# Highly Stereoselective Formal [3+3] Cycloaddition of Enals and Azomethine Imines Catalyzed by *N*-Heterocyclic Carbenes

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## Supporting Information

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**General Methods:**

Dichloromethane was purified by passage through a bed of activated alumina. Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and potassium permanganate ($KMnO_4$) stain. Melting points (**mp**) were obtained on a Thomas Hoover Capillary Melting Point Apparatus and are uncorrected. Infrared spectra (**IR**) were obtained on a Bio-Rad FTS-40 FTIR spectrophotometer. Infrared spectra were obtained as a thin film on a NaCl disk. Proton nuclear magnetic resonances (**$^1$H NMR**) were recorded in deuterated solvents on a Varian Inova 400 (400 MHz) spectrometer. Chemical shifts are reported in parts per million (ppm, δ) relative to the residual protio solvent ($CDCl_3$, δ 7.26; $D_2O$, δ 4.80; DMSO, δ 2.50, $CD_3OD$, δ 3.31) $^1$H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), or septet (sep). Splitting patterns that could not be interpreted or easily visualized are designated as multiplet (m) or broad (br). Coupling constants are reported in Hertz (Hz). Proton-decoupled (**$^{13}$C-NMR**) spectra were recorded on a Varian Inova 400 (100 MHz) spectrometer and are reported in ppm using the solvent as an internal standard ($CDCl_3$, δ 77.23; DMSO, δ 39.5, $CD_3OD$, δ 49.15). 1-D NOE and 2-D COSY experiments were performed on a Varian Inova 500 (500 MHz) spectrometer. Electrospray mass spectra (**ESI-MS**) were obtained using a Micromass Quattro II Triple Quadrupole HPLC/MS/MS Mass Spectrometer. Unless otherwise noted, all other commercially available reagents and solvents were purchased from Aldrich and used without further purification.

Azomethine imines were prepared according to the general procedure of by Fu and coworkers[1,2] and Hayashi and coworkers.[3] 4-Chloro cinnamaldehyde was prepared according to a procedure analogous to Moloney.[4] 3-methyl cinnamaldehyde, 3-(naphthalene-5-yl)acrylaldehyde, 3-(naphthalene-6-yl)acrylaldehyde, and 3-methoxy cinnamaldehyde were prepared according to the general procedure of Cacchi and coworkers.[5] 4-Methoxy cinnamaldehyde was purchased from Acros Chemical Company and the remaining aldehydes were commercially available from Sigma-Aldrich Chemical Company and distilled prior to their use.

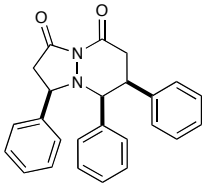
**Typical Procedure for the [3+3] cycloaddition of azomethine imine and enals:**

Into an oven-dried screw-capped test tube (SCTT) equipped with a stirbar was charged with the *N*-mesityl-*N*-methylbenzimidazolium iodide salt (16.6 mg, 0.044 mmol) and the 1-benzylidene-3-oxo-5-phenylpyrazolidin-1-ium-2-ide (55 mg, 0.22 mmol), capped and then purged with $N_2$. The two solids were then dissolved in $CH_2Cl_2$ (880 μL) and to the solution was added cinnamaldehyde (55 μL, 0.44 mmol). The SCTT was heated to 45 $^o$C and DBU (6.6 μL, 0.044 mmol) was added last. Upon DBU addition, the reaction turned immediately green then orange/red. After 2 h at 45 °C, analysis of the reaction (by thin layer chromatography) indicated completely conversion. The dark green or deep red (depending on the substrate) reaction mixture was cooled room temperature. The reaction was diluted with $CH_2Cl_2$, washed with water (1 x 5 mL) and saturated NaCl (1 x 5 mL). The layers were separated and the aqueous layer was

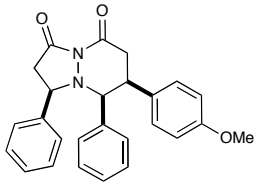
1. Shintani, R. J.; Fu, G.C. *J. Am. Chem. Soc.* **2003**, *125*, 10778-10779.
2. Suarez, A.; Downey, C.W.; Fu, G.C. *J. Am. Chem. Soc.* **2005**, *127*, 11244-11245.
3. Shintani, R.; Hayashi, T. *J. Am. Chem. Soc.* **2006**, *128*, 6330-6331.
4. Baldwin, J.E.; Turner, S.C.M.; Moloney, M.G. *Tetrahedron*, **1994**, *50*, 9411-9424.
5. Battistuzzi, G.; Cacchi, S.; Fabrizi, G. *Org. Lett.* **2003**, *5(5)*, 777-780.

extracted two additional times with $CH_2Cl_2$ (2 x 10 ml). The organic layers were combined, dried over $Na_2SO_4$, filtered and concentrated in vacuo. The resulting residue was purified by flash column chromatography on silica gel.

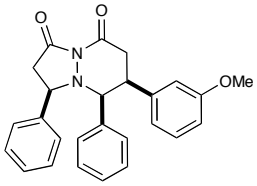
**Characterization of pyridazinediones**



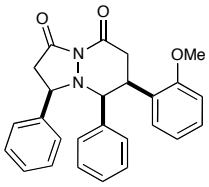
**tetrahydro-3,5,6-triphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (4):** Purified with 8% ethyl acetate/dichloromethane, yielding 66 mg (79%) of **4** as a white foam. $R_f$ = 0.63 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3031.2, 2920.7, 1773.6, 1299.9, 698.2 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.13-7.04 (m, 8H); 6.94-6.91 (m, 1H); 6.81 (m, 4H); 6.69-6.67 (d, *J* = 7.6 Hz, 2H); 4.48 (d, *J* = 7.6 Hz, 1H); 4.26 (t, *J* = 8.2, 1H); 3.67-3.61 (m, 1H); 3.34-3.22 (m, 2H); 2.85-2.74 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 169.1, 168.7, 140.1, 138.5, 136.2, 129.2, 128.9, 128.8, 128.7, 128.2, 127.9, 127.6, 127.2, 126.9, 72.2, 63.6, 44.6, 40.4, 37.6; LRMS (electrospray): Mass calculated for $C_{25}H_{22}N_2O_2$ $[2M + Na]^+$, 787.9. Found 787.5.



**tetrahydro-6-(4-methoxyphenyl)-3,5-diphenyl-5*H*pyrazolo[1,2*a*]pyridazine-1,8-dione (5):** Purified with 8% ethyl acetate/dichloromethane, yielding 75 mg (76%) of **5** as a yellow foam. $R_f$ = 0.60 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3033.2, 2931.8, 2836.8, 1773.0, 1296.7, 699.8 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.13-7.11 (m, 3H); 7.03-7.02 (m, 2H); 6.93 (t, *J* = 7.0 Hz, 1H); 6.82 (t, *J* = 7.6 Hz, 2H); 6.74-6.71 (m, 2H); 6.67-6.60 (m, 4H); 4.42 (d, *J* = 7.0 Hz, 1H); 4.24 (t, *J* = 8.2 Hz, 1H); 3.70 (s, 3H); 3.59-3.53 (m, 1H); 3.26-3.20 (m, 2H); 2.83-2.73 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 169.0, 168.6, 158.6, 140.1, 136.3, 130.6, 129.9, 129.3, 128.7, 128.6, 127.9, 127.6, 126.9, 113.5, 72.3, 63.9, 55.4, 43.9, 40.5, 38.0; LRMS (electrospray): Mass calculated for $C_{26}H_{24}N_2O_3$ $[2M + Na]^+$, 847.9. Found 847.2.

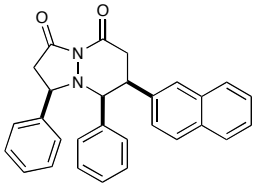


**tetrahydro-6-(3-methoxyphenyl)-3,5-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (6):** Purified with 8% ethyl acetate/dichloromethane, yielding 78 mg (79%) of **6** as a light tan foam. $R_f$ = 0.65 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3031.6, 2919.6, 2836.7, 1773.6, 1297.7, 699.9 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.15-7.13 (m, 3H); 7.06-7.04 (m, 2H); 7.01-6.93 (m, 2H); 6.85 (t, *J* = 7.6 Hz, 2H); 6.74 (d, *J* = 7.6 Hz, 2H); 6.61-6.59 (m, 1H); 6.44 (d, *J* = 7.6 Hz, 1H); 6.29 (s, 1H); 4.48 (d, *J* = 7.6 Hz, 1H); 4.27 (t, *J* = 8.8 Hz, 1H); 3.68-3.55 (m, 4H); 3.33-3.23 (m, 2H); 2.82-2.73 (m, 2H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 169.1, 168.6, 159.3, 140.1, 139.9, 136.3, 129.2, 129.1, 128.7, 127.9, 127.6, 126.9, 126.8, 121.3, 114.7, 112.6, 72.0, 63.5, 55.3, 44.6, 40.3, 37.5; LRMS (electrospray): Mass calculated for $C_{26}H_{24}N_2O_3$ $[2M + Na]^+$, 847.9. Found 847.4.

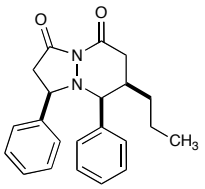


**tetrahydro-6-(2-methoxyphenyl)-3,5-diphenyl-5*H*-pyrazolo[1,2-*a*]pyrida-zine-1,8-dione (7):** Purified with 8% ethyl acetate/dichloromethane, yielding 93 mg (94%) of **7** as a light yellow foam. $R_f$ = 0.62 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3036.4, 2921.5, 2843.8, 1773.6, 1297.2, 699.1 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.18-7.16 (m, 3H); 7.07-6.97 (m, 3H); 6.93-6.84 (m, 5H); 6.65-6.61 (m, 2H); 6.57 (d, *J* = 8.2 Hz,

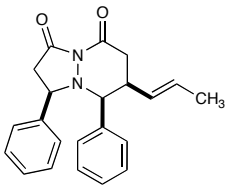
1H); 4.67 (d, *J* = 8.8 Hz, 1H); 4.30-4.27 (m, 1H); 4.11-4.05 (m, 1H); 3.76 (s, 3H); 3.45-3.32 (m, 2H); 2.79-2.63 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.6, 169.4, 156.4, 140.4, 137.2, 128.8, 128.7, 128.1, 127.9, 127.8, 127.5, 127.4, 127.3, 126.7, 120.3, 109.7, 69.7, 62.4, 55.2, 39.9, 37.5, 36.4; LRMS (electrospray): Mass calculated for $C_{26}H_{24}N_2O_3$ [2M + Na]$^+$, 847.9. Found 847.3.



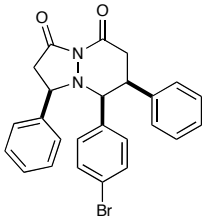
**tetrahydro-6-(naphthalene-3-yl)-3,5-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (8):** Purified with 8% ethyl acetate/dichloromethane, yielding 80 mg (77%) of **8** as a light brown foam. $R_f$ = 0.60 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3052.51, 2921.1, 1773.3, 1298.3, 699.1 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.71-7.63 (m, 2H); 7.53 (d, *J* = 8.2 Hz, 1H); 7.42-7.39 (m, 2H); 7.30 (s,1H); 7.12-7.11 (m, 3H); 7.04-6.99 (m, 2H); 6.93-6.91 (m, 1H); 6.87-6.83 (m, 1H); 6.75-6.68 (m, 4H); 4.56 (d, *J* = 7.0 Hz, 1H); 4.28 (t, *J* = 8.2 Hz, 1H); 3.81-3.75 (m, 1H); 3.43-3.23 (m, 2H); 2.94-2.76 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.1, 168.5, 140.1, 136.1, 136.0, 133.1, 132.4, 129.2, 128.7, 128.6, 128.0, 127.9, 127.8, 127.8, 127.7, 127.7, 127.6, 126.9, 126.3, 126.1, 72.2, 63.8, 44.8, 40.5, 37.9; LRMS (electrospray): Mass calculated for $C_{25}H_{22}N_2O_2$ [2M + Na]$^+$, 888.0. Found 888.2.



