Efficient, mild and one-pot quantitative procedures for the synthesis of functionalized benzo[*a*]chromeno phenazine fused ring systems using H₂SO₄, phosphotungstic acid and [NMP]H₂PO₄.

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Experimental

All the chemicals were commercial and were purchased from Sigma-Aldrich or Merck and were used as received. Structures of all the compounds were identified by their spectral data. Precoated aluminium plates with Silica gel 60 F₂₅₄ from Merck were used to monitor reaction progress. Melting points were determined on Buchi melting point 545 apparatus and are uncorrected. The ¹H and ¹³C NMR spectra were recorded on Jeol JNM ECX-400P at 400 MHz and 100 MHz, respectively. The chemical shift values are recorded on δ scale and the coupling constants (J) are in Hertz.

Method A: General procedure for the synthesis of benzo[a]phenazine derivatives (5a-5n) using H₂SO₄ as catalyst.

A mixture of 2-hydroxynaphthalene-1,4-dione (**1**) (1.0 mmol) and 1,2-phenylenediamine (**2**) (1.0 mmol) was placed in a 50 mL round bottomed flask along with EtOH:H₂O (1:1, v/v) (10 mL) as solvent. The flask was attached to a reflux condenser and heated in an oil-bath maintained at 80°C. H₂SO₄ (20 mol%) was added dropwise to the reaction mixture. The contents were stirred magnetically in an oil-bath until an orange solid was formed. Aldehyde (**3**) (1.2 mmol) and cyclohexane-1,3-dione/dimedone/5-methyl-cyclohexyl-1,3-dione (**4**) (1.2 mmol) were then added to the above reaction mixture which was heated further for an appropriate time as shown in Table 1. The progress of the reaction was monitored by TLC (Ethyl acetate: Petroleum ether, 30:70, v/v). After completion of the reaction, the reaction mixture was allowed to cool at room temperature and it was quenched with water (~5 mL). The solid formed was collected by suction pump and washed with acetone to give the pure yellow solid. The products were characterized by IR, ¹H NMR, ¹³C NMR and Mass spectra.

Method B: General procedure for the synthesis of benzo[a]phenazine derivatives (5a-5n) using Phosphotungstic acid as catalyst.

A mixture of 2-hydroxynaphthalene-1,4-dione (**1**) (1.0 mmol), 1,2-phenylenediamine (**2**) (1.0 mmol) and EtOH: H₂O (1:1, v/v) (10 mL) was placed in 50 mL round bottomed flask. The flask was connected to a reflux condenser and heated in oil-bath maintained 80°C. Phosphotungstic acid (20 mol%) was added to the reaction mixture and the contents were stirred magnetically in an oil-bath until an orange solid was formed. Aldehyde (**3**) (1.2 mmol) and cyclohexane-1,3-dione/dimedone/5-methyl-cyclohexyl-1,3-dione (**4**) (1.2 mmol) were added to the above reaction mixture which was heated further for an appropriate time as given in Table 1. The progress of the reaction was monitored by TLC (Ethyl acetate: Petroleum ether, 30:70, v/v). After completion of the reaction, the contents were allowed to cool at room temperature and quenched with water (~5 mL). The solid formed was collected by suction pump and washed with acetone to give the pure yellow solid. The products were characterized by IR, ¹H NMR, ¹³C NMR and Mass spectra.

Method C: General procedure for the synthesis of benzo[a]phenazine derivatives (5a-5n) using [NMP]H₂PO₄ as catalyst and medium.

A mixture of 2-hydroxynaphthalene-1,4-dione (**1**) (1.0 mmol), 1,2-phenylenediamine (**2**) (1.0 mmol) and [NMP]H₂PO₄ (20 mol%) was placed in a 50 mL round bottomed flask. The contents were stirred magnetically in an oil-bath maintained at 80°C until an orange solid was formed. Aldehyde (**3**) (1.2 mmol) and cyclohexane-1,3-dione/dimedone/5-methyl-cyclohexyl-1,3-dione (**4**) (1.2 mmol) were then added to the above reaction mixture which was heated further for an appropriate time as shown in Table 1. The progress of the reaction was monitored by TLC (Ethyl acetate: Petroleum ether, 30:70, v/v). After completion of the reaction, the reaction mixture was allowed to cool at room temperature and was quenched with water (~5 mL). The precipitate

formed was collected by suction pump and washed with acetone to give the pure yellow solid.

