Supplementary Information

Construction of Benzoheterocycles by the Reaction of α-Arylglyoxylic Acids And *Ortho*-Functionalized Aniline under Mild and Minimal Conditions

Hang Gong^{a,b*}, Fangyuan Zhou^b and Changqun Cai^b

- ^a College of Chemistry and Chemical Engineering, Yunnan Normal University, Kunming, 650500 (P. R. China)
- ^b The key Laboratory for Green Organic Synthesis and Application of Hunan Province, Key Laboratory of Environmentally Friendly Chemistry and Application of Ministry of Education, College of Chemistry, Xiangtan University, Xiangtan 411105 (P. R. China)

* E-mail: hgong@xtu.edu.cn

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1. General information

Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254). IR spectra were recorded on a new Fourier transform infrared spectroscopy. ¹H and ¹³C NMR spectra were recorded on 400, 100 MHz NMR spectrometer using CDCl₃ as solvent unless otherwise stated. HRMS were made by means of ESI with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. Unless otherwise noted, all reagents were weighed and handled in air, and all reactions were carried out in a sealed tube under an atmosphere of argon. Oil bath was used for reactions that require heating. Unless otherwise noted, reagents were purchased from commercial suppliers and used without further purification.

2. Characterization data for products

3-Phenyl-benzo[1,4]oxazin-2-one (3a)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.4$; White solid; Yield 91%; M.p. = 108-109 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.33 (dd, J = 8.1, 1.5Hz, 2H), 7.83 (dd, J = 7.9, 1.4 Hz, 1H), 7.57–7.44 (m, 4H), 7.37 (td, J = 7.8, 1.2 Hz, 1H), 7.31 (dd, J = 8.2, 1.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ =152.3, 150.9, 146.5, 134.1, 131.7, 131.4, 131.1, 129.4, 128.4, 125.5, 116.2.

3-p-Tolyl-benzo[1,4]oxazin-2-one (3b)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:20, v:v), $R_f = 0.7$; Brown solid; Yield 87%; M.p. = 108-109 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.27-8.25$ (d, J = 8.4 Hz, 2H), $\delta = 7.85-7.82$ (dd, J = 1.6, 8.0 Hz, 1H), $\delta = 7.52-7.47$ (dt, J = 1.6, 8.0 Hz, 1H), $\delta = 7.40-7.36$ (dt, J = 0.8, 8.0 Hz, 1H), $\delta = 7.33-7.30$ (m, 3H), 2.44 (s, 3H); 13C NMR (100 MHz, CDCl₃): $\delta = 152.3$, 150.6, 146.4, 142.0, 131.7, 131.4, 130.8, 129.4, 129.3, 129.1, 125.5, 116.1, 21.6.

3-p-Tolyl-benzo[1,4]oxazin-2-one (3c)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.4$; Beige solid; Yield 97%; M.p. = 162-163 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.41-8.39$ (d, J = 8.8 Hz, 2H), $\delta = 7.82-7.80$ (d, J = 8.0 Hz, 1H), $\delta = 7.49-7.45$ (t, J = 8.0 Hz, 1H), $\delta = 7.39-7.35$ (t, J = 8.0 Hz, 1H), $\delta = 7.32-7.30$ (d, J = 8.4 Hz, 1H), 7.01-6.99 (d, J = 8.8 Hz, 2H), 3.89 (s, 3H); ¹³C NMR (100 MHz, 200 MHz,

CDCl₃): δ = 162.4,152.5, 149.9, 146.3, 131.8, 131.4, 130.4, 129.1, 126.8, 125.4, 116.1, 113.8, 55.4.

3-(Benzo[1,3]dioxol-5-yl)-benzo[1,4]oxazin-2-one (3d)



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.6$; Yellow solid; Yield 52%; M.p. = 161-163 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.09 (dd, J = 2.0, 8.0 Hz, 1H), 7.89 (s, 1H), 7.80 (d, J = 7.6 Hz, 1H), 7.50-7.46 (t, J = 8.0 Hz, 1H), 7.39-7.35 (t, J = 7.6 Hz, 1H), 7.32-7.30 (d, J = 8.4 Hz, 1H), 6.92-6.90 (d, J = 8.0 Hz, 1H), 6.05 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 152.3, 150.6, 149.4, 147.9, 146.3, 131.6, 130.6, 129.1, 128.3, 125.5, 125.3, 116.0, 109.3, 108.1, 101.6; IR (KBr) 2359, 2341, 1738, 1601, 1504, 1482, 1465, 1441, 1276, 1250, 1223, 1085, 1036, 761 cm⁻¹.HRMS (ESI): m/z [M+Na]⁺ calcd for C₁₅H₉NaNO₄ 290.0424, found 290.0436.$