**tetrahydro-3,5-diphenyl-6-propyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (9):** Purified with 8% ethyl acetate/dichloromethane, yielding 54 mg (70%) of **9** as a white foam. $R_f$ = 0.57 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3030.9, 2957.2, 1774.4, 1308.9, 699.7 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.16-7.11 (m, 8H); 7.03-7.01 (m, 2H); 4.27 (d, *J* = 8.8 Hz, 1H); 4.22-4.18 (m, 1H); 3.30-3.23 (m, 1H); 2.80-2.67 (m, 2H); 2.59-2.55 (m, 1H); 2.37-2.30 (m, 1H); 1.31-1.22 (m, 1H); 1.20-1.03 (m, 1H); 1.01-0.92 (m, 1H); 0.86-0.76 (m, 1H); 0.71 (t, *J* = 7.0 Hz; 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.6, 169.3, 140.5, 137.3, 129.5, 128.7, 128.4, 128.3, 127.9, 126.7, 71.2, 62.3, 39.8, 37.9, 37.4, 34.4, 20.9, 14.1; LRMS (electrospray): Mass calculated for $C_{29}H_{24}N_2O_2$ [2M + Na]$^+$, 719.9. Found 719.3.

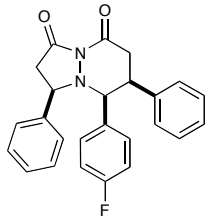


**tetrahydro-3,5-diphenyl-6-((E)-prop-1-enyl)-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (10):** Purified with 8% ethyl acetate/dichloromethane, yielding 39 mg (51%) of **10** as a white foam. $R_f$ = 0.58 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3030.9, 2918.0, 1774.6, 1696.9, 1297.7, 699.4 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.17-7.03 (m, 10H); 5.35-5.26 (m, 1H); 4.91-4.80 (m,1H); 4.29-4.18 (m, 2H); 3.27-3.20 (m, 1H); 2.95-2.88 (m, 2H); 2.73-2.68 (m, 1H); 2.54-2.49 (m, 1H); 1.46 (d, *J* = 7.0 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.2, 168.8, 140.5, 136.9, 129.4, 129.3, 129.1, 128.7, 128.2, 128.1, 127.8, 126.8, 71.6, 62.7, 42.4, 40.0, 38.8, 36.3; LRMS (electrospray): Mass calculated for $C_{22}H_{22}N_2O_2$ [2M + Na]$^+$, 715.8. Found 715.4.

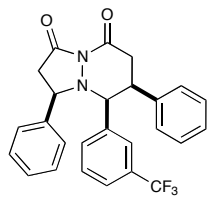


**5-(4-bromophenyl)-tetrahydro-3,6-diphenyl-5H-pyrazolo[1,2-a]pyridazine-1,8-dione (12):** Purified with 8% ethyl acetate/dichloromethane, yielding 85 mg (87%) of **12** as a light tan foam. $R_f$ = 0.59 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3047.5, 2919.6, 1773.3, 1300.0, 698.5 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.14-7.11 (m, 6H); 7.00-6.98 (m, 2H); 6.87 (m, 2H); 6.82 (m, 2H); 6.48 (m, 2H); 4.41 (d, *J* = 7.0 Hz, 1H); 4.19 (t, *J* = 8.2 Hz,

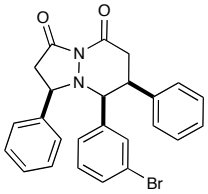
1H); 3.62-3.57 (m, 1H); 3.26-3.17 (m, 2H); 2.88-2.76 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.7, 168.2, 138.0, 135.2, 130.8, 130.6, 128.9, 128.8, 128.7, 128.4, 128.1, 127.5, 127.0, 121.9, 71.8, 64.7, 44.3, 40.6, 37.5; LRMS (electrospray): Mass calculated for $C_{25}H_{21}BrN_2O_2$ [2M + Na]$^+$, 945.7. Found 944.8.



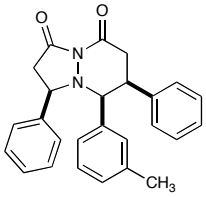
**5-(4-fluorophenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine -1,8-dione (13):** Purified with 8% ethyl acetate/dichloromethane, yielding 73 mg (82%) of **13** as a yellow foam. $R_f$ = 0.65 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3037.5, 2921.6, 1773.8, 1300.7, 699.3 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.13-7.09 (m, 6H); 7.00-6.98 (m, 2H); 6.82-6.80 (m, 2H); 6.62-6.58 (m, 2H); 6.49-6.45 (m, 2H); 4.44 (d, *J* = 7.6 Hz, 1H); 4.20 (t, *J* = 8.2 Hz, 1H); 3.63-3.57 (m, 1H); 3.29-3.19 (m, 2H); 2.87-2.75 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.8, 168.4, 139.9, 138.3, 130.9, 130.8, 128.9, 128.7, 128.3, 128.0, 127.4, 126.9, 114.5, 114.3, 71.8, 64.5, 44.6, 40.6, 37.5; LRMS (electrospray): Mass calculated for $C_{25}H_{21}FN_2O_2$ [2M + Na]$^+$, 823.9. Found 823.1.



**5-(3-trifluoromethylphenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]-pyridazine-1,8-dione (14):** Purified with 8% ethyl acetate/dichloromethane, yielding 92 mg (93%) of **14** as a yellow foam. $R_f$ = 0.63 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3032.8, 2920.8, 1774.7, 1326.6, 1300.9, 1122.8, 699.4 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.14-7.06 (m, 7H); 7.00-6.97 (m, 1H); 6.93-6.76 (m, 6H); 4.50 (d, *J* = 7.0 Hz, 1H); 4.20 (t, *J* = 8.8 Hz, 1H); 3.67-3.62 (m, 1H); 3.29-3.17 (m, 2H); 2.89-2.81 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.5, 168.3, 139.2, 137.7, 137.3, 132.4, 132.3, 128.8, 128.7, 128.4, 128.3, 127.9, 127.6, 127.1, 126.2, 126.2, 124.6, 72.5, 65.5, 44.5, 40.7, 37.3; LRMS (electrospray): Mass calculated for $C_{26}H_{21}F_3N_2O_2$ [2M + Na]$^+$, 923.9. Found 923.2.

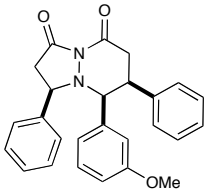


**5-(3-bromophenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine -1,8-dione (15):** Purified with 8% ethyl acetate/dichloromethane, yielding 76 mg (78%) of **15** as a white foam. $R_f$ = 0.65 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3035.2, 2921.5, 1774.2, 1296.2, 698.9 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.14-7.11 (m, 6H); 7.03-7.01 (m, 3H); 6.82-6.80 (m, 2H); 6.73 (s, 1H); 6.68-6.64 (m, 1H); 6.59-6.57 (m, 1H); 4.39 (d, *J* = 7.6 Hz, 1H); 4.21 (t, *J* = 8.2 Hz, 1H); 3.63-3.58 (m, 1H); 3.28-3.17 (m, 2H); 2.87-2.79 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.7, 168.3, 139.4, 138.4, 137.9, 132.6, 130.9, 129.0, 128.8, 128.7, 128.4, 128.3, 127.7, 127.5, 127.0, 121.6, 72.1, 65.0, 44.4, 40.5, 37.3; LRMS (electrospray): Mass calculated for $C_{25}H_{21}BrN_2O_2$ [2M + Na]$^+$, 945.7. Found 945.1.



**5-(3-methylphenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine -1,8-dione (16):** Purified with 8% ethyl acetate/dichloromethane, yielding 68 mg (76%) of **16** as a tan solid. $R_f$ = 0.66 (96/2/2 dichloromethane/ethyl acetate/methanol); Mp: 169-171 °C; IR (film) 3033.6, 2919.7, 1770.7, 1298.0, 698.7 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.12-7.08 (m, 6H); 7.02-7.00 (m, 2H); 6.84-6.82 (m, 2H); 6.76-6.70 (m, 2H); 6.55-6.53 (d, *J* = 6.4 Hz, 1H); 6.28 (s, 1H); 4.41 (d, *J* = 7.0 Hz, 1H); 4.23 (t, *J* = 8.2 Hz, 1H); 3.60-3.55 (m, 1H); 3.31-3.19 (m, 2H); 2.88-2.73 (m, 2H); 1.89 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.0, 168.5, 140.3, 138.3,

136.9, 135.8, 130.5, 129.0, 128.6, 128.5, 128.0, 127.9, 127.4, 127.2, 126.9, 126.2, 72.4, 63.8, 44.6, 40.4, 37.8, 21.2; LRMS (electrospray): Mass calculated for $C_{26}H_{24}N_2O_2$ [2M + Na]$^+$, 815.9. Found 815.5.




**5-(3-methoxyphenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (17):** Purified with 8% ethyl acetate/dichloromethane, yielding 59 mg (67%) of **17** as a yellow foam. $R_f$ = 0.61 (96/2/2 dichloromethane/ethyl acetate/methanol); IR (film) 3047.4, 2920.9, 2836.1, 1770.7, 1296.2, 698.5 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.13-7.11 (m, 6H); 7.05-7.03 (m, 2H); 6.88-6.80 (m, 3H); 6.48-6.43 (m, 2H); 5.96 (s, 1H); 4.44 (d, *J* = 7.0 Hz, 1H); 4.25 (t, *J* = 8.2 Hz, 1H); 3.61-3.58 (m, 1H); 3.29-3.21 (m, 5H); 2.91-2.73 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 168.9, 168.1, 159.5, 140.5, 138.8, 137.4, 129.1, 128.7, 128.6, 128.2, 127.9, 127.3, 126.8, 121.7, 114.7, 114.1, 72.3, 63.7, 54.8, 44.8, 40.5, 38.0; LRMS (electrospray): Mass calculated for $C_{26}H_{24}N_2O_3$ [2M + Na]$^+$, 847.9. Found 847.3.

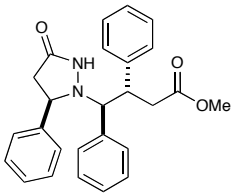
**Typical procedure for ring-opening of pyridazinedione:**

*Ester formation:*

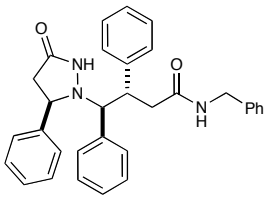
Into a flame-dried 10 mL round-bottom flask equipped with a stirbar was added the tetrahydro-3,5,6-triphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (**4**) (50 mg, 0.131 mmol) and dissolved in methanol (2.6 mL, 0.05 M). It was purged with $N_2$ and into it was added a solution of 0.1 N NaOH in methanol (1.31 mL, 0.131 mmol). The reaction mixture was allowed to stir at room temperature under a positive pressure of $N_2$. After 45 minutes, TLC showed the reaction was complete. The reaction was neutralized with 1N HCl in methanol. Solvent was removed and reaction was purified via flash chromatography.

*Amide formation:*

Into a flame-dried 10 mL round-bottom flask equipped with a stirbar was added the tetrahydro-3,5,6-triphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine-1,8-dione (**4**) (26 mg, 0.068 mmol) and dissolved in THF (1.13 mL, 0.06 M). It was purged with $N_2$ and into it was added benzyl amine (7.5 μL, 0.068 mmol). The reaction was allowed to stir at room temperature for overnight (12 h) under a positive pressure of $N_2$. TLC showed the reaction was complete and it was purified via flash chromatography.