The products were characterized by IR, ^1H NMR, ^{13}C NMR and Mass spectra.

Spectral data

16-(4-Methoxyphenyl)-2,3,4,16-tetrahydro-1*H*-benzo[*c*]chromeno-[2,3-*a*]phenazine-1-one(5a)^{13a}.

Colour: Yellow Solid; m.p: 283-289°C; IR (KBr) ν/cm^{-1} : 2926, 2836, 1655, 1370, 1207, 1029; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.10-2.20 (m, 2H, CH_2), 2.41-2.55 (m, 2H, CH_2), 2.80-2.88 (m, 1H, CH_2), 2.93-3.00 (m, 1H, CH_2), 3.62 (s, 3H, OCH_3), 6.09 (s, 1H, CH), 6.68 (d, J = 8.8 Hz, 2H, Ar-H), 7.50 (d, J = 8.8, 2H, Ar-H), 7.75-7.82 (m, 4H, Ar-H), 8.21-8.24 (m, 2H, Ar-H), 8.36 (d, 1H, J 7.3 Hz, Ar-H), 9.31-9.33 (m, 1H, Ar-H).

16-(4-Methylphenyl)-2,3,4,16-tetrahydro-1*H*-benzo[*c*]chromeno-[2,3-*a*]phenazine-1-one (5b)^{13a}.

Colour: Yellow Solid; m.p: 270-271°C; IR (KBr) ν/cm^{-1} : 2954, 1656, 1372, 1209; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.14 (s, 3H, CH_3), 2.15 -2. 20 (m, 2H, CH_2), 2.41-2.54 (m, 2H, CH_2), 2.80-2.88 (m, 1H, CH_2), 2.94-3.01 (m, 1H, CH_2), 6.13 (s, 1H, CH), 6.94 (d, 2H, J 7.79, Ar-H), 7.47 (d, 2H, J 7.79, Ar-H), 7.76-7.86 (m, 4H, Ar-H), 8.23-8.25 (m, 2H, Ar-H), 8.38-8.40 (m, 1H, Ar-H), 9.35-9.37 (m, 1H, Ar-H).

16-(4-Bromophenyl)-2,3,4,16-tetrahydro-1*H*-benzo[*c*]chromeno[3,2-*a*]phenazin-1-one (5c).

Colour: Yellow Solid; m.p: 274-278°C; IR (KBr) ν/cm^{-1} : 2955, 1601, 1375, 1207; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.08 -2.25 (m, 2H, CH_2) 2.45 -2.63 (m, 2H, CH_2), 2.81-2.89 (m, 1H, CH_2), 2.94-3.01 (m, 1H, CH_2), 6.08 (s, 1H, CH), 7.24-7.26 (m, 2H, Ar-H), 7.43-7.48 (m, 2H, Ar-H), 7.78-7.87 (m, 4H, Ar-H), 8.18-8.22 (m, 1H, Ar-H), 8.27-8.30 (m, 1H, Ar-H), 8.36-8.38 (m, 1H, Ar-H), 9.38-9.39 (m, 1H, Ar-H),; ^{13}C NMR (100 MHz, CDCl_3) δ_{C} = 20.50, 27.11, 27.56, 33.25,

37.09, 115.61, 115.94, 120.07, 122.00, 125.63, 126.37, 128.57, 129.22, 129.32, 129.74, 130.05, 130.19, 130.70, 130.80, 130.93, 140.51, 140.61, 141.69, 142.51, 144.17, 147.00, 165.28, 196.93; HRMS (ESI) m/z calcd for $C_{29}H_{19}BrN_2O_2$ [M^+]: 506.0630, found: 507.0691 [$M^+ + H$], 509.0716 [$(M^+ + H) + 2$].

16-(4-Chlorophenyl)-2,3,4,16-tetrehydro-1*H*-benzo[*c*]chromeno-[2,3-*a*]phenazine-1-one(5d)^{13a}.

