Supporting Information
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Supporting Information

An Expedient Direct Three-Component Approach for the Synthesis of 4-Thioarylpyrroles

Venkatachalam Rajeshkumar*, Chinnaraj Neelamegam, and Sambandam Anandan

Department of Chemistry, National Institute of Technology Tiruchirappalli, Tamil Nadu, India-620015
E-mail: orgrajeshkumar@gmail.com; vrajesh@nitt.edu

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General procedure for synthesis of 1, 4-enediones

A mixture of aryl methyl ketones (1.0 mmol), 1, 3-dicarbonyl compound (1.0 mmol), iodine (1.1 mmol), and CuO (1.1 mmol) in 10 mL of DMSO was stirred at 70 °C. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was extracted with CH₂Cl₂ (3×20 mL). The extract was washed with aqueous Na₂S₂O₃ and brine solution. After drying over Na₂SO₄ and evaporation, the crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc) to afford the E/Z mixtures of 1, 4-enediones. Further recrystallization from ethanol/petroleum ether gave (E)-isomer as a pure major compound.

Synthesis of compound 1n:
The compound 1n was synthesized by using the literature procedure.

To the reaction tube was added 1-(p-tolyl)ethan-1-one (134.2 mg, 1.0 mmol), CuBr₂ (45 mg, 0.2 mmol), I₂ (508 mg, 2 mmol) and dry DMF (1 mL). Then the mixture was heated to 80 °C. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was extracted with CH₂Cl₂ (3×20 mL). The extract was washed with aqueous Na₂S₂O₃ and brine solution. After drying over Na₂SO₄ and evaporation, the crude product was purified by column chromatography on silica gel to afford the desired enedione 1n as a yellow solid in 190 mg, 72%. 
Spectroscopic and physical data of 1, 4-enediones

Ethyl (E)-2-benzoyl-4-oxo-4-phenylbut-2-enoate\(^1\)

![Structure 1a]

FT-IR (KBr) \(\nu\) (cm\(^{-1}\)) 3066, 2982, 1724, 1667, 1449, 1366, 1007.

\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.15 (s, 1H), 8.00 – 7.87 (m, 4H), 7.65 – 7.53 (m, 2H), 7.47 (td, \(J = 7.4, 4.0\) Hz, 4H), 4.28 (q, \(J = 7.1\) Hz, 2H), 1.21 (t, \(J = 7.1\) Hz, 3H).

Ethyl (E)-2-benzoyl-4-(3,4-dimethoxyphenyl)-4-oxobut-2-enoate

![Structure 1b]

Yellow solid; yield: 0.309 mg, 84%; mp: 82–83 \(^\circ\)C.

FT-IR (KBr) \(\nu\) (cm\(^{-1}\)) 2923, 1726, 1661, 1590, 1522, 1367, 1279, 1237, 1136, 1016.

\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.15 (s, 1H), 7.91 (d, \(J = 7.6\) Hz, 2H), 7.70 (dd, \(J = 8.4, 1.9\) Hz, 1H), 7.58 (t, \(J = 7.4\) Hz, 1H), 7.49 – 7.45 (m, 3H), 6.92 (d, \(J = 8.4\) Hz, 1H), 4.27 (q, \(J = 7.1\) Hz, 2H), 3.97 (s, 3H), 3.86 (s, 3H), 1.21 (t, \(J = 7.1\) Hz, 3H)

\(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 193.59, 186.36, 163.72, 154.39, 149.39, 144.19, 135.85, 133.38, 133.27, 129.53, 128.60, 128.38, 124.24, 110.31, 109.92, 62.29, 56.09, 55.86, 13.80

HRMS (m/z) [M + H]\(^+\) calcd for C\(_{21}\)H\(_{21}\)O\(_6\): 369.1338; found: 369.1329.

Ethyl (E)-2-benzoyl-4-(4-chlorophenyl)-4-oxobut-2-enoate\(^1\)

![Structure 1c]

FT-IR (KBr) \(\nu\) (cm\(^{-1}\)) 3088, 2980, 1714, 1663, 1584, 1369, 1267.

\(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 8.09 (s, 1H), 7.95 – 7.85 (m, 4H), 7.64 – 7.54 (m, 1H), 7.47 (dd, \(J = 8.2, 6.6\) Hz, 4H), 4.28 (q, \(J = 7.1\) Hz, 2H), 1.21 (t, \(J = 7.1\) Hz, 3H).