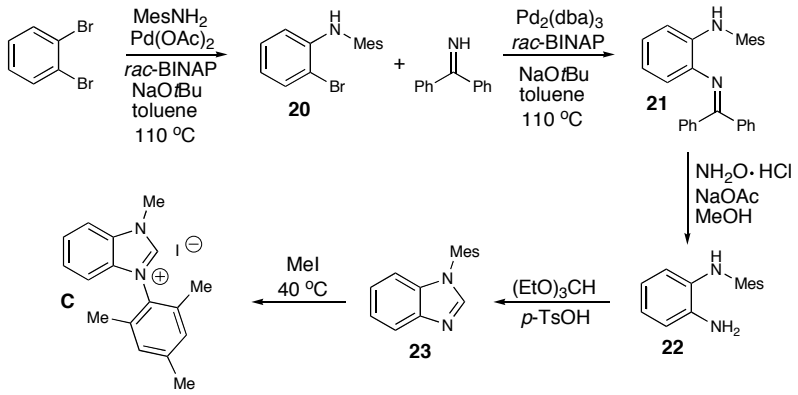


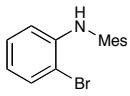

**Methyl 4-(3-oxo-5-phenylpyrazolidin-1-yl)-3,4-diphenylbutanoate (19):** Purified with 10% ethyl acetate/dichloromethane to 90/9/1 dichloromethane/ethyl acetate/methanol, yielding 54 mg (99%) of **19** as a white foam. $R_f$ = 0.59 (90/5/5 dichloromethane/ethyl acetate/methanol); IR (film) 3029.6, 2923.2, 2849.8, 1735.4, 1696.9, 700.4 cm$^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 8.01 (s. 1H); 7.28-7.09 (m, 11H); 6.70-6.69 (m, 4H); 4.27 (d, *J* = 8.8 Hz, 1H); 4.08-4.07 (m, 1H); 4.01-3.98 (m, 1H); 3.68 (s, 3H); 3.24-3.18 (m, 1H); 2.96-2.89 (m, 1H); 2.39-2.34 (m, 1H); 2.24-2.20 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.7, 174.5, 142.3, 140.8, 135.7, 129.6, 128.8, 128.7, 128.3, 128.2, 128.0, 127.5, 127.2, 126.1, 76.9, 63.6, 52.3, 43.3, 36.6, 33.3; LRMS (electrospray): Mass calculated for $C_{26}H_{26}N_2O_3$ [2M]$^+$, 829.0. Found 829.0.



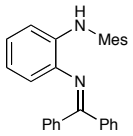

***N*-benzyl-4-(3-oxo-5-phenylpyrazolidin-1-yl)-3,4-diphenylbutanamide (20):** Purified with 90/9/1 dichloromethane/ethyl acetate/methanol, yielding 32 mg (96%) of **20** as a white foam. $R_f$ = 0.37 (90/5/5 dichloromethane/ethyl acetate/methanol); IR (film) 3046.4, 2920.1, 1687.7, 699.2 $cm^{-1}$; $^1H$ NMR (400 MHz, $CDCl_3$) δ 9.46 (s, 1H); 7.28-7.03 (m, 16H); 6.73-6.71 (m, 4H); 6.33 (s, 1H); 4.50-4.45 (m, 1H); 4.33-4.28 (m, 1H); 4.21 (d, J = 8.2 Hz, 1H); 4.04-4.03 (m, 2H); 3.09-3.02 (m, 1H); 2.84-2.79 (m, 1H); 2.40-2.35 (m, 1H); 2.01-1.97 (m, 1H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 174.5, 172.3, 142.6, 140.5, 138.2, 135.9, 129.2, 128.9, 128.8, 128.7, 128.3, 128.2, 128.0, 127.9, 127.6, 127.4, 127.2, 126.2, 77.7, 63.4, 44.0, 43.8, 37.0, 35.5; LRMS (electrospray): Mass calculated for $C_{32}H_{31}N_3O_2$ $[M + H]^+$, 489.6. Found 489.6.

**Synthesis of *N*1-mesityl-*N*3-methylbenzimidazolium Iodide (C):**

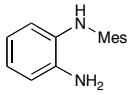


***N*-(2-bromophenyl)-2,4,6-trimethylbenzenamine (21):** To a flame-dried 50 ml Schlenk flask equipped with a stirbar was added $Pd(OAc)_2$ (149 mg, 0.714 mmol), *rac*-BINAP (620 mg, 0.995 mmol) and toluene (16.5 mL). It was heated to 60 °C and an orange suspension resulted. Into it was then added 1,2-dibromobenzene (1.96 g, 8.29 mmol), 2,4,6-trimethylaniline (1.35 g, 9.95 mmol) and NaO*t*Bu (620 mg, 10.78 mmol). The reaction was allowed to reflux at 110 °C with a condenser attached and stirred vigorously until reaction was complete as judged by thin layer chromatography (10% $CH_2Cl_2$/hexanes). The reaction was filtered through a pad of celite and rinsed with ethyl acetate (50 mL). Solvent was removed and purified via flash chromatography 0-5% $CH_2Cl_2$/hexanes. Isolated a white solid (1.99 g, 83% yield). $R_f$=0.83 (10% $CH_2Cl_2$/hexanes); $^1$H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.9 Hz, 1H); 7.01 (t, *J* = 7.6 Hz, 1H); 6.58 (t, *J* = 7.6 Hz, 1H); 6.14 (d, *J* = 7.9 Hz, 1H); 5.61 (s, 1H); 2.31 (s, 6H); 2.15 (s, 6H).

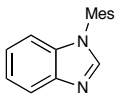


***N*1-mesityl-*N*2-(diphenylmethylene)benzene-1,2-diamine (22):** To a flame-dried 50 ml Schlenk flask equipped with a stirbar was added $Pd_2(dba)_3$ (68 mg, 0.0737 mmol), *rac*-BINAP (92 mg, 0.147 mmol) and toluene (15 mL). It was heated to 110 °C with condenser attached for 30 minutes and cooled to room temperature. Into it was then added **21** (1.07 g, 3.69 mmol), benzophenone imine (869 mg, 4.79 mmol) and NaO*t*Bu (461 mg, 4.79 mmol). The reaction was allowed to reflux at 110 °C until reaction was complete as judged by thin layer chromatography (10% $CH_2Cl_2$/hexanes). The reaction was filtered through a pad of celite and rinsed with ethyl acetate (50 mL). Solvent was removed and purified via flash chromatography 15-30% $CH_2Cl_2$/hexanes. Isolated a yellow solid (1.38 g, 96% yield). $R_f$=0.31 (10% $CH_2Cl_2$/hexanes); $^1$H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.5 Hz, 2H); 7.49-7.40 (m, 3H); 7.34 (m, 3H); 7.26 (m, 2H); 6.94 (s, 2H); 6.73 (t, *J* = 7.5 Hz, 1H); 6.35 (t, *J* = 7.5 Hz, 1H); 6.23 (d, *J* = 7.5 Hz, 1H); 6.13 (d, *J* = 7.9 Hz, 1H); 5.88 (s, 2H); 2.30 (s, 3H); 2.24 (s, 6H).

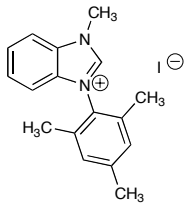


***N*1-mesitylbenzene-1,2-diamine (23):** To a flame-dried 100 ml round bottom flask equipped with a stirbar was added **22** (1.35 g, 3.46 mmol) and dissolved in THF (22 mL) and MeOH (22 mL). Into it was then added NaOAc (681 mg, 8.30 mmol) and $NH_2OH$•HCl (433 mg, 6.22 mmol). The cloudy orange solution was

stirred at room temperature until the reaction was complete as judged by thin layer chromatography. After 13 h, the light yellow solution was diluted by ethyl acetate and washed with 0.1 N NaOH (2 x 40 mL). Aqueous layer was extracted with ethyl acetate (2 x 30 mL). All organics were dried with $Na_2SO_4$, filtered and solvent removed. Reaction was purified via flash chromatography 15-30% $Et_2O$/hexanes. Isolated a tan solid (708 mg, 91% yield). $R_f$=0.29 (25% $Et_2O$/hexanes); $^1$H NMR (500 MHz, $CDCl_3$) δ 6.92 (s, 2H); 6.79-6.72 (m, 2H); 6.64 (t, *J* = 7.6 Hz, 1H); 6.23 (d, *J* = 7.9 Hz, 1H); 4.76 (s, b, 1H); 3.61 (s, b, 1H); 2.30 (s, 3H); 2.12 (s, 6H).

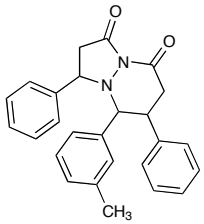



**1-mesityl-1*H*-benzo[*d*]imidazole (24):** To a flame-dried 200 ml round bottom flask equipped with a stirbar was added **23** (1.27g, 5.61 mmol) and dissolved in $(EtO)_3CH$ (61 mL). Into it was added *p*TsOH (107 mg, 0.561 mmol) and the orange solution was stirred at room temperature until the reaction was complete as judged by thin layer chromatography. After 14 h, the orange solution was diluted with EtOAc and washed with saturated $NaHCO_3$ (2 x 50 mL). The aqueous layer was extracted with ethyl acetate (2 x 30 mL). The organics were dried with $Na_2SO_4$, filtered and removed solvent. The reaction was purified via flash chromatography 5% MeOH/$CH_2Cl_2$. Isolated an orange oil (1.32 g, 99% yield). $R_f$=0.45 (5% MeOH/$CH_2Cl_2$); $^1$H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.9 Hz, 1H); 7.86 (s, 1H); 7.34-7.25 (m, 2H); 7.05-7.02 (m, 3H); 2.39 (s, 3H); 1.92 (s, 6H).




***N*1-mesityl-*N*3-methylbenzimidazolium Iodide (C):** Into a flame-dried 50 mL round bottom flask equipped with a stirbar was added **24** (1.31 g, 5.54 mmol) and MeI (22 mL, 0.25 M). Heat to 40 $^o$C with a condenser attached. After 16 h, reaction was complete as judged by thin layer chromatography. Excess MeI was removed via vacuo and solid was washed with ether and filtered to give pure product; yielding 1.91 g (91%) of **C** as a tan solid. Mp: 266-269 °C; IR (film) 3018.1, 2947.8, 1610.2, 1560.3, 1260.7, 753.8, 731.9 $cm^{-1}$; $^1$H NMR (400 MHz, $CDCl_3$) δ 10.75 (s, 1H); 7.90 (d, *J* = 8.2 Hz, 1H); 7.72 (t, *J* = 7.6 Hz, 1H); 7.62 (t, *J* = 7.6, 1H); 7.23 (t, *J* = 8.2 Hz, 1H); 7.06 (s, 2H); 4.54 (s, 3H); 2.37 (s, 3H); 2.02 (s, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 142.6, 141.9, 135.5, 132.0, 131.4, 130.3, 128.2, 127.9, 127.8, 113.5, 113.3, 35.2, 21.3, 18.2; LRMS (electrospray): Mass calculated for $C_{17}H_{19}IN_2$ $[2M - I]^+$, 629.6. Found 629.1.