Colour: Yellow Solid; m.p: 308-311°C; IR (KBr) v/cm^{-1} : 2922, 1653, 1364, 1206; ^1H NMR (400 MHz, $CDCl_3$) δ_H = 2.09-2.21 (m, 2H, CH_2), 2.42 -2.54 (m, 2H, CH_2), 2.81 -2.90 (m, 1H, CH_2), 2.81-3.01 (m, 1H, CH_2), 6.10 (s, 1H, CH), 7.08-7.11 (m, 2H, Ar-H), 7.48-7.51 (m, 2H, Ar-H), 7.82-7.87 (m, 4H, Ar-H), 8.22-8.24 (m, 1H, Ar-H), 8.38-8.40 (m, 1H, Ar-H), 8.43-8.46 (m, 1H, Ar-H), 9.51-9.52 (m, 1H, Ar-H).

16-(2-Methoxyphenyl)-2,3,4,16-tetrehydro-1*H*-benzo[*c*]chromeno-[2,3-*a*]phenazine-1-one (5e).

Colour: Yellow Solid; m.p: 286-288°C; IR (KBr) v/cm^{-1} : 2943, 1617, 1369, 1208; ^1H NMR (400 MHz, $CDCl_3$) δ_H = 2.08-2.20 (m, 2H, CH_2) 2.40-2.55 (m, 2H, CH_2), 2.80-2.88 (m, 1H, CH_2), 2.94-3.01 (m, 1H, CH_2), 3.63 (s, 3H, OCH_3), 6.66-6.68 (m, 2H, Ar-H), 7.48-7.51 (m, 2H, Ar-H), 7.77-7.84 (m, 4H, Ar-H), 8.23-8.26 (m, 2H, Ar-H), 8.38 (d, 1H, J 7.32, Ar-H), 9.34-9.36 (m, 1H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C = 27.42, 29.46, 32.44, 32.76, 41.24, 50.96, 55.00, 113.26, 115.15, 116.29, 122.10, 126.10, 128.59, 129.38, 129.89, 130.19, 130.45, 137.13, 139.52, 142.14, 142.25, 142.68, 147.12, 157.78, 163.41, 196.85; HRMS (ESI) m/z calcd. for $C_{31}H_{24}N_2O_4$ [M^+]: 458.1630, found: 459.1708 [$M^+ + H$].

16-(3-Hydroxy-4-methoxyphenyl)-2,3,4,16-tetrahydro-1*H*-benzo[*c*]chromeno[3,2-*a*]phenazin-1-one (5f).

Colour: Yellow Solid; m.p: 200-205°C; IR (KBr) ν/cm^{-1} : 3431, 2958, 2834, 1655, 1370, 1202, 1174; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.16-2.22 (m, 2H, CH_2), 2.44-2.54 (m, 2H, CH_2), 2.82-2.86 (m, 1H, CH_2), 2.92-2.99 (m, 1H, CH_2), 5.31-5.46 (brs, 1H, OH), 6.07 (s, 1H, CH), 6.64 (d, 1H, J 8.54, Ar-H), 6.98-6.99 (m, 1H, Ar-H), 7.25-7.26 (m, 1H, Ar-H), 7.72-7.83 (m, 4H, Ar-H), 8.23-8.26 (m, 2H, Ar-H), 8.34 (d, 1H, J 7.32, Ar-H), 9.34 (d, 1H, J 7.93, Ar-H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} = 20.53, 27.57, 30.87, 32.78, 37.18, 55.70, 109.95, 114.71, 116.44, 121.14, 121.91, 125.41, 126.57, 128.24, 129.36, 129.49, 129.73, 129.83, 130.83, 138.47, 140.89, 141.70, 141.95, 142.56, 144.93, 145.10, 146.88, 165.10, 197.01; HRMS (ESI) m/z calcd for $\text{C}_{30}\text{H}_{22}\text{N}_2\text{O}_4$ [M^+]: 474.1580, found: 475.1670 [$\text{M}^+ + \text{H}$].

16-(3,4-Dimethoxyphenyl)-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[3,2-a]phenazin-1-one (5g).