Ethyl (E)-2-benzoyl-4-(4-bromophenyl)-4-oxobut-2-enoate\(^1\)

![Structure 1d]
FT-IR (KBr) ν (cm$^{-1}$) 3089, 2980, 1716, 1665, 1581, 1366, 1262.

$^1$H NMR (300 MHz, CDCl$_3$) δ 8.08 (s, 1H), 7.94 – 7.86 (m, 2H), 7.86 – 7.79 (m, 2H), 7.69 – 7.55 (m, 3H), 7.46 (dd, $J = 10.4, 4.6$ Hz, 2H), 4.28 (q, $J = 7.2$ Hz, 2H), 1.21 (t, $J = 7.1$ Hz, 3H).

**Ethyl (E)-2-(4-methylbenzoyl)-4-oxo-4-(p-tolyl)but-2-enoate**

Yellow oil; yield: 0.266 mg, 79%.

FT-IR (KBr) ν (cm$^{-1}$) 2923, 2853, 1722, 1667, 1363, 1267, 1241, 1179, 1013.

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.12 (s, 1H), 7.86 (d, $J = 8.2$ Hz, 2H), 7.80 (d, $J = 8.2$ Hz, 2H), 7.27 – 7.23 (m, 4H), 4.26 (q, $J = 7.1$ Hz, 2H), 2.40 (s, 3H), 2.39 (s, 3H), 1.21 (t, $J = 7.2$ Hz, 3H);

$^{13}$C NMR (101 MHz, CDCl$_3$) δ 193.08, 187.79, 163.81, 145.28, 144.54, 144.37, 133.79, 133.45, 133.32, 129.53, 129.35, 128.98, 128.59, 62.29, 21.72, 21.69, 13.85.

HRMS (m/z) [M + H]$^+$ calcld for C$_{21}$H$_{21}$O$_4$: 337.1440; found: 337.1434.

**Ethyl (E)-2-(4-nitrobenzoyl)-4-oxo-4-phenylbut-2-enoate$^1$**

FT-IR (KBr) ν (cm$^{-1}$) 3086, 2983, 1720, 1603, 1514, 1341, 1085, 1010.

$^1$H NMR (300 MHz, CDCl$_3$) δ 8.38 – 8.28 (m, 2H), 8.22 (s, 1H), 8.11 – 8.03 (m, 2H), 8.01 – 7.93 (m, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.51 (t, $J = 7.7$ Hz, 2H), 4.30 (q, $J = 7.1$ Hz, 2H), 1.24 (t, $J = 7.1$ Hz, 3H).

**Ethyl (E)-2-(4-nitrobenzoyl)-4-oxo-4-(p-tolyl)but-2-enoate$^3$**

FT-IR (KBr) ν (cm$^{-1}$) 3049, 2985, 1720, 1603, 1514, 1341, 1085, 101; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.32 (d, $J = 8.3$ Hz, 2H), 8.21 (s, 1H), 8.07 (d, $J = 8.5$ Hz, 2H), 7.87 (d, $J = 8.2$ Hz, 2H), 7.30 (d, $J = 8.2$ Hz, 2H), 4.29 (q, $J = 7.1$ Hz, 2H), 2.43 (s, 3H), 1.23 (t, $J = 7.1$ Hz, 3H).

**Ethyl (E)-4-(4-methoxyphenyl)-2-(4-nitrobenzoyl)-4-oxobut-2-enoate$^3$**
$\text{FT-IR (KBr) } \nu (\text{cm}^{-1})$ 3049, 2986, 1722, 1685, 1601, 1517, 1342, 1082, 1024.

$^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta$ 8.31 (d, $J = 8.4$ Hz, 2H), 8.20 (s, 1H), 8.07 (d, $J = 8.6$ Hz, 2H), 7.96 (d, $J = 8.7$ Hz, 2H), 6.96 (d, $J = 8.7$ Hz, 2H), 4.29 (q, $J = 7.1$ Hz, 2H), 3.89 (s, 3H), 1.23 (t, $J = 7.1$ Hz, 3H).

**Ethyl (E)-4-(3,4-dimethoxyphenyl)-2-(4-nitrobenzoyl)-4-oxobut-2-enoate**

$\text{FT-IR (KBr) } \nu (\text{cm}^{-1})$ 3080, 2978, 1728, 1685, 1592, 1525, 1345, 1083, 1017.