**Structural Analysis of X-ray Crystal Structure:**
**5-(3-methylphenyl)-tetrahydro-3,6-diphenyl-5*H*-pyrazolo[1,2-*a*]pyridazine -1,8-dione (16):**



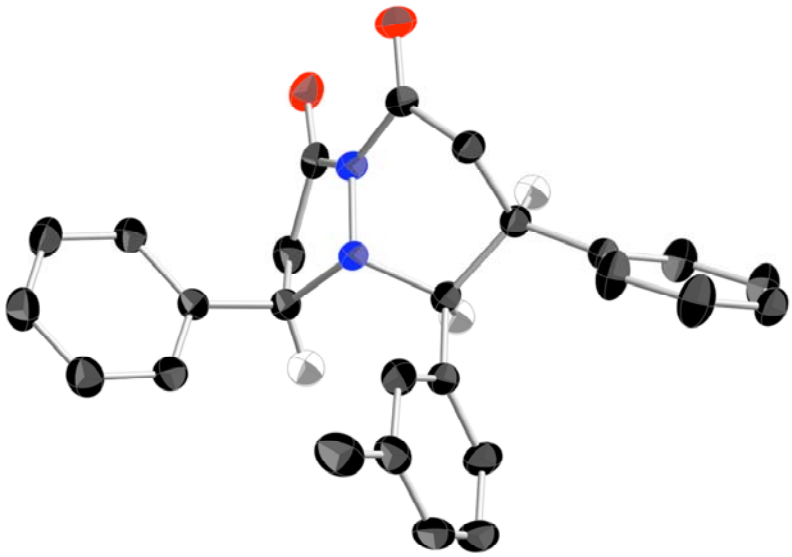



Table 1. Crystal data and structure refinement for **16**.

| | | |
|---|---|---|
| Identification Code | s26um | |
| Empirical formula | $C_{26}H_{24}N_2O_2$ | |
| Formula weight | 396.47 | |
| Temperature | 153 (2) K | |
| Wavelength | 0.71073 Å | |
| Crystal System | Monoclinic | |
| Space group | P-1 | |
| Unit cell dimensions | a = 9.4020 (7) Å | α = 93.6650(10)°. |
| | b = 10.0753 (7) Å | β = 95.4380 (10)°. |
| | c = 10.8800 (8) Å | χ = 91.5370 (10)°. |
| Volume | 1023.34 (13) $Å^3$ | |
| Z | 2 | |
| Absorption coefficient | 0.082 $mm^{-1}$ | |
| F(000) | 420 | |
| Theta range for data collection | 1.88 to 28.82° | |
| Index ranges | -12<=h<=12, -13<=k<=13, -14<=l<=14 | |
| Reflections collected | 4750 | |
| Independent reflections | 4137 | |
| Completeness of theta = 28.82° | 98.7% | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |

| | |
|---|---|
| Data/ restraints/ parameters | 4750/0/281 |
| Goodness-of-fit on $F^2$ | 1.061 |
| Final R indices [I>2sigma(I)] | R1 = 0.0678, wR2 = 0.1805 |
| R indices (all data) | R1 = 0.0612, wR2 = 0.1746 |

Table 2. Atomic coordinates and equivalent isotropic displacement parameters for **16**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C1 | 0.89261(19) | 0.40262(17) | 0.40866(15) | 0.0261(4) |
| C2 | 0.76481(19) | 0.48806(18) | 0.42032(17) | 0.0290(4) |
| H2A | 0.7326 | 0.5243 | 0.3403 | 0.035 |
| H2B | 0.7869 | 0.5628 | 0.4835 | 0.035 |
| C3 | 0.65163(18) | 0.39174(17) | 0.46004(15) | 0.0256(4) |
| H3 | 0.590(2) | 0.441(2) | 0.517(2) | 0.031 |
| C4 | 0.55712(18) | 0.32172(17) | 0.35358(15) | 0.0251(3) |
| C5 | 0.60381(19) | 0.29806(18) | 0.23619(16) | 0.0292(4) |
| H5 | 0.6979 | 0.3257 | 0.2219 | 0.035 |
| C6 | 0.5142(2) | 0.2346(2) | 0.14022(17) | 0.0342(4) |
| H6 | 0.5476 | 0.2176 | 0.0612 | 0.041 |
| C7 | 0.3755(2) | 0.1962(2) | 0.15991(19) | 0.0379(4) |
| H7 | 0.3131 | 0.1554 | 0.0937 | 0.046 |
| C8 | 0.3283(2) | 0.2175(2) | 0.27644(19) | 0.0362(4) |
| H8 | 0.2340 | 0.1902 | 0.2903 | 0.043 |
| C9 | 0.41930(19) | 0.27897(19) | 0.37289(17) | 0.0304(4) |
| H9 | 0.3871 | 0.2919 | 0.4528 | 0.036 |
| C10 | 0.96511(19) | 0.19543(17) | 0.51457(16) | 0.0269(4) |
| C11 | 0.92647(19) | 0.14191(17) | 0.63379(16) | 0.0269(4) |
| H11A | 1.0063 | 0.0916 | 0.6718 | 0.032 |
| H11B | 0.8407 | 0.0818 | 0.6183 | 0.032 |
| C12 | 0.89631(18) | 0.26403(17) | 0.71980(15) | 0.0243(3) |
| H12 | 0.977(2) | 0.330(2) | 0.714(2) | 0.029 |
| C13 | 0.75874(18) | 0.33226(17) | 0.66682(15) | 0.0246(3) |
| H13 | 0.775(2) | 0.431(2) | 0.678(2) | 0.029 |
| C14 | 0.61976(19) | 0.29545(19) | 0.71858(16) | 0.0294(4) |
| C15 | 0.5444(2) | 0.1783(2) | 0.67904(17) | 0.0341(4) |
| H15 | 0.5826 | 0.1174 | 0.6216 | 0.041 |
| C16 | 0.4124(2) | 0.1481(2) | 0.72244(19) | 0 .0399(5) |
| C17 | 0.3596(2) | 0.2378(3) | 0.8082(2) | 0.0470(6) |
| H17 | 0.2709 | 0.2179 | 0.8392 | 0.056 |
| C18 | 0.4327(2) | 0.3536(3) | 0.8486(2) | 0.0448(5) |
| H18 | 0.3949 | 0.4135 | 0.9072 | 0.054 |
| C19 | 0.5632(2) | 0.3841(2) | 0.80346(18) | 0.0382(5) |
| H19 | 0.6135 | 0.4654 | 0.8307 | 0.046 |
| C20 | 0.90166(18) | 0.23525(17) | 0.85482(15) | 0.0258(3) |
| C21 | 0.9809(2) | 0.3210(2) | 0.94208(18) | 0.0357(4) |
| H21 | 1.0269 | 0.3979 | 0.9160 | 0.043 |

| | | | | |
|---|---|---|---|---|
| C22 | 0.9943(2) | 0.2964(2) | 1.06702(19) | 0.0408(5) |
| H22 | 1.0492 | 0.3562 | 1.1252 | 0.049 |
| C23 | 0.9277(2) | 0.1854(2) | 1.10637(17) | 0.0368(4) |
| H23 | 0.9368 | 0.1677 | 1.1914 | 0.044 |
| C24 | 0.8481(3) | 0.1004(2) | 1.0208(2) | 0.0461(5) |
| H24 | 0.8016 | 0.0240 | 1.0474 | 0.055 |
| C25 | 0.8345(2) | 0.1248(2) | 0.89591(18) | 0.0400(5) |
| H25 | 0.7787 | 0.0651 | 0.8383 | 0.048 |
| C26 | 0.3280(3) | 0.0294(3) | 0.6728(3) | 0.0519(6) |
| H26A | 0.2685 | 0.0499 | 0.5979 | 0.078 |
| H26B | 0.2666 | 0.0007 | 0.7348 | 0.078 |
| H26C | 0.3922 | -0.0421 | 0.6525 | 0.078 |
| N1 | 0.86872(15) | 0.29142(14) | 0.47540(13) | 0.0242(3) |
| N2 | 0.73646(15) | 0.29301(14) | 0.53109(12) | 0.0236(3) |
| O1 | 0.99650(14) | 0.42288(13) | 0.35390(11) | 0.0317(3) |
| O2 | 0.07027(15) | 0.17087(15) | 0.46236(13) | 0.0376(3) |

Table 3. Anisotropic displacement parameters for **16**.

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0312(8) | 0.0274(8) | 0.0185(7) | 0.0002(6) | -0.0011(6) | -0.0035(6) |
| C2 | 0.0333(9) | 0.0264(8) | 0.0265(8) | 0.0033(6) | -0.0022(7) | 0.0013(7) |
| C3 | 0.0275(8) | 0.0273(8) | 0.0222(8) | 0.0024(6) | 0.0012(6) | 0.0062(6) |
| C4 | 0.0260(8) | 0.0253(8) | 0.0239(8) | 0.0031(6) | 0.0006(6) | 0.0054(6) |
| C5 | 0.0278(8) | 0.0343(9) | 0.0258(8) | 0.0044(7) | 0.0024(7) | 0.0021(7) |
| C6 | 0.0395(10) | 0.0399(10) | 0.0231(8) | 0.0025(7) | 0.0021(7) | 0.0012(8) |
| C7 | 0.0402(10) | 0.0417(11) | 0.0298(9) | 0.0016(8) | -0.0045(8) | -0.0066(8) |
| C8 | 0.0298(9) | 0.0417(11) | 0.0368(10) | 0.0050(8) | 0.0012(8) | -0.0043(8) |
| C9 | 0.0294(9) | 0.0344(9) | 0.0282(9) | 0.0046(7) | 0.0043(7) | 0.0050(7) |
| C10 | 0.0279(8) | 0.0285(8) | 0.0245(8) | -0.0008(6) | 0.0036(6) | 0.0056(6) |
| C11 | 0.0308(8) | 0.0266(8) | 0.0239(8) | 0.0030(6) | 0.0041(6) | 0.0069(6) |
| C12 | 0.0259(8) | 0.0248(8) | 0.0222(8) | 0.0020(6) | 0.0025(6) | 0.0016(6) |
| C13 | 0.0270(8) | 0.0277(8) | 0.0194(7) | 0.0011(6) | 0.0034(6) | 0.0048(6) |
| C14 | 0.0279(8) | 0.0404(10) | 0.0218(8) | 0.0081(7) | 0.0052(6) | 0.0094(7) |
| C15 | 0.0293(9) | 0.0466(11) | 0.0275(9) | 0.0063(8) | 0.0052(7) | 0.0020(8) |
| C16 | 0.0297(9) | 0.0582(13) | 0.0337(10) | 0.0168(9) | 0.0034(8) | 0.0010(9) |
| C17 | 0.0325(10) | 0.0796(17) | 0.0347(11) | 0.0267(11) | 0.0144(8) | 0.0176(10) |
| C18 | 0.0479(12) | 0.0601(14) | 0.0320(10) | 0.0165(9) | 0.0178(9) | 0.0236(11) |
| C19 | 0.0446(11) | 0.0465(11) | 0.0273(9) | 0.0113(8) | 0.0114(8) | 0.0195(9) |
| C20 | 0.0259(8) | 0.0302(8) | 0.0218(8) | 0.0025(6) | 0.0027(6) | 0.0051(6) |
| C21 | 0.0446(11) | 0.0327(10) | 0.0288(9) | 0.0002(7) | 0.0001(8) | -0.0019(8) |
| C22 | 0.0518(12) | 0.0424(11) | 0.0259(9) | -0.0036(8) | -0.0041(8) | 0.0019(9) |
| C23 | 0.0445(11) | 0.0453(11) | 0.0214(8) | 0.0045(7) | 0.0037(7) | 0.0088(9) |
| C24 | 0.0630(14) | 0.0460(12) | 0.0295(10) | 0.0115(9) | 0.0031(9) | -0.0107(10) |
| C25 | 0.0525(12) | 0.0408(11) | 0.0254(9) | 0.0045(8) | -0.0009(8) | -0.0114(9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C26 | 0.0439(12) | 0.0538(14) | 0.0598(15) | 0.0146(11) | 0.0078(11) | 0.0000(10) |
| N1 | 0.0248(7) | 0.0278(7) | 0.0209(7) | 0.0029(5) | 0.0055(5) | 0.0032(5) |
| N2 | 0.0232(7) | 0.0296(7) | 0.0189(6) | 0.0031(5) | 0.0038(5) | 0.0051(5) |
| O1 | 0.0335(7) | 0.0374(7) | 0.0244(6) | 0.0021(5) | 0.0057(5) | -0.0072(5) |
| O2 | 0.0354(7) | 0.0460(8) | 0.0342(7) | 0.0049(6) | 0.0124(6) | 0.0141(6) |

Table 4. Bond lengths for **16**.

| | |
|---|---|
| C1-O1 | 1.211 |
| C1-N1 | 1.398(2) |
| C1-C2 | 1.507(3) |
| C2-C3 | 1.532(2) |
| C3-N2 | 1.499(2) |
| C3-C4 | 1.515(2) |
| C4-C9 | 1.394(2) |
| C4-C5 | 1.398(2) |
| C5-C6 | 1.388(3) |
| C6-C7 | 1.390(3) |
| C7-C8 | 1.388(3) |
| C8-C9 | 1.391(3) |
| C10-O2 | 1.210(2) |
| C10-N1 | 1.402(2) |
| C10-C11 | 1.508(2) |
| C11-C12 | 1.547(2) |
| C12-C20 | 1.512(2) |
| C12-C13 | 1.560(2) |
| C13-N2 | 1.498(2) |
| C13-C14 | 1.517(2) |
| C14-C15 | 1.383(3) |
| C14-C19 | 1.395(3) |
| C15-C16 | 1.402(3) |
| C16-C17 | 1.392(3) |
| C16-C26 | 1.461(3) |
| C17-C18 | 1.365(4) |
| C18-C19 | 1.398(3) |
| C20-C25 | 1.384(3) |
| C20-C21 | 1.389(3) |
| C21-C22 | 1.392(3) |
| C22-C23 | 1.379(3) |
| C23-C24 | 1.375(3) |
| C24-C25 | 1.391(3) |
| N1-N2 | 1.4339(19) |

