image3)

**3-(4-cyanophenyl)-5-(4-methoxyphenyl)-1H-pyrazole (13).** Recrystallized from toluene. **$^1$H NMR** (400 MHz, d$_6$-DMSO) δ 13.47 (s, 1H), 8.04 (d, $J$ = 7.6 Hz, 2H), 7.90 (d, $J$ = 7.6, 2H), 7.76 (d, $J$ = 8.0 Hz, 2H), 7.25 (s, 1H), 7.05 (d, $J$ = 8 Hz, 2H), 3.81 (s, 3H), 3.58 (s, 3H); **HRMS** calcd for C$_{19}$H$_{20}$BrN$_3$O, 313.0384; found, 313.0387.
5-(4-chlorophenyl)-3-(4-methoxyphenyl)-4-phenyl-1H-pyrazole (14). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, $d_6$-DMSO) $\delta$ 13.36 (s, 1H), 7.34-7.16 (m, 11H), 6.91-6.83 (m, 2H), 3.74 (s, 3H); $^{13}$C NMR (400 MHz, $d_6$-DMSO) $\delta$ 159.0, 147.7, 140.4, 133.7, 131.7, 130.5, 128.9, 128.7, 128.6, 128.1, 127.1, 121.7, 116.2, 114.0, 113.5, 55.1; HRMS calcd for C$_{122}$H$_{18}$ClN$_2$O, 361.1108; found, 361.1111.

5-(4-chlorophenyl)-3-(4-methoxyphenyl) 4-propyl-1H-pyrazole (15). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, $d_6$-DMSO) $\delta$ 13.00 (s, 1H), 7.67-7.46 (m, 6H), 7.09-7.03 (m, 2H), 3.81 (s, 3H), 2.64 (t, 2H), 1.41-1.35 (hex, 2H), 0.78 (t, 3H); $^{13}$C NMR (400 MHz, $d_6$-DMSO) $\delta$ 159.0, 148.5, 140.6, 133.8, 131.7, 129.4, 129.0, 128.8, 128.4, 122.6, 114.2, 55.1, 25.3, 23.4, 13.9; HRMS calcd for C$_{19}$H$_{20}$ClN$_2$O, 327.1264; found, 327.1257.

3-(4-methoxyphenyl)-5-(4-nitrophenyl)-1H-pyrazole (16). Crystallized from 2-propanol/water. $^1$H NMR (400 MHz, $d_6$-DMSO) $\delta$ 13.60 (s, 1H), 8.30 (d, $J = 8$ Hz, 2H),
8.12 (d, \( J = 8 \) Hz, 2H), 7.76 (d, \( J = 8 \) Hz, 2H), 7.29 (s, 1H), 7.06 (d, \( J = 8 \) Hz, 2H), 3.81 (s, 3H); \(^{13}\text{C} \text{NMR} \) (400 MHz, \( \text{d}_6\text{-DMSO} \)) \( \delta \) 159.4, 146.3, 126.7, 125.8, 124.1, 100.0, 55.2; \text{HRMS} \) calcd for \( \text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_3 \), 296.1035; found, 296.1019.

5-(4-methoxyphenyl)-3-(4-nitrophenyl)-1\( H \)-pyrazole (17). Purified by column chromatography (hexanes:ethyl acetate, 19:1). \(^1\text{H} \text{NMR} \) (400 Mhz, \( \text{CDCl}_3 \)) \( \delta \) 8.30 (d, \( J = 7.2 \) Hz, 2H), 8.08 (d, \( J = 7.2 \) Hz, 2H), 7.38 (m, 5H) 7.20 (d, \( J = 8.8 \) Hz, 2H), 6.87 (d, \( J = 8.8 \) Hz, 2H), 6.86 (s, 1H), 3.82 (s, 3H); \text{MS} \) calcd for \( \text{C}_{22}\text{H}_{17}\text{N}_3\text{O} \), 372.13; found, 372.04. \text{m.p.} 176-178°C (Lit. 178-179°C)\(^2\)

3-(4-pyridyl)-5-(4-methoxyphenyl)-1\( H \)-pyrazole (19). Crystallized from ethanol/water. \(^1\text{H} \text{NMR} \) (400 MHz, \( \text{d}_6\text{-DMSO} \)) \( \delta \) 13.53 (s, 1H), 8.63 (d, \( J = 4.8 \) Hz, 2H), 7.81 (d, \( J = 4.8 \) Hz, 2H), 7.77 (d, \( J = 8 \) Hz, 2H), 7.28 (s, 1H), 7.06 (d, \( J = 8 \) Hz, 2H), 3.81 (s, 3H); \(^{13}\text{C} \text{NMR} \) (400 MHz, \( \text{d}_6\text{-DMSO} \)) \( \delta \) 159.2, 150.2, 126.6, 119.4, 114.4, 99.9, 55.2; \text{MS} \) calcd for \( \text{C}_{15}\text{H}_{14}\text{N}_3\text{O} \), 252.11; found, 252.18.