$^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta$ 8.33 (d, $J = 8.8$ Hz, 2H), 8.21 (s, 1H), 8.08 (d, $J = 8.9$ Hz, 2H), 7.71 (dd, $J = 8.4$, 2.0 Hz, 1H), 7.42 (d, $J = 1.9$ Hz, 1H), 6.94 (d, $J = 8.4$ Hz, 1H), 4.29 (q, $J = 7.1$ Hz, 2H), 3.98 (s, 1H), 3.86 (s, 3H), 1.23 (t, $J = 7.2$ Hz, 2H).

**Ethyl (E)-2-(4-nitrobenzoyl)-4-(4-nitrophenyl)-4-oxobut-2-enoate**

$\text{FT-IR (KBr) } \nu (\text{cm}^{-1})$ 3109, 2982, 1716, 1675, 1603, 1520, 1345, 1266, 1007.

$^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta$ 8.39 – 8.32 (m, 4H), 8.17 (s, 1H), 8.14 (d, $J = 8.8$ Hz, 2H), 8.07 (d, $J = 8.8$ Hz, 2H), 4.32 (q, $J = 7.2$ Hz, 2H), 1.25 (t, $J = 7.1$ Hz, 2H).

**2-Benzoyl-1,4-diphenylbut-2-ene-1,4-dione**

$\text{FT-IR (KBr) } \nu (\text{cm}^{-1})$ 3063, 1665, 1645, 1594, 1446.
$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.10 – 8.03 (m, 2H), 8.04 – 7.97 (m, 2H), 7.94 – 7.85 (m, 2H), 7.70 – 7.40 (m, 10H).

**2-Benzoyl-1-phenyl-4-(p-tolyl)but-2-ene-1,4-dione**

![Diagram](image)

FT-IR (KBr) $\nu$ (cm$^{-1}$) 3060, 2921, 1665, 1597, 1448, 1377, 1267, 1231, 1020.

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.10 – 8.03 (m, 2H), 8.02 – 7.96 (m, 2H), 7.79 (d, $J = 8.2$ Hz, 2H), 7.65 (t, $J = 7.4$ Hz, 1H), 7.60 – 7.49 (m, 4H), 7.44 (t, $J = 7.4$ Hz, 2H), 7.23 (s, 1H), 2.40 (s, 3H).

**2-Benzoyl-4-(4-bromophenyl)-1-phenylbut-2-ene-1,4-dione**

![Diagram](image)

FT-IR (KBr) $\nu$ (cm$^{-1}$) 3086, 3058, 1644, 1581, 1446, 1403, 1372, 1261, 1175.

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.05 (dd, $J = 5.2$, 3.3 Hz, 2H), 7.98 (dd, $J = 5.3$, 3.3 Hz, 2H), 7.79 – 7.72 (m, 2H), 7.69 – 7.41 (m, 9H).

**(E)-1,4-di-p-tolybut-2-ene-1,4-dione**

![Diagram](image)

FT-IR (KBr) $\nu$ (cm$^{-1}$) 2922, 1647, 1601, 1324, 1299, 1182, 1034.

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 8.01 (s, 2H), 7.98 (d, $J = 8.3$ Hz, 4H), 7.33 (d, $J = 8.0$ Hz, 4H), 2.45 (s, 6H).

References:
Figure S1. $^1$H NMR spectrum of compound 1a (300 MHz, CDCl$_3$)

Figure S2. $^1$H NMR spectrum of compound 1b (300 MHz, CDCl$_3$)
Figure S3. $^{13}$C NMR spectrum of compound 1b (75 MHz, CDCl$_3$)

Figure S4. $^1$H NMR spectrum of compound 1c (300 MHz, CDCl$_3$)
Figure S5. $^1$H NMR spectrum of compound 1d (300 MHz, CDCl$_3$)

Figure S6. $^1$H NMR spectrum of compound 1e (400 MHz, CDCl$_3$)
Figure S7. $^{13}$C NMR spectrum of compound 1e (101 MHz, CDCl$_3$)

Figure S8. $^1$H NMR spectrum of compound 1f (300 MHz, CDCl$_3$)
Figure S9. $^1$H NMR spectrum of compound 1g (400 MHz, CDCl$_3$)

Figure S10. $^1$H NMR spectrum of compound 1h (400 MHz, CDCl$_3$)
Figure S11. $^1$H NMR spectrum of compound 1i (400 MHz, CDCl$_3$)

Figure S12. $^1$H NMR spectrum of compound 1j (400 MHz, CDCl$_3$)
Figure S13. $^1$H NMR spectrum of compound 1jk (300 MHz, CDCl$_3$)
Figure S14. $^1$H NMR spectrum of compound 1l (300 MHz, CDCl$_3$)