Colour: Yellow Solid; m.p: 284-286°C; IR (KBr) ν/cm^{-1} : 2931, 1654, 1368, 1205; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.09-2.19 (m, 2H, CH_2), 2.43-2.57 (m, 2H, CH_2), 2.84-2.90 (m, 1H, CH_2), 2.94-3.01 (m, 1H, CH_2), 3.67 (s, 3H, OCH_3), 3.89 (s, 3H, OCH_3), 6.11 (s, 1H, CH), 5.58-6.65 (m, 1H, Ar-H), 6.84-6.86 (m, 1H, Ar-H), 7.43 (s, 1H, Ar-H), 7.83-7.85 (m, 4H, Ar-H), 8.24-8.27 (m, 1H, Ar-H), 8.38-8.40 (m, 1H, Ar-H), 8.52-8.58 (m, 1H, Ar-H), 9.56-9.65 (m, 1H, Ar-H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} = 20.57, 27.54, 32.85, 37.15, 55.62, 55.90, 110.67, 116.33, 122.00, 125.87, 128.50, 128.86, 129.11, 129.92, 130.05, 130.16, 130.25, 139.89, 140.09, 141.99, 142.48, 147.34, 148.12, 165.10, 197.07; HRMS (ESI) m/z calcd for $\text{C}_{31}\text{H}_{24}\text{N}_2\text{O}_4$ [M^+]: 488.1736, found: 489.1796 [$\text{M}^+ + \text{H}$].

16-(Naphthalen-2-yl)-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[3,2-a]phenazin-1-one (5h).

Colour: Yellow Solid; m.p: 291-293°C; IR (KBr) ν/cm^{-1} : 2950, 1618, 1366, 1209; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.09-2.20 (m, 2H, CH_2), 2.41-2.45 (m, 2H, CH_2), 2.84-2.93 (m, 1H, CH_2), 2.99-3.07 (m, 1H, CH_2), 6.31 (s, 1H, CH), 7.26-7.32 (m, 2H, Ar-H), 7.62-7.64 (m, 2H, Ar-H),

7.67-7.71 (m, 1H, Ar-H), 7.78-7.81 (m, 3H, Ar-H), 7.86-7.92 (m, 3H, Ar-H), 8.24-8.28 (m, 1H, Ar-H), 8.43-8.48 (m, 2H, Ar-H), 9.54-9.56 (m, 1H, Ar-H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} = 20.51, 27.60, 33.77, 37.12, 116.17, 116.35, 122.05, 125.22, 125.51, 125.68, 126.56, 127.31, 127.39, 127.50, 127.77, 128.45, 129.03, 129.38, 129.67, 129.93, 130.05, 130.59, 132.18, 133.24, 140.37, 141.92, 142.56, 142.73, 147.02, 196.97; HRMS (ESI) m/z calcd for $\text{C}_{33}\text{H}_{22}\text{N}_2\text{O}_2$ [M^+]: 478.1681, found: 479.1747 [$\text{M}^+ + \text{H}$].

16-(2,4-Dichlorophenyl)-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[3,2-a]phenazin-1-one (5i).

Colour: Yellow Solid; m.p: 298-300°C; IR (KBr) ν/cm^{-1} : 2922, 1653, 1364, 1206; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 2.12-2.15 (m, 2H, CH_2), 2.48-2.49 (brs, 2H, CH_2), 2.87-2.92 (m, 2H, CH_2), 6.18 (s, 1H, CH), 6.99-7.01 (m, 1H, Ar-H), 7.22 (brs, 1H, Ar-H), 7.46-7.47 (m, 1H, Ar-H), 7.78-7.80 (m, 4H, Ar-H), 8.33-8.34 (m, 1H, Ar-H), 9.30 (brs, 1H, Ar-H); ^{13}C NMR (100 MHz, CDCl_3) δ_{C} = 20.53, 27.68, 32.70, 37.21, 114.39, 114.45, 122.07, 125.40, 126.08, 126.41, 128.61, 129.31, 129.35, 129.55, 129.89, 129.92, 130.94, 132.25, 133.26, 135.08, 140.40, 140.69, 140.74, 141.67, 142.44, 146.97, 165.38, 196.88; HRMS (ESI) m/z calcd for $\text{C}_{29}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_2$ [M^+]: 496.0745, found: 497.0815 [$\text{M}^+ + \text{H}$].

16-(4-Methoxyphenyl)-3,3-dimethyl-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[2,3-a]phenazin-1-one (5j) ^{13a} .