3-(4-Fluoro-phenyl)-benzo[1,4]oxazin-2-one (3e)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.7$; Beige solid; Yield 86%; M.p. > 180 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.43-8.39$ (m, 2H), $\delta = 7.85-7.83$ (d, J = 8.0 Hz, 1H), $\delta = 7.54-7.50$ (dt, J = 1.6, 8.0 Hz, 1H), $\delta = 7.42-7.38$ (t, J = 7.2 Hz, 1H), $\delta = 7.35-7.33$ (dd, J = 1.2, 8.0 Hz, 1H), 7.20-7.16 (t, J = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 164.8$ (d, J = 251.8 Hz), 152.3, 149.5, 146.4, 131.9, 131.8, 131.5, 131.2, 130.3, 129.4, 125.6, 116.2, 115.6, 115.4.

3-(4-Chloro-phenyl)-benzo[1,4]oxazin-2-one (3f)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:4, v:v), Rf = 0.3; Beige solid; Yield 94%; M.p. = 165-166 °C; ¹H NMR (400 MHz, CDCl₃): δ =8.35-8.33 (d, *J* = 8.4 Hz, 2H), δ = 7.85-7.83 (dd, *J* = 1.2, 8.0 Hz, 1H), δ = 7.55-7.51 (t, *J* = 7.2 Hz, 1H), δ = 7.47-7.45 (d, *J* = 8.8 Hz, 2H), δ = 7.42-7.38 (t, *J* = 7.6 Hz, 1H), 7.35-7.33 (d, J = 8.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 152.1, 149.5, 146.5, 137.8, 132.5, 131.5, 131.4, 130.8, 129.5, 128.7, 125.7, 116.2.

3-(4-Bromo-phenyl)-benzo[1,4]oxazin-2-one (3g)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:4, v:v), $R_f = 0.3$; Beige solid; Yield 77%; M.p. = 152-153 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.27-8.25$ (d, J = 8.4 Hz, 2H), $\delta = 7.85-7.83$ (dd, J = 0.8, 8.0 Hz, 1H), $\delta = 7.63-7.61$ (d, J = 8.8 Hz, 2H), $\delta = 7.55-7.51$ (dt, J = 0.8, 8.4 Hz, 1H), $\delta = 7.42-7.38$ (t, J = 7.6 Hz, 1H), 7.34-7.32 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 152.1$, 149.6, 146.4, 132.9, 131.7,131.6, 131.5, 131.0, 129.5, 126.5, 125.7, 116.2.

3-Naphthalen-2-yl-benzo[1,4]oxazin-2-one (3h)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.7$; Yellow solid; Yield 62%; M.p. > 180 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 9.07$ (s, 1H), $\delta = 8.42-8.39$ (dd, J = 2.0, 8.8 Hz, 1H), $\delta = 8.02-8.00$ (d, J = 8.0 Hz, 1H), $\delta = 7.95-7.87$ (m, 3H), $\delta = 7.60-7.51$ (m, 3H), $\delta = 7.43-7.40$ (t, J = 7.6 Hz, 1H), $\delta = 7.37-7.35$ (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 152.4$, 150.2, 146.4,134.7, 132.8, 131.7, 131.4, 131.1, 129.6, 129.4, 128.0, 127.9, 127.6, 126.5, 125.6,125.3, 116.1..

3-Thiophen-2-yl-benzo[1,4]oxazin-2-one (3i)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.5$; Beige solid; Yield 90%; M.p. > 180 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.42 (dd, J = 1.2, 4.0 Hz, 1H), 7.78 (dd, J = 1.6, 8.0 Hz, 1H), 7.62-7.60 (dd, J = 1.2, 5.2 Hz, 1H), 7.49-7.45 (dt, J = 1.6, 7.6 Hz, 1H), 7.38 - 7.35 (m, 1H), 7.32 - 7.30 (dd, J = 1.2, 8.0 Hz 1H), 7.22 - 7.15 (t, J = 4.4 Hz 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 151.5, 146.0, 145.6, 138.5, 133.3, 132.5, 131.5, 130.5, 128.9, 128.6, 125.7, 116.2.