Figure S15. $^1$H NMR spectrum of compound 1m (300 MHz, CDCl$_3$)
Figure S16. $^1$H NMR spectrum of compound 1n (300 MHz, CDCl$_3$)

Figure S17. $^1$H NMR spectrum of compound 3aa (400 MHz, CDCl$_3$)

Figure S18. $^{13}$C NMR spectrum of compound 3aa (101 MHz, CDCl$_3$)
Figure S19. $^1$H NMR spectrum of compound 3ab (400 MHz, CDCl$_3$)

Figure S20. $^{13}$C NMR spectrum of compound 3ab (101 MHz, CDCl$_3$)
Figure S21. $^1$H NMR spectrum of compound 3ac (300 MHz, CDCl$_3$)

Figure S22. $^{13}$C NMR spectrum of compound 3ac (75 MHz, CDCl$_3$)
Figure S23. $^1$H NMR spectrum of compound 3ad (400 MHz, CDCl$_3$)

Figure S24. $^{13}$C NMR spectrum of compound 3ad (101 MHz, CDCl$_3$)
Figure S25. $^1$H NMR spectrum of compound 3ae (400 MHz, CDCl$_3$)

Figure S26. $^{13}$C NMR spectrum of compound 3ae (101 MHz, CDCl$_3$)
Figure S27. $^1$H NMR spectrum of compound 3af (400 MHz, CDCl$_3$)

Figure S28. $^{13}$C NMR spectrum of compound 3af (101 MHz, CDCl$_3$)
Figure S29. $^1$H NMR spectrum of compound 3ag (300 MHz, CDCl$_3$)

Figure S30. $^{13}$C NMR spectrum of compound 3ag (75 MHz, CDCl$_3$)
Figure S31. $^1$H NMR spectrum of compound 3ah (300 MHz, CDCl$_3$)

Figure S32. $^{13}$C NMR spectrum of compound 3ah (75 MHz, CDCl$_3$)
Figure S33. $^1$H NMR spectrum of compound 3ai (400 MHz, CDCl$_3$)

Figure S34. $^{13}$C NMR spectrum of compound 3ai (101 MHz, CDCl$_3$)
Figure S35. $^1$H NMR spectrum of compound 3aj (400 MHz, CDCl$_3$)

Figure S36. $^{13}$C NMR spectrum of compound 3aj (75 MHz, CDCl$_3$/DMSO-$d_6$ = 3:1)
Figure S37. $^1$H NMR spectrum of compound 3ak (300 MHz, CDCl$_3$)

Figure S38. $^{13}$C NMR spectrum of compound 3ak (75 MHz, CDCl$_3$)
**Figure S39.** $^1$H NMR spectrum of compound 3al (400 MHz, CDCl$_3$)

**Figure S40.** $^{13}$C NMR spectrum of compound 3al (75 MHz, CDCl$_3$)
Figure S41. $^1$H NMR spectrum of compound 3am (400 MHz, CDCl$_3$)

Figure S42. $^{13}$C NMR spectrum of compound 3am (101 MHz, DMSO-d$_6$)
Figure S43. $^1$H NMR spectrum of compound 3an (400 MHz, CDCl$_3$)

Figure S44. $^{13}$C NMR spectrum of compound 3an (101 MHz, CDCl$_3$)
Figure S45. $^1$H NMR spectrum of compound 3ao (400 MHz, CDCl$_3$)

Figure S46. $^{13}$C NMR spectrum of compound 3ao (75 MHz, CDCl$_3$)
Figure S47. $^1$H NMR spectrum of compound 3ap (400 MHz, CDCl₃)

Figure S48. $^{13}$C NMR spectrum of compound 3ap (101 MHz, CDCl₃)
Figure S49. $^1$H NMR spectrum of compound **3aq** (300 MHz, CDCl$_3$)

Figure S50. $^{13}$C NMR spectrum of compound **3aq** (75 MHz, CDCl$_3$)
Figure S51. $^1$H NMR spectrum of compound 3ar (300 MHz, CDCl$_3$)

Figure S52. $^{13}$C NMR spectrum of compound 3ar (75 MHz, CDCl$_3$/DMSO-d$_6$ = 3:1)
Figure S53. $^1$H NMR spectrum of compound 3as (300 MHz, CDCl$_3$)

Figure S54. $^{13}$C NMR spectrum of compound 3as (75 MHz, CDCl$_3$)
Figure S55. $^1$H NMR spectrum of compound 3at (400 MHz, CDCl$_3$)