Table 5. Bond angles for **16**.

| | |
|---|---|
| O1-C1-N1 | 125.47(16) |
| O1-C1-C2 | 105.95(14) |

| | |
|---|---|
| C1-C2-C3 | 103.46(14) |
| N2-C3-C4 | 110.55(13) |
| N2-C3-C2 | 104.22(13) |
| C4-C3-C2 | 114.26(14) |
| C9-C4-C5 | 118.69(16) |
| C9-C4-C3 | 119.21(15) |
| C5-C4-C3 | 122.10(16) |
| C6-C5-C4 | 120.72(17) |
| C5 C6 C7 | 119.92(18) |
| C8-C7-C6 | 119.97(18) |
| C7-C8-C9 | 119.91(18) |
| C8-C9-C4 | 120.75(17) |
| O2-C10-N1 | 122.27(16) |
| O2-C10-C11 | 126.74(16) |
| N1-C10-C11 | 110.70(14) |
| C10-C11-C12 | 106.41(14) |
| C20-C12-C11 | 113.62(14) |
| C20-C12-C13 | 115.47(14) |
| C11-C12-C13 | 109.98(13) |
| N2-C13- C14 | 105.82(13) |
| N2-C13-C12 | 107.38(13) |
| C14-C13-C12 | 117.16(14) |
| C15-C14-C19 | 119.15(18) |
| C15-C14-C13 | 121.30(16) |
| C19-C14-C13 | 119.48(18) |
| C14-C15-C16 | 121.03(19) |
| C17-C16-C15 | 118.5(2) |
| C17-C16-C26 | 120.7(2) |
| C15-C16-C26 | 120.7(2) |
| C18-C17-C16 | 121.35(19) |
| C17-C18-C19 | 119.9(2) |
| C14-C19-C18 | 120.1(2) |
| C25-C20-C21 | 117.99(17) |
| C25-C20-C12 | 123.09(16) |
| C21-C20-C12 | 118.89(16) |
| C20-C21-C22 | 121.32(19) |
| C23-C22-C21 | 120.00(19) |
| C24-C23-C22 | 119.12(18) |
| C23-C24-C25 | 121.0(2) |
| C20-C25-C24 | 120.58(19) |
| C1-N1-C10 | 128.99(15) |
| C1-N1-N2 | 113.68(13) |
| C10-N1-N2 | 116.01(13) |
| N1-N2-C13 | 111.84(12) |
| N1-N2-C3 | 103.17(12) |
| C13-N2-C3 | 112.51(13) |

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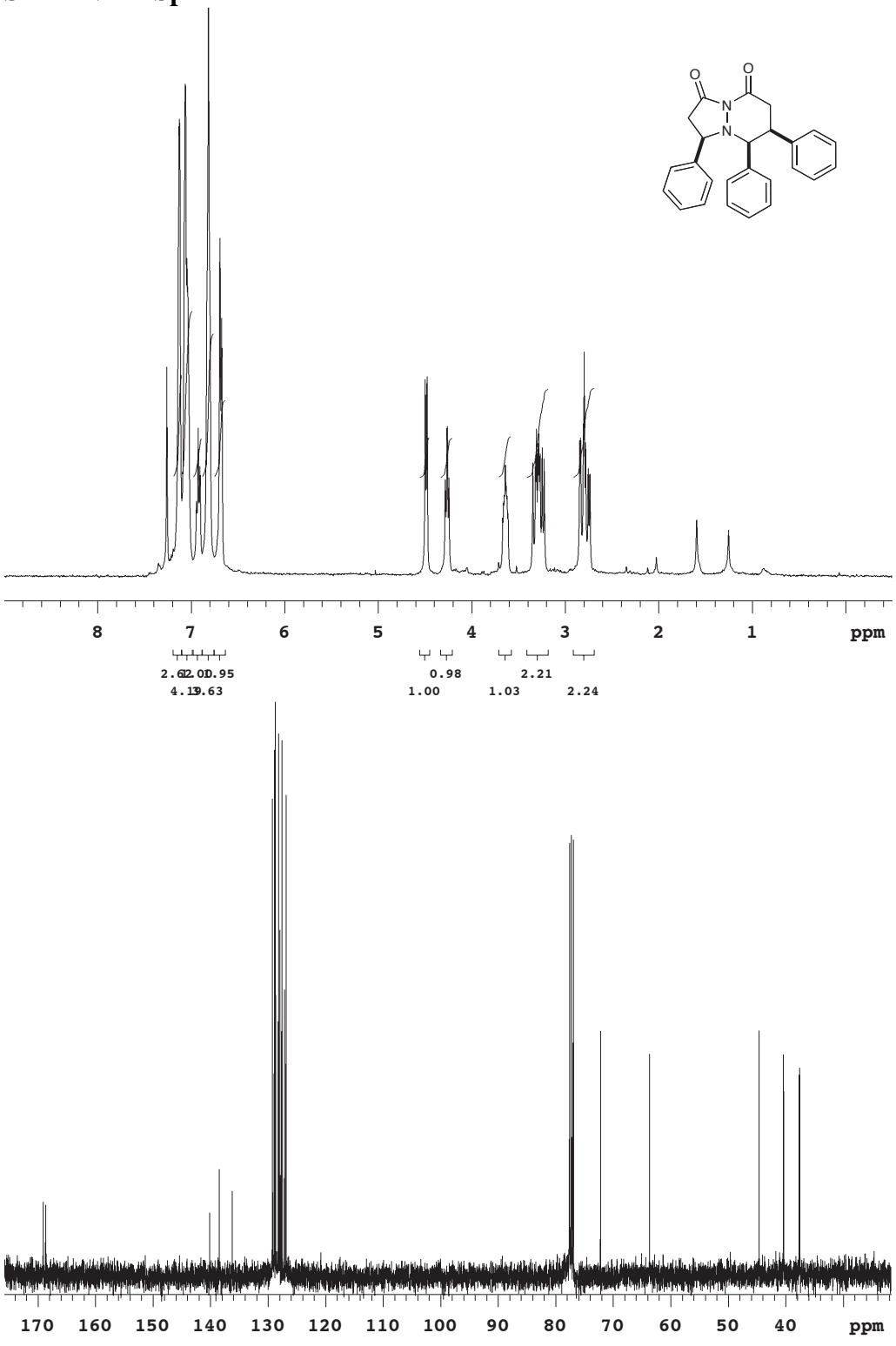