3-(Indol-3-yl)-benzo[1,4]oxazin-2-one (3j)^[2]



Purified by TLC (ethyl cetate/petroleum ether = 1:4, v:v), $R_f = 0.4$; Yellow solid; Yield 61%; M.p. > 180 °C; ¹H NMR (400 MHz, DMSO-d₆): $\delta = 11.98$ (s, 1H), $\delta = 8.77-8.75$ (m, 1H), $\delta = 8.70-8.69$ (d, J = 2.8 Hz 1H), $\delta = 7.87-7.85$ (d, J = 7.6 Hz 1H), $\delta = 7.55-7.53$ (m, 1H), $\delta = 7.49-7.45$ (m, 1H), $\delta = 7.44-7.39$ (m, 2H), $\delta = 7.30-7.24$ (m, 2H); ¹³C NMR (100 MHz, DMSO-d₆): $\delta = 152.5$, 148.4, 145.3 137.1, 134.2, 132.4, 129.1, 128.2, 126.4, 125.8, 123.5, 123.3, 122.0, 116.4, 112.7, 111.10.

3-Benzyl-benzo[1,4]oxazin-2-one (3k)^[3]



Purified by TLC (ethyl cetate/petroleum ether = 1:10, v:v), Rf = 0.5; White solid; Yield 40%; M.p. = 125-127 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 7.74-7.72$ (dd, J = 1.6, 8.0 Hz, 1H), $\delta = 7.46-7.42$ (m, 3H), $\delta = 7.34-7.29$ (m, 3H), $\delta = 7.26-7.22$ (m, 2H), $\delta = 4.19$ (s, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = .156.3, 152.8, 146.6, 135.5, 131.3, 130.8, 129.6, 129.1, 128.7, 127.1, 125.5, 116.4, 40.6.$

5-hydroxy-3-phenyl-benzo[1,4]oxazin-2-one (3l)^[3]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.5$; Yellow solid; Yield 78%; M.p. > 180 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.30-8.26$ (m, 2H), $\delta = 7.56-7.47$ (m, 3H), $\delta = 7.43-7.39$ (t, *J* =8.4 Hz), 7.30 (1H, s), $\delta = 6.94-6.92$ (dd, *J* = 1.2, 8.2 Hz 1H), $\delta = 6.85-6.83$ (dd, *J* = 1.2, 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 153.3$, 152.8, 148.0, 147.0, 133.7, 132.7, 131.6, 129.4, 128.5, 120.5, 110.6, 107.1.

7-Methyl-3-phenyl-benzo[1,4]oxazin-2-one (3m)^[1]



Purified by TLC (ethyl cetate/petroleum ether = 1:4, v:v), $R_f = 0.6$; Yellow solid; Yield 95%; M.p. = 150-151 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.32-8.30$ (m, 2H), 7.73-7.71 (d, J = 8.4 Hz, 1H) $\delta = 7.52-7.47$ (m, 3H), $\delta = 7.21-7.18$ (dd, J = 2.0, 8.0 Hz, 1H), $\delta = 7.13$ (s, 1H), $\delta = 2.48$ (s, 3H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 152.5$, 149.6, 146.4, 142.6, 134.3, 131.2, 129.8, 129.3, 129.0, 128.3, 126.7, 116.2, 21.8.

6-Fluoro-3-phenyl-benzo[1,4]oxazin-2-one (3n)^[2]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.6$; Yellow solid; Yield 98%; M.p. = 142-143 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.39 - 8.28$ (m, 2H), $\delta = 7.61 - 7.44$ (m, 4H), $\delta = 7.32-7.29$ (m, 1H), $\delta = 7.28 - 7.20$ (m, 1H).; ¹³C NMR (100 MHz, CDCl₃): δ 160.6, 151.9, 151.8, 142.8, 133.7, 132.1, 131.8, 129.6, 128.4, 118.5, 118.3, 117.2, 117.1, 115.1, 114.9.

7-Chloro-3-phenyl-benzo[1,4]oxazin-2-one (3o)^[2]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.5$; Brown solid; Yield 86%; M.p. = 104-106 °C, ¹H NMR (400 MHz, CDCl₃): $\delta = 8.34-8.31$ (m, 2H), 7.79-7.72 (d, J = 8.4 Hz, 1H), 7.57–7.48 (m, 3H), 7.38-7.35 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 151.5$, 150.6, 146.8, 136.85, 133.8, 131.7, 130.3, 130.2, 129.5, 128.5, 126.2, 116.5.