Figure S56. $^{13}$C NMR spectrum of compound 3at (75 MHz, CDCl$_3$)
Figure S57. $^1$H NMR spectrum of compound 3au (300 MHz, CDCl$_3$/DMSO-d$_6$ = 3:1)

Figure S58. $^{13}$C NMR spectrum of compound 3au (75 MHz, CDCl$_3$/DMSO-d$_6$ = 3:1)
Figure S59. $^1$H NMR spectrum of compound 3ba (400 MHz, CDCl$_3$)

Figure S60. $^{13}$C NMR spectrum of compound 3ba (101 MHz, CDCl$_3$)
Figure S61. $^{1}H$ NMR spectrum of compound 3bb (400 MHz, CDCl$_3$)

Figure S62. $^{13}C$ NMR spectrum of compound 3bb (101 MHz, CDCl$_3$)
Figure S63. $^1$H NMR spectrum of compound 3bc (300 MHz, CDCl$_3$)

Figure S64. $^{13}$C NMR spectrum of compound 3bc (75 MHz, CDCl$_3$)
Figure S65. $^1$H NMR spectrum of compound 3ca (400 MHz, CDCl$_3$)

Figure S66. $^{13}$C NMR spectrum of compound 3ca (75 MHz, CDCl$_3$)
Figure S67. $^1$H NMR spectrum of compound 3cb (400 MHz, CDCl$_3$)

Figure S68. $^{13}$C NMR spectrum of compound 3cb (75 MHz, CDCl$_3$)
Figure S69. $^1$H NMR spectrum of compound 3cc (400 MHz, CDCl$_3$)

Figure S70. $^{13}$C NMR spectrum of compound 3cc (101 MHz, CDCl$_3$)
Figure S71. $^1$H NMR spectrum of compound 3cd (300 MHz, CDCl$_3$)

Figure S72. $^{13}$C NMR spectrum of compound 3cd (75 MHz, CDCl$_3$)
Figure S73. $^1$H NMR spectrum of compound 3da (300 MHz, CDCl$_3$)

Figure S74. $^{13}$C NMR spectrum of compound 3da (75 MHz, CDCl$_3$)
Figure S75. $^1$H NMR spectrum of compound 4 (300 MHz, CDCl$_3$)

Figure S76. $^{13}$C NMR spectrum of compound 4 (75 MHz CDCl$_3$)
Figure S77. $^1$H NMR spectrum of compound 5 (300 MHz, CDCl$_3$)

Figure S78. $^{13}$C NMR spectrum of compound 5 (75 MHz, CDCl$_3$)
Figure S79. $^1$H NMR spectrum of compound 6 (400 MHz, CDCl$_3$)

Figure S80. $^{13}$C NMR spectrum of compound 6 (101 MHz, CDCl$_3$)
Crystal Growing
The crystal was obtained by the slow evaporation technique. The compound 3ap (50 mg) was dissolve in 1 mL of DCM and the solution was kept in a sample vial. The vial was closed with cap and allowed to evaporate slowly.

Single crystal XRD data for compound 3ap.

Figure S65. X-ray crystal structure of compound 3ap.

Table 1. Sample and crystal data for compound 3ap

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<td></td>
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Table 2. Data collection and structure refinement for compound 3b.
Theta range for data collection 0.98 to 24.67°
Index ranges  -13<=h<=13, -11<=k<=11, -45<=l<=49
Reflections collected 31890
Independent reflections 8116 [R(int) = 0.0502]
Coverage of independent reflections 99.3%
Absorption correction multi-scan
Max. and min. transmission 0.8380 and 0.6580
Refinement method Full-matrix least-squares on F^2
Refinement program SHELXL-2014/7 (Sheldrick, 2014)
Function minimized \( \Sigma w(F_o^2 - F_c^2)^2 \)
Data / restraints / parameters 8116 / 0 / 597
Goodness-of-fit on F^2 1.014
\( \Delta/\sigma_{\text{max}} \) 0.002
Final R indices 4902 data; I>2\( \sigma(I) \) R1 = 0.0509, wR2 = 0.0924
all data R1 = 0.1068, wR2 = 0.1107
Weighting scheme w=1/[\( \sigma^2(F_o^2) + (0.0316P)^2 + 5.0089P \)]
where P=(\( F_o^2 + 2F_c^2 \))/3
Largest diff. peak and hole 0.411 and -0.567 eÅ^3
R.M.S. deviation from mean 0.055 eÅ^3