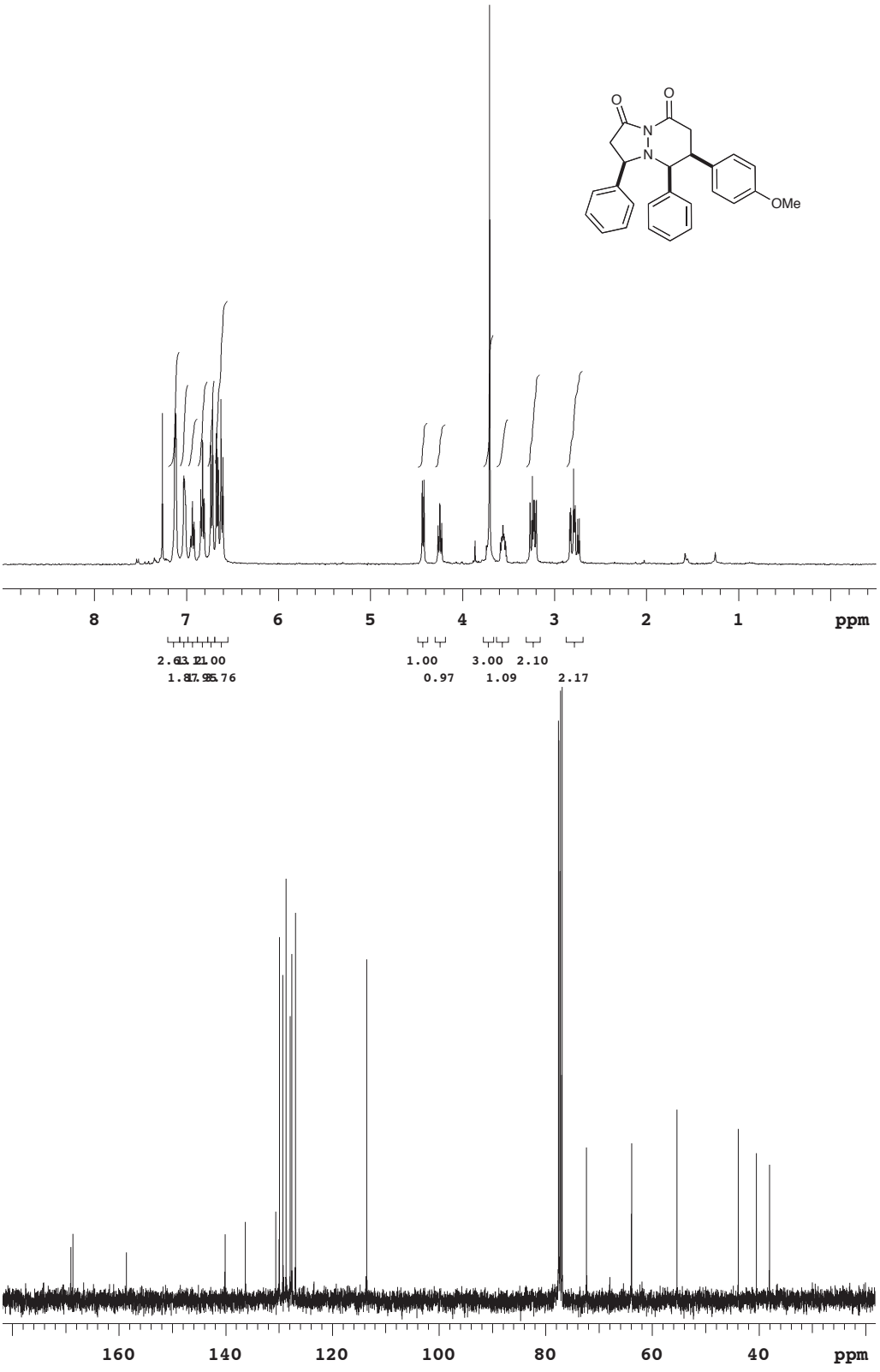
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3.00
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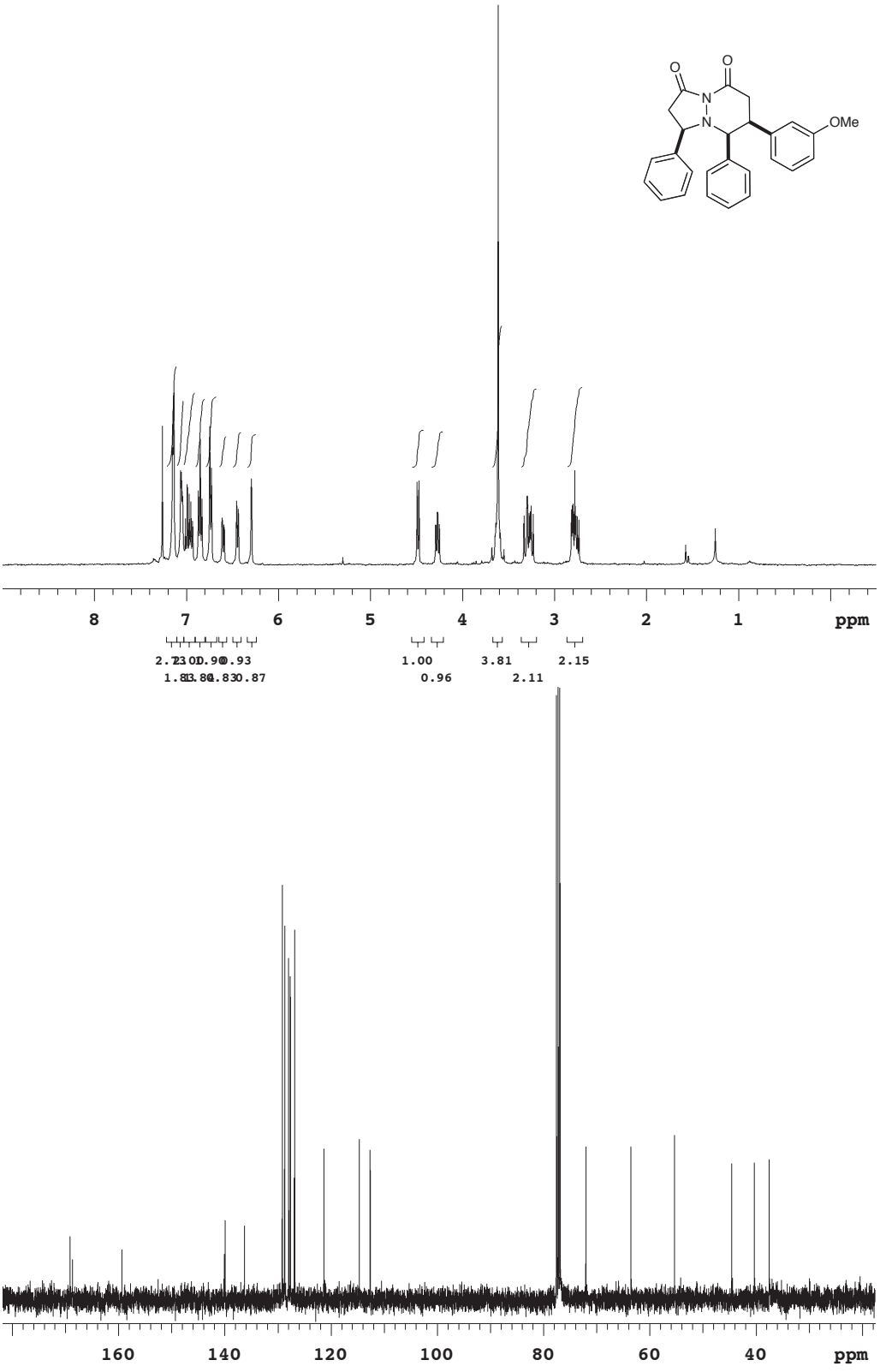
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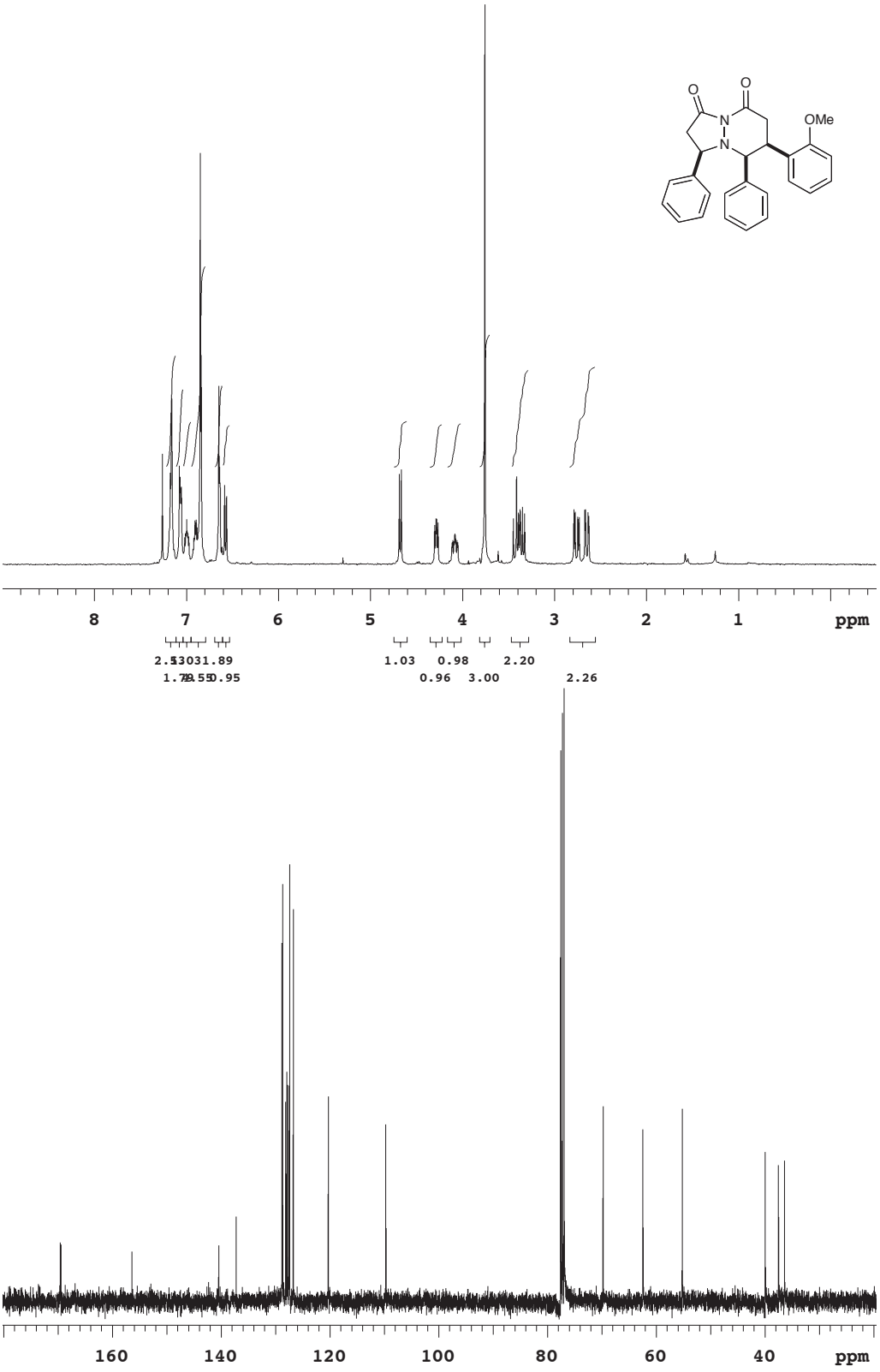
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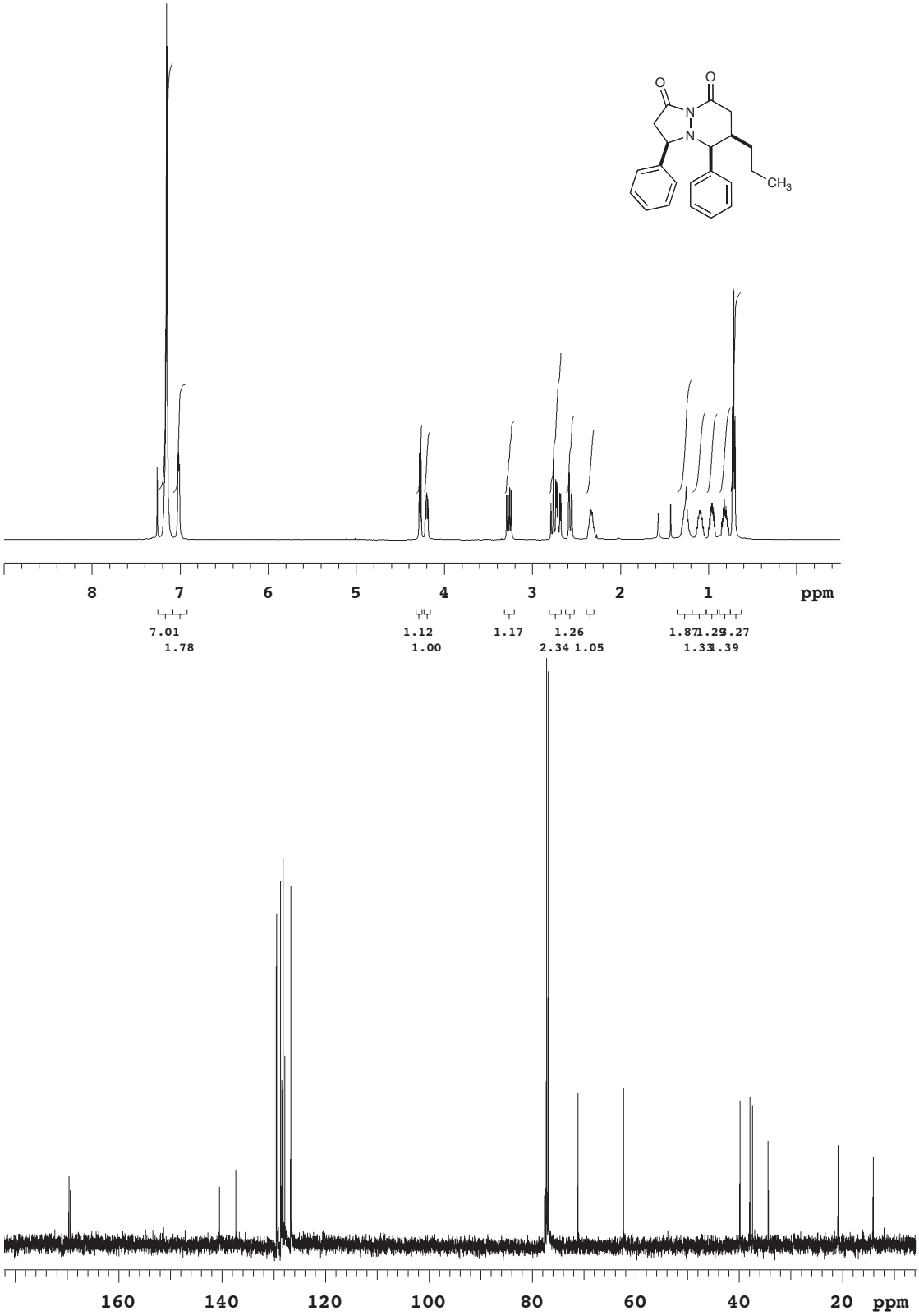