3-Phenyl-naphtho[1,4]oxazin-2-one (**3p**)^[3]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.5$; White solid; Yield 63%; M.p. >180 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.00$ (d, J = 8.0 Hz, 1H), $\delta = 7.90$ (d, J = 8.0 Hz, 1H), $\delta = 7.70-7.69$ (m, 1H), $\delta = 7.62-7.50$ (m,5H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 152.2$, 151.1, 144.2, 134.2, 133.9, 131.5, 130.9, 130.8, 129.5, 129.2, 128.9, 128.5, 128.4, 127.4, 126.0, 112.3.

3-Phenylquinoxalin-2(1H)-one (3q)^[4]



Purified by TLC (ethyl cetate/petroleum ether = 1:1, v:v), $R_f = 0.4$; Yellow solid; Yield 80%; M.p. > 180 °C; ¹H NMR (400 MHz, DMSO-d₆): $\delta = 12.56$ (s, 1H), $\delta = 8.27-8.25$ (m, 2H), $\delta = 7.82-7.80$ (d, J = 8.0 Hz, 1H), $\delta = 7.54-7.44$ (m, 4 H), $\delta = 7.33-7.28$ (m, 2 H); ¹³C NMR (100 MHz, DMSO-d₆): $\delta = 155.1$, 154.6, 136.1, 132.5, 130.2, 130.7, 129.7, 129.2, 128.4, 123.9, 115.6.

3-(4-methoxyphenyl)quinoxalin-2(1H)-one (3r)^[4]



Purified by TLC (ethyl cetate/petroleum ether = 1:4, v:v), $R_f = 0.3$; White solid; Yield 92%; M.p. > 180 °C; ¹H NMR (400 MHz, DMSO-d₆): $\delta = 12.48$ (s, 1 H), 8.37-8.35 (d, J = 8.8 Hz, 2 H), $\delta = 7.76$ -7.78 (d, J = 8.0 Hz, 1 H), $\delta = 7.49$ -7.45 (t, J = 7.6 Hz, 1H), $\delta = 7.30$ -7.25 (m, 2 H), $\delta = 7.02$ -7.00 (d, J = 8.8 Hz, 2 H), $\delta = 3.85$ (s, 3 H); ¹³C NMR (100 MHz, DMSO-d₆): $\delta = 161.5$, 155.2, 153.6, 132.5, 132.2, 131.5, 130.2, 128.9, 128.6, 123.8, 115.5, 113.8, 55.8.

3-(4-Chlorophenyl)quinoxalin-2(1*H*)-one (3s)^[4]



Purified by TLC (ethyl cetate/petroleum ether = 1:1, v:v), $R_f = 0.4$; Yellow solid; Yield 91%; M.p. > 180 °C; ¹H NMR (400 MHz, DMSO-d₆): $\delta = 12.62$ (s, 1H), $\delta = 8.34-8.32$ (d, J = 8.8 Hz, 2 H), $\delta = 7.81-7.79$ (d, J = 8.0 Hz, 2 H), $\delta = 7.54-7.50$ (m, 3 H), $\delta = 7.31-7.27$ (m, 2H); ¹³C NMR (100 MHz, DMSO-d₆): $\delta = 13$ C NMR (100 MHz, DMSO-d₆) $\delta = 155.0$, 153.2, 135.6, 134.8, 132.5, 132.4, 131.4, 131.0, 129.3, 128.4, 124.0, 115.6.

2-Phenylbenzo[d]thiazole (4t)^[5]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.6$; White solid; Yield 96%; M.p. = 122-123 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.12-8.08$ (m, 3H), $\delta = 7.91-7.89$ (d, J = 7.6 Hz, 1H), $\delta = 7.52-7.48$ (m, 4H), $\delta = 7.41-7.37$ (m, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 166.7$, 154.1, 137.1, 135.1, 132.1, 129.3, 128.7, 126.5, 125.5, 123.3, 121.7.

2-(4-chlorophenyl)benzothiazole (4u)^[5]

Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.5$; White solid; Yield 95%; M.p. = 115-116 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.08-8.01$ (m, 3H), 7.92-7.90 (d, J = 8.0 Hz, 1H) $\delta = 7.53-7.45$ (m, 3H), $\delta = 7.42-7.38$ (m, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 166.7$, 154.1, 137.1, 135.1, 132.1, 129.3, 128.7, 126.5, 125.5, 123.3, 121.7.