3-(2-thienyl)-5-(4-methoxyphenyl)-1\( H \)-pyrazole (20). Crystallized from 2-propanol/water. \(^1\text{H} \text{NMR} \) (400 MHz, \( \text{d}_6\text{-DMSO} \), \( T = 70°C \)) \( \delta \) 12.98 (s, 1H), 7.71 (d, \( J = 6 \) Hz, 2H), 7.40 (b, 2H), 7.09 (s, 1H), 7.01 (d, \( J = 6.8 \) Hz, 2H), 6.83 (s, 1H), 3.80 (s, 3H); \(^{13}\text{C} \text{NMR} \) (400 MHz, \( \text{d}_6\text{-DMSO} \)) \( \delta \) 159.2, 146.8, 143.3, 137.1, 127.5, 126.6, 124.5,

123.5, 121.7, 114.4, 98.5, 55.2; HRMS calcd for C_{14}H_{13}N_{2}OS, 257.0749; found, 257.0742.

5-(4-methylphenyl)-3-phenyl-1-methylpyrazole (21a). Purified by column chromatography (hexanes:ethyl acetate, 24:1). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J$ = 7.2 Hz, 2H), 7.41-7.25 (m, 7H), 6.58 (s, 1H), 3.92 (s, 3H), 2.42 (s, 3H); $^3$ MS calcd for C$_{17}$H$_{16}$N$_2$, 249.13; found, 249.32.

3-(4-methylphenyl)-5-phenyl-1-methylpyrazole (21b). Purified by column chromatography (hexanes:ethyl acetate, 24:1). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.73 (d, $J$ = 8.4 Hz, 2H), 7.48-7.45 (m, 5H), 7.22 (d, $J$ = 8.4 Hz, 2H), 6.58 (s, 1H), 3.92 (s, 3H), 2.37 (s, 3H); $^3$ MS calcd for C$_{17}$H$_{16}$N$_2$, 249.13; found, 249.27.

3-(4-methoxyphenyl)-4,5,6,7-tetrahydro-1H-indazole (22). Flash chromatography (2:1, dichloromethane:acetone). Analytical sample crystallized (0°C) from dichloromethane/hexanes. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.55 (d, $J$ = 8.8 Hz, 2H), 6.90 (d, $J$ = 8.8 Hz, 2H), 3.82 (s, 3H), 2.68 (t, 2H), 2.63 (t, 2H), 1.82-1.76 (m, 4H); $^{13}$C NMR (400 MHz, CDCl$_3$) $\delta$ 158.9, 127.8, 125.5, 114.0, 112.4, 55.2, 28.4, 23.6, 22.3, 22.0; MS calcd for C$_{14}$H$_{17}$N$_2$O, 229.13; found, 229.37.

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3-pentyl-4,5,6,7-tetrahydro-1\textit{H}-indazole (23). Flash chromatography (2:1, dichloromethane : acetone). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.64 (t, 2H), 2.55 (t, 2H), 2.43 (t, 2H), 1.76 (quint, 4H), 1.61 (m, 2H), 1.31 (m, 4H), 0.88 (t, 3H); $^{13}$C NMR (400 MHz, CDCl$_3$) $\delta$ 144.3, 112.3, 31.6, 28.6, 25.8, 23.4, 23.0, 22.42, 22.4, 20.2, 13.9; MS calcd for C$_{12}$H$_{21}$N$_2$, 193.17; found, 193.67.

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\textit{tert}-butyl 3-(4-methoxyphenyl)-1,4,6,7-tetrahydro-5\textit{H}-pyrazolo[4,3-c]pyridine-5-carboxylate (24). Flash chromatography (3:2, dichloromethane : acetone). Analytical sample crystallized from 2-propanol/water. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.46 (d, $J$ = 8.8 Hz, 2H), 6.90 (bd, 2H), 4.62 (s, 2H), 3.81 (s, 3H), 3.70 (b, 2H), 2.73 (t, 2H), 1.48 (s, 9H); $^{13}$C NMR (400 MHz, CDCl$_3$) $\delta$ 159.3, 155.0, 127.5, 114.3, 109.8, 80.1, 55.3, 41.6, 28.4; MS calcd for C$_{18}$H$_{24}$N$_3$O$_3$, 330.18; found, 330.17.

\begin{center}
\includegraphics[width=0.5\textwidth]{image}
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3-(4-methoxyphenyl)-1,4,6,7-tetrahydropyrano[4,3-c]pyrazole (25). Flash chromatography (3:2, dichloromethane : acetone). Analytical sample crystallized from dichloromethane/hexanes. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39 (d, $J$ = 8.8 Hz, 2H), 6.95 (d, $J$ = 8.8 Hz, 2H), 4.88 (s, 2H), 3.98 (t, 2H), 3.84 (s, 3H), 2.83 (t, 2H); $^{13}$C NMR (400 MHz, CDCl$_3$) $\delta$ 159.3, 155.0, 127.5, 114.3, 109.8, 80.1, 55.3, 41.6, 28.4; MS calcd for C$_{18}$H$_{24}$N$_3$O$_3$, 330.18; found, 330.17.
MHz, CDCl₃) δ 159.5, 127.3, 123.9, 114.4, 111.0, 64.5, 64.2, 55.3, 23.7; MS calcd for 
C₁₃H₁₅N₂O, 231.11; found, 230.96.
NMR Spectrum: Cl3
Instrument: Varian Inova400
Solvent: DMSO Temp: 25.0

S27
\[ \text{HN-N} \text{-} \text{NMe} \text{-} \text{OMe} \]
NMR Spectrum: H1
Instrument: Varian inova400
Solvent: DMSO Temp: 25.4
ISID: bellerat Date: Feb 27 2006
NMR spectrum: H1
Instrument: Varian inova400
Solvent: CDCl3 Temp: 25.6
ISID: hellerat Date: Feb 14 2006

ppm
HN-N
N
Boc

OMe