Colour: Yellow Solid; m.p: 197-200°C; IR (KBr) ν/cm^{-1} : 2927, 1601, 1375, 1202; ^1H NMR (400 MHz, CDCl_3) δ_{H} = 1.11 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 2.29 and 2.39 (AB system, 2H, J 16.48, $\text{CH}_a\text{CH}_b\text{CO}$), 2.75 and 2.81 (AB system, 2H, J 17.08, $\text{CH}_a\text{CH}_b\text{C}(\text{CH}_3)_2$), 3.63 (s, 3H, OCH_3), 6.09 (s, 1H, CH), 6.67 (d, 2H, J 8.54, Ar-H), 7.49 (d, 2H, J 8.54, Ar-H), 7.82-7.87 (m, 4H, Ar-

H), 8.27-8.30 (m, 1H, Ar-H), 8.38-8.40 (m, 1H, Ar-H), 8.49-8.51 (m, 1H, Ar-H) 9.55-9.57 (m, 1H, Ar-H).

3,3-Dimethyl-16-(4-methylphenyl)-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[2,3-a]phenazin-1-one (5k) ^{13a}.

Colour: Yellow Solid; m.p: 309-310°C ; IR (KBr) ν/cm^{-1} : 2928, 1601, 1367, 1206; ¹H NMR (400 MHz, CDCl₃) δ_{H} = 1.12 (s, 3H, CH₃) 1.18 (s, 3H, CH₃), 2.14 (s, 1H, CH₃), 2.29 and 2.39 (AB system, 2H, J 16.1, CH_aCH_bCO), 2.72 and 2.83 (AB system, 2H, J 17.6, CH_aCH_bC(CH₃)₂), 6.10 (s, 1H, CH), 6.99 (d, 2H, J 8.0, Ar-H), 7.49 (d, 2H, J 8.0, Ar-H), 7.74-7.85 (m, 4H, Ar-H), 8.22-8.25 (m, 2H, Ar-H), 8.36-8.38 (m, 1H, Ar-H), 9.33-9.35 (m, 1H, Ar-H).

16-(2-Methoxyphenyl)-3,3-dimethyl-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[2,3-a]phenazin-1-one (5l).

Colour: Yellow Solid; m.p: 228-230°C; IR (KBr) ν/cm^{-1} : 2952, 1657, 1373, 1202; ¹H NMR (400 MHz, CDCl₃) δ_{H} = 1.12 (s, 3H, CH₃) 1.18 (s, 3H, CH₃), 2.31 and 2.38 (AB system, 2H, J 16.48, CH_aCH_bCO) 2.76 and 2.78 (AB system, 2H, J 16.17, CH_aCH_b C(CH₃)₂), 3.63 (s, 3H, OCH₃), 6.08 (s, 1H, CH), 6.67 (d, 2H, J 8.54, Ar-H), 7.49 (d, 2H, J 9.16, Ar-H), 7.76-7.82 (m, 4H, Ar-H), 8.22-8.25 (m, 2H, Ar-H), 8.34 (d, 1H, J 8.54, Ar-H), 9.35 (d, 1H, J 7.32, Ar-H); ¹³C NMR (100 MHz, CDCl₃) δ_{C} = 27.44, 29.44, 32.43, 32.72, 41.29, 51.01, 55.00, 113.26, 115.27, 116.52, 121.89, 125.49, 126.59, 128.25, 129.30, 129.43, 129.79, 129.85, 129.91, 130.76, 137.27, 140.77, 141.93, 142.56, 146.84, 157.85, 163.37, 196.84; HRMS (ESI) m/z calcd for C₃₂H₂₆N₂O₃ [M⁺]: 486.1943, found: 487.2013 [M⁺+H].

16-(4-Methoxyphenyl)-3-methyl-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[3,2-a]phenazin-1-one (5m)

Colour: Yellow Solid; m.p: 244-246°C; IR (KBr) ν/cm^{-1} : 2925, 1654, 1374, 1289, 1200; ¹H NMR (400 MHz, CDCl₃) δ_{H} = 1.16-1.18 (m, 3H, CH₃), 2.14-2.21 (m, 1H, CH₂), 2.34-2.41 (m, 1H, CH), 2.50-2.56 (m, 2H, CH₂), 2.94-3.01 (m, 1H, CH₂), 3.62 (s, 3H, OCH₃), 6.06-6.08 (m,