2-(4-Methoxyphenyl)benzo[d]thiazole (4v)^[5]



Purified by TLC (ethyl cetate/petroleum ether = 1:15 v:v), Rf = 0.6; Beige solid; Yield 90%; M.p. = 132-133 °C; ¹H NMR (400 MHz, CDCl₃): δ = 8.06-8.02 (m, 3H), δ = 7.88-7.86 (d, *J* = 8.0 Hz, 1H), δ = 7.49-7.45(m, 1H), δ = 7.38-7.34 (m, 1H), δ = 7.01-6.99 (d, *J* = 8.8 Hz, 2H), δ = 3.88 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 167.9, 161.9, 154.2, 134.9, 129.1, 126.4, 126.2, 124.8, 122.8, 121.5, 114.4, 55.5.

5-Chloro-2-phenylbenzo[d]thiazole (4w)^[5]



Purified by TLC (ethyl cetate/petroleum ether = 1:15, v:v), $R_f = 0.6$; White solid; Yield 70%; M.p. = 148-149 °C; ¹H NMR (400 MHz, CDCl₃): $\delta = 8.09-8.05$ (m, 3H), $\delta = 7.82-7.80$ (d, J = 8.4 Hz, 1H), 7.51-7.50 (m,3H), 7.37 (dd, J = 2.0, 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 13$ C NMR (101 MHz, Chloroform-d) $\delta = 170.0, 155.0, 133.3, 133.2, 132.3, 131.4, 129.1, 127.6, 125.7, 123.1, 122.4.$

3. References

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4. Copies of ¹H and ¹³C NMR spectra

¹H NMR spectrum of 3a (400 MHz, CDCl₃)



¹³C NMR spectrum of 3a (100 MHz, CDCl₃)



^{160 150 140 130 120} 80 70 60 30 20 10 0 -10 210 200 190 180 170 110 100 f1 (ppm) 50 40 90



¹³C NMR spectrum of 3b (100 MHz, CDCl₃)





¹³C NMR spectrum of 3c (100 MHz, CDCl₃)



¹H NMR spectrum of 3d (400 MHz, CDCl₃)



¹³C NMR spectrum of 3d (100 MHz, CDCl₃)









¹³C NMR spectrum of 3e (100 MHz, CDCl₃)



¹H NMR spectrum of 3f (400 MHz, CDCl₃)





¹³C NMR spectrum of 3f (100 MHz, CDCl₃)



¹H NMR spectrum of 3g (400 MHz, CDCl₃)





¹³C NMR spectrum of 3g (100 MHz, CDCl₃)



¹H NMR spectrum of 3h (400 MHz, CDCl₃)











¹³C NMR spectrum of 3i (100 MHz, CDCl₃)



¹H NMR spectrum of 3j (400 MHz, CDCl₃)



¹³C NMR spectrum of 3j (100 MHz, CDCl₃)





¹³C NMR spectrum of 3k (100 MHz, CDCl₃)









¹³C NMR spectrum of 3l (100 MHz, CDCl₃)





¹³C NMR spectrum of 3m (100 MHz, CDCl₃)



¹H NMR spectrum of 3n (400 MHz, CDCl₃)



¹³C NMR spectrum of 3n (100 MHz, CDCl₃)







¹³C NMR spectrum of 30 (100 MHz, CDCl₃)













¹H NMR spectrum of 3q (400 MHz, CDCl₃)



¹³C NMR spectrum of 3q (100 MHz, CDCl₃)



¹H NMR spectrum of 3r (400 MHz, CDCl₃)



¹³C NMR spectrum of 3r (100 MHz, CDCl₃)



¹H NMR spectrum of 4s (400 MHz, CDCl₃)



¹³C NMR spectrum of 4s (100 MHz, CDCl₃)







¹³C NMR spectrum of 4t (100 MHz, CDCl₃)





¹³C NMR spectrum of 4u (100 MHz, CDCl₃)





¹³C NMR spectrum of 4v (100 MHz, CDCl₃)



¹H NMR spectrum of 4w (400 MHz, CDCl₃)





¹³C NMR spectrum of 4w (100 MHz, CDCl₃)