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1.00
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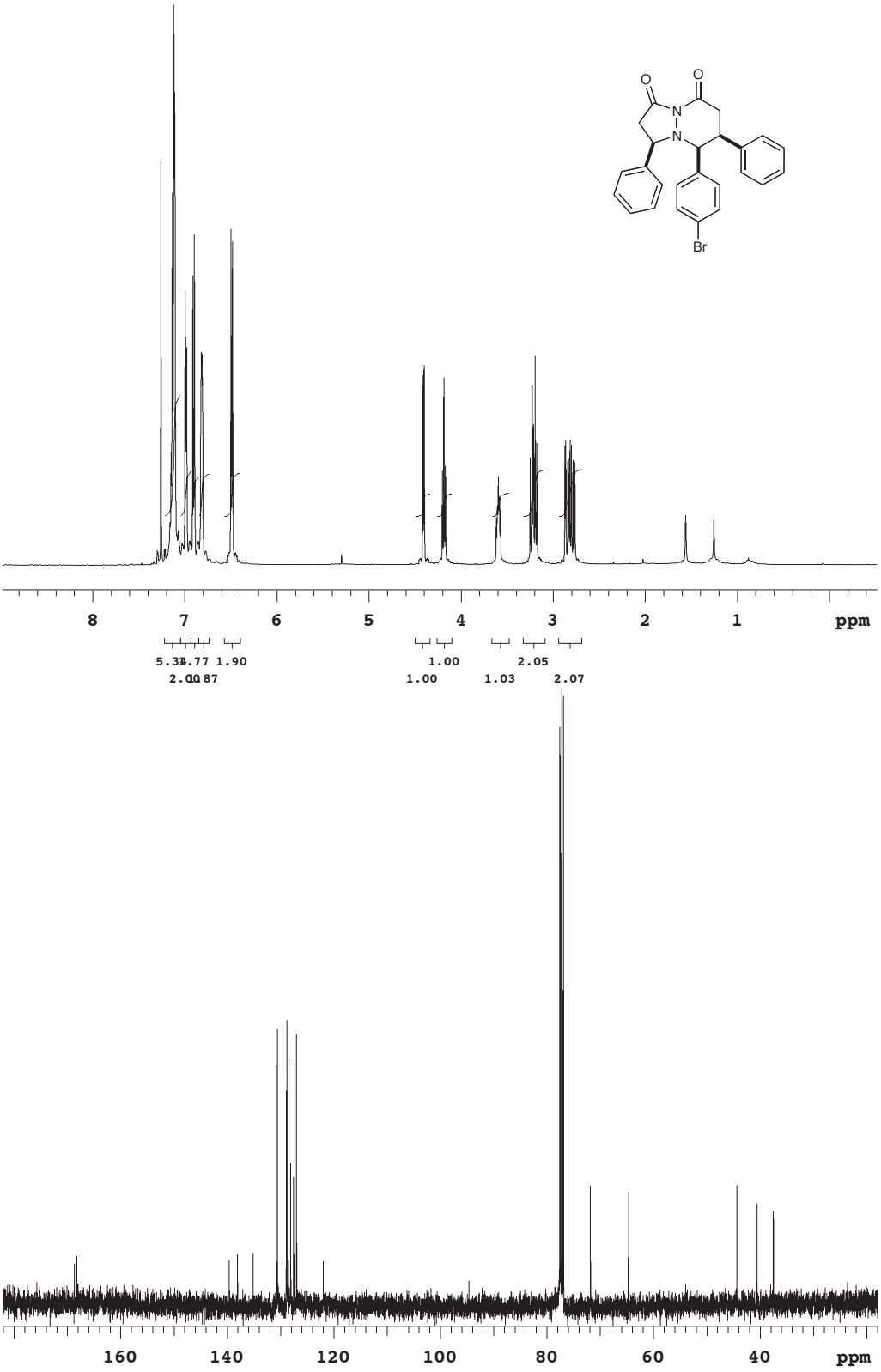
40
20
ppm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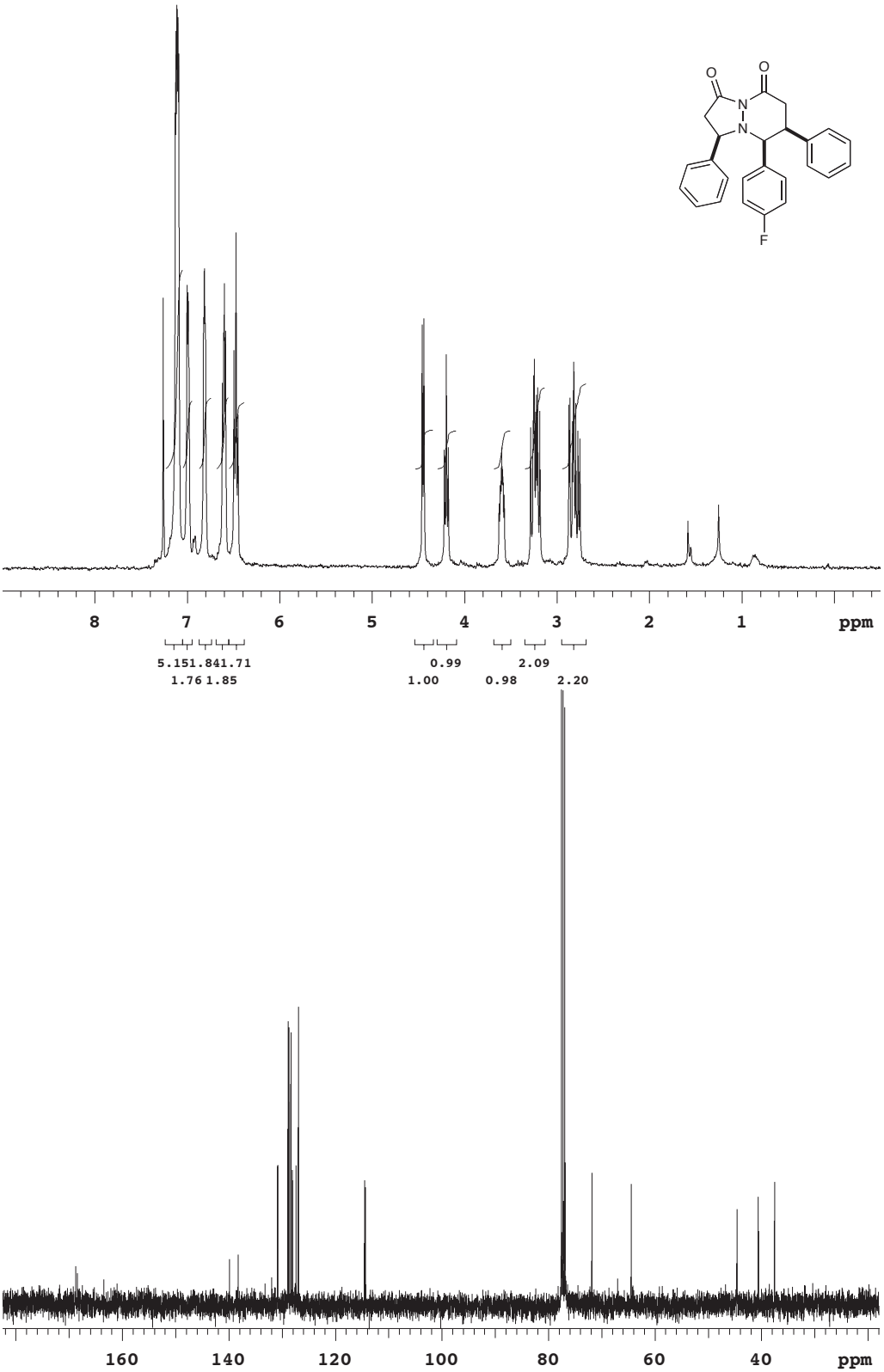
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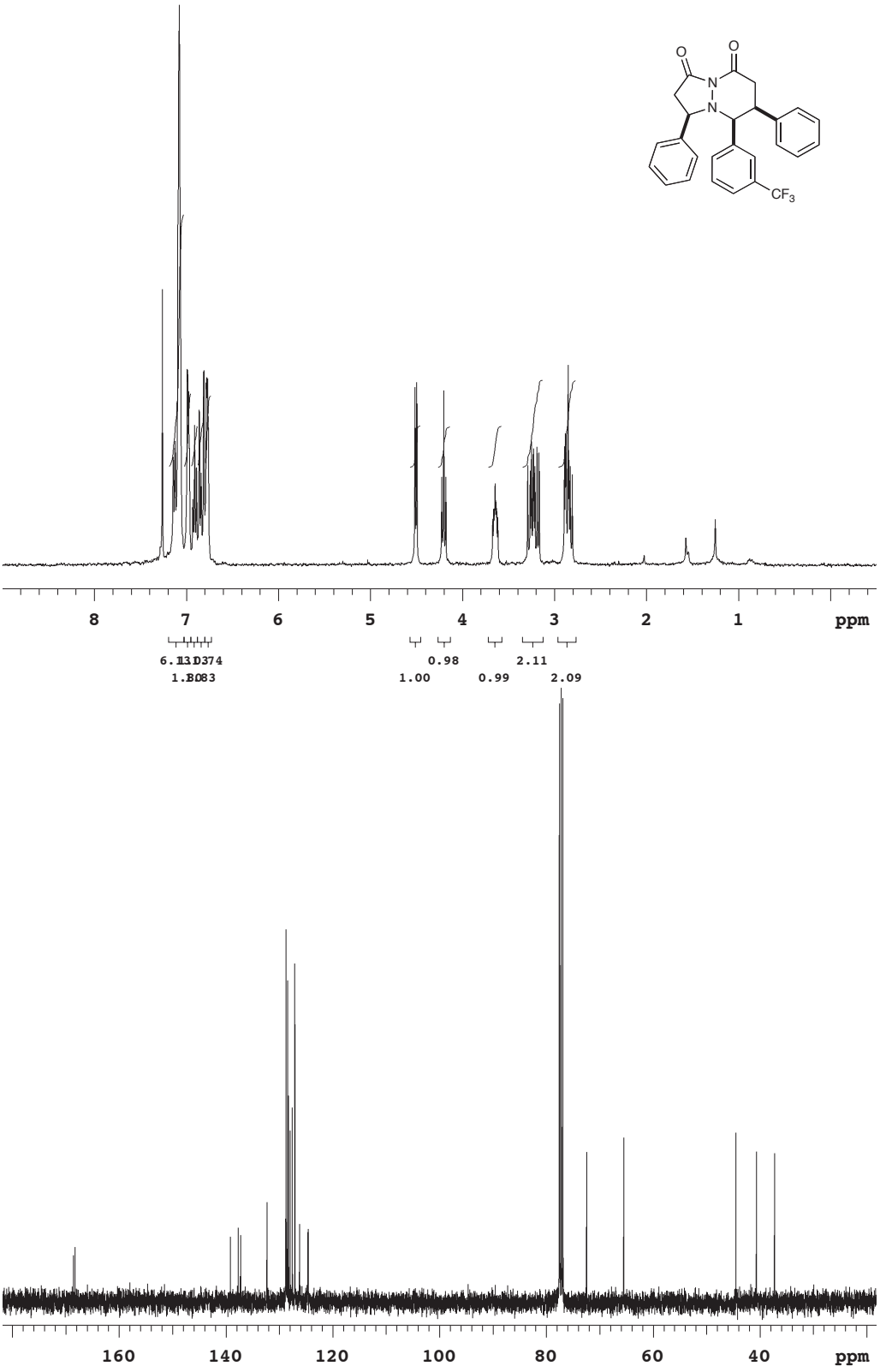
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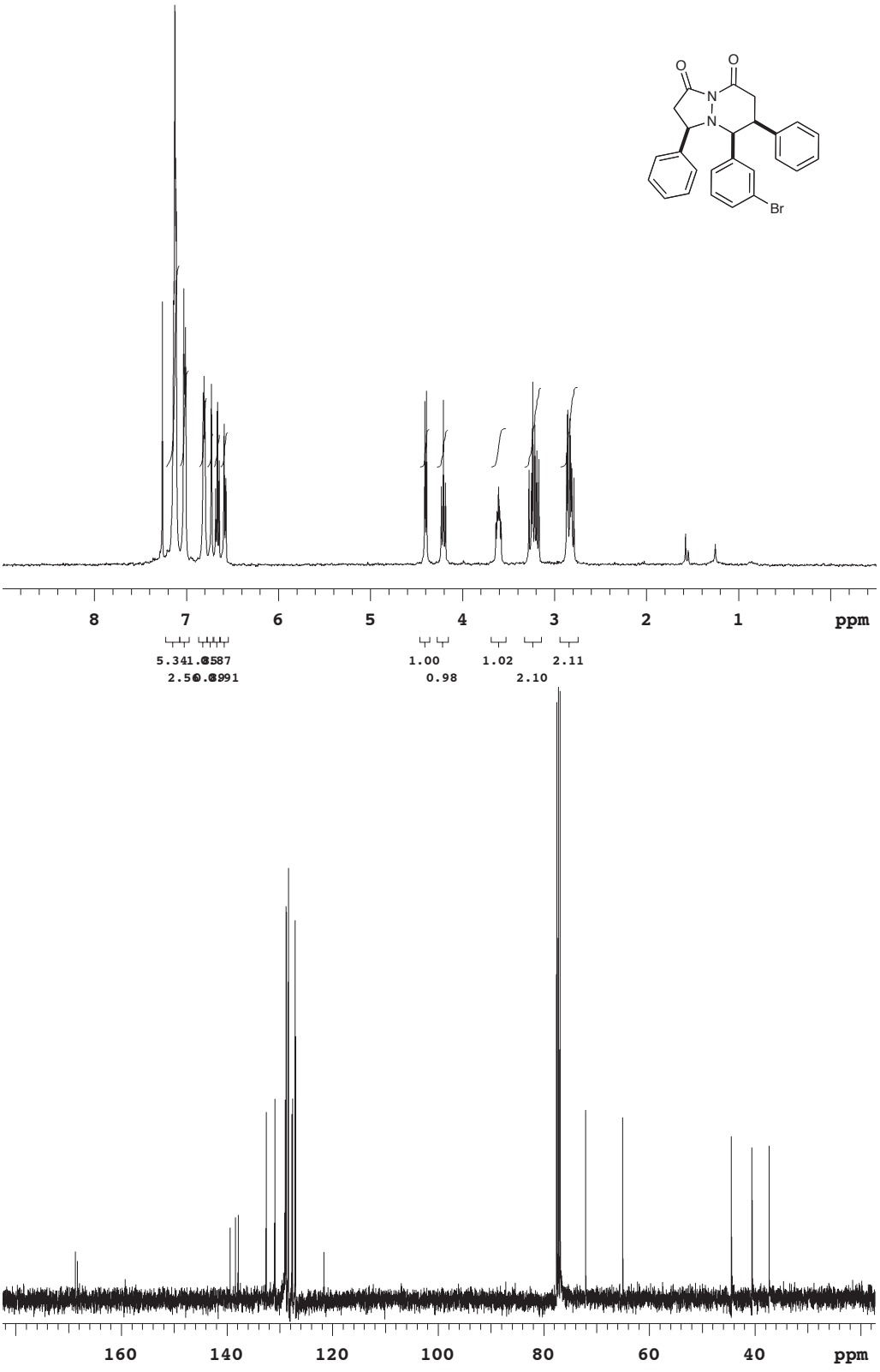
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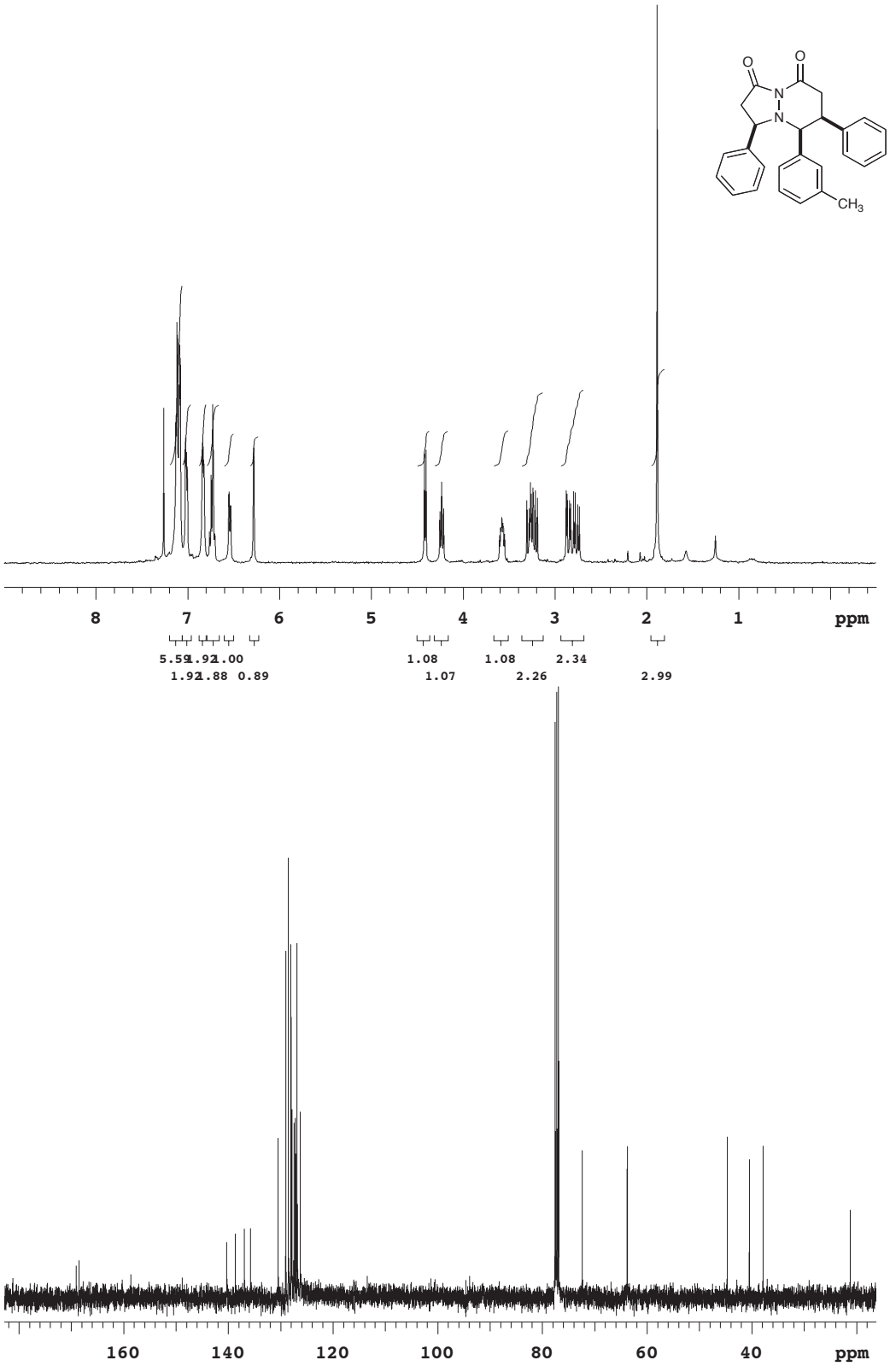
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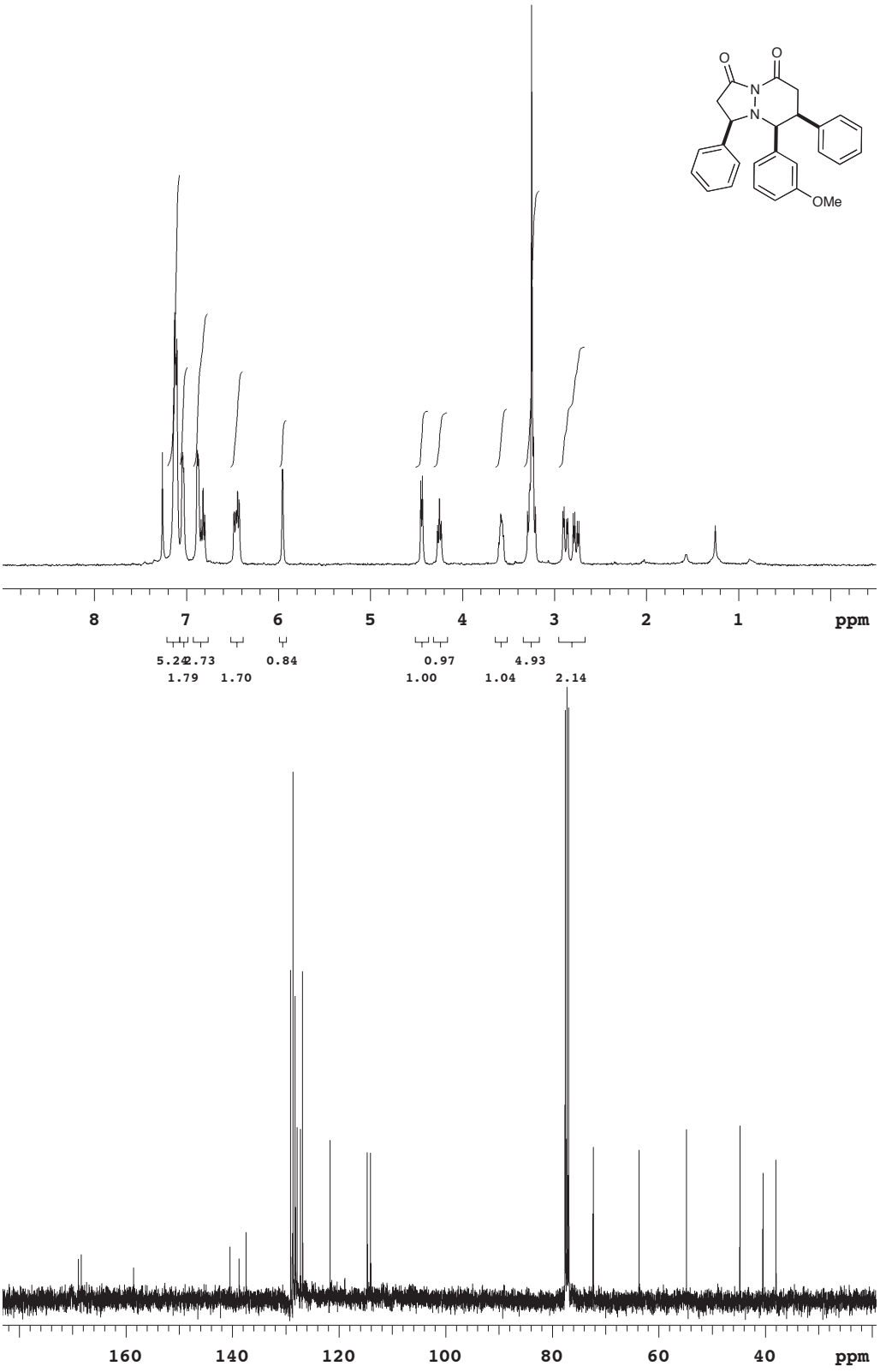
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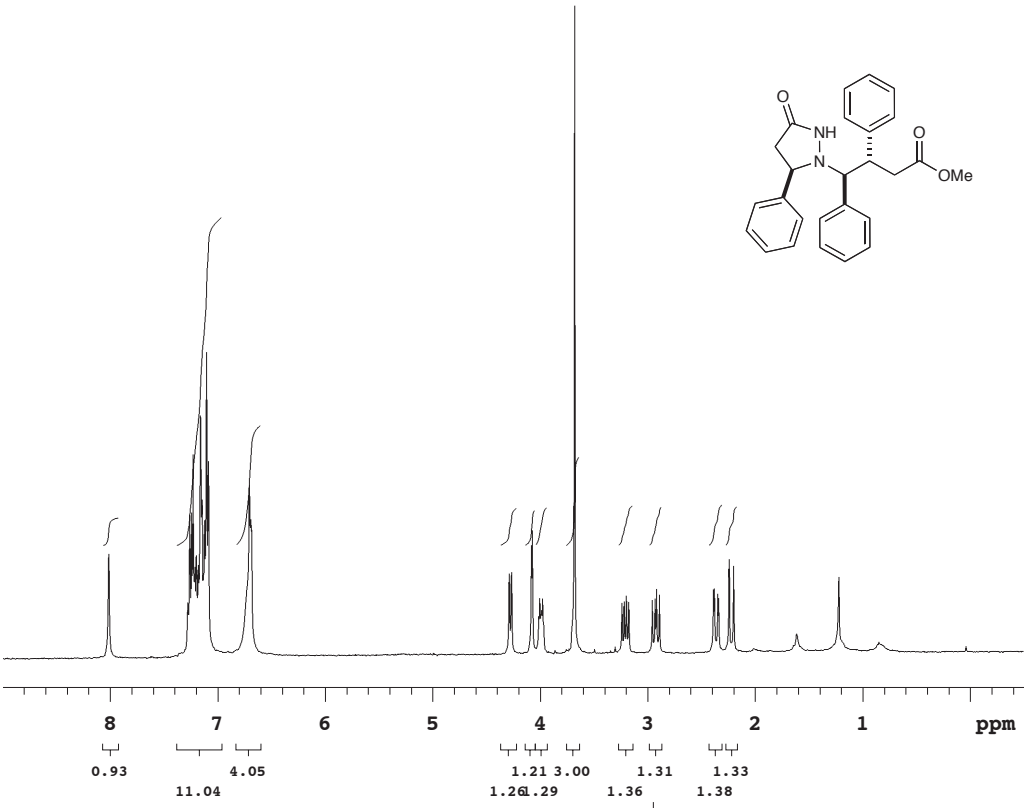
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