1H, CH), 6.66-6.68 (m, 2H, Ar-H), 7.46-7.49 (m, 2H, Ar-H), 7.77-7.82 (m, 4H, Ar-H), 8.22-8.24 (m, 1H, Ar-H), 8.28-8.29 (m, 1H, Ar-H), 8.34-8.35 (m, 1H, Ar-H), 9.36-9.38 (m, 1H, Ar-H); ¹³C NMR (100 MHz, CDCl₃) δ_C = 20.96, 28.10, 32.60, 35.67, 45.46, 55.00, 113.26, 116.08, 116.51, 121.91, 125.65, 126.58, 126.58, 128.35, 128.98, 129.41, 129.68, 129.85, 129.92, 130.05, 130.35, 137.37, 140.25, 140.33, 141.94, 142.55, 146.88, 157.83, 164.70, 197.04; HRMS (ESI) m/z calcd for C₃₁H₂₄N₂O₃[M⁺]: 472.1787, found: 473.1854 [M⁺ + H].

16-(4-Bromophenyl)-3-methyl-2,3,4,16-tetrahydro-1H-benzo[c]chromeno[3,2-a]phenazin-1-one (5n).

Colour: Yellow Solid; m.p: 256-258°C; IR (KBr) ν/cm^{-1} : 2926, 1654, 1374, 1199; ¹H NMR (400 MHz, CDCl₃) δ 1.19-1.17 (m, 3H, CH₃) 2.15-2.22 (m, 1H, CH₂), 2.32-2.40 (m, 1H, CH), 2.52-2.59 (m, 2H, CH₂), 2.96-3.02 (m, 1H, CH₂), 6.07 (s, 1H, CH), 7.25-7.26 (m, 2H, Ar-H), 7.42-7.45 (m, 2H, Ar-H), 7.77-7.87 (m, 4H, Ar-H), 8.19-8.21 (m, 1H, Ar-H), 8.29-8.31 (m, 1H, Ar-H), 8.35-8.37 (m, 1H, Ar-H), 9.38-9.40 (m, 1H, Ar-H); ¹³C NMR (100 MHz, CDCl₃) δ_C = 20.94, 28.08, 33.23, 35.65, 45.39, 115.36, 115.50, 115.64, 120.06, 121.97, 125.64, 126.41, 128.56, 129.13, 129.30, 129.78, 130.05, 130.74, 130.82, 130.93, 140.42, 141.67, 142.48, 144.16, 146.95, 164.96, 196.94; HRMS (ESI) m/z calcd for C₃₀H₂₁BrN₂O₂ [M⁺]: 520.0786, found: 521.1649 [M⁺ + H], 523.0838 [(M⁺ + H) + 2].

Figure 1. ^1H NMR spectrum of compound 5a (400 MHz, CDCl_3).

Figure 2. ^1H NMR spectrum of compound 5b (400 MHz, CDCl_3).

Figure 3. ^1H NMR spectrum of compound 5c (400 MHz, CDCl_3).

Figure 4. ^{13}C NMR spectrum of compound 5c (100 MHz, CDCl_3).

Figure 5. ¹H NMR spectrum of compound 5d (400 MHz, CDCl₃).

Figure 6. ¹H NMR spectrum of compound 5e (400 MHz, CDCl₃).

Figure 7. ^{13}C NMR spectrum of compound 5e (100 MHz, CDCl_3).

Figure 9. ^{13}C NMR spectrum of compound 5f (100 MHz, CDCl_3).

Figure 10. ^1H NMR spectrum of compound 5g (400 MHz, CDCl_3).

Figure 11. ¹³C NMR spectrum of compound 5g (100 MHz, CDCl₃).

Figure 12. ¹H NMR spectrum of compound 5h (400 MHz, CDCl₃).

Figure 13. ¹³C NMR spectrum of compound 5h (100 MHz, CDCl₃).

Figure 14. ¹H NMR spectrum of compound 5i (400 MHz, CDCl₃).

Figure 21. ^{13}C NMR spectrum of compound 5m (100 MHz, CDCl_3).

Figure 22. ^1H NMR spectrum of compound 5n (400 MHz, CDCl_3).

Figure 23. ^{13}C NMR spectrum of compound 5n (100 MHz, CDCl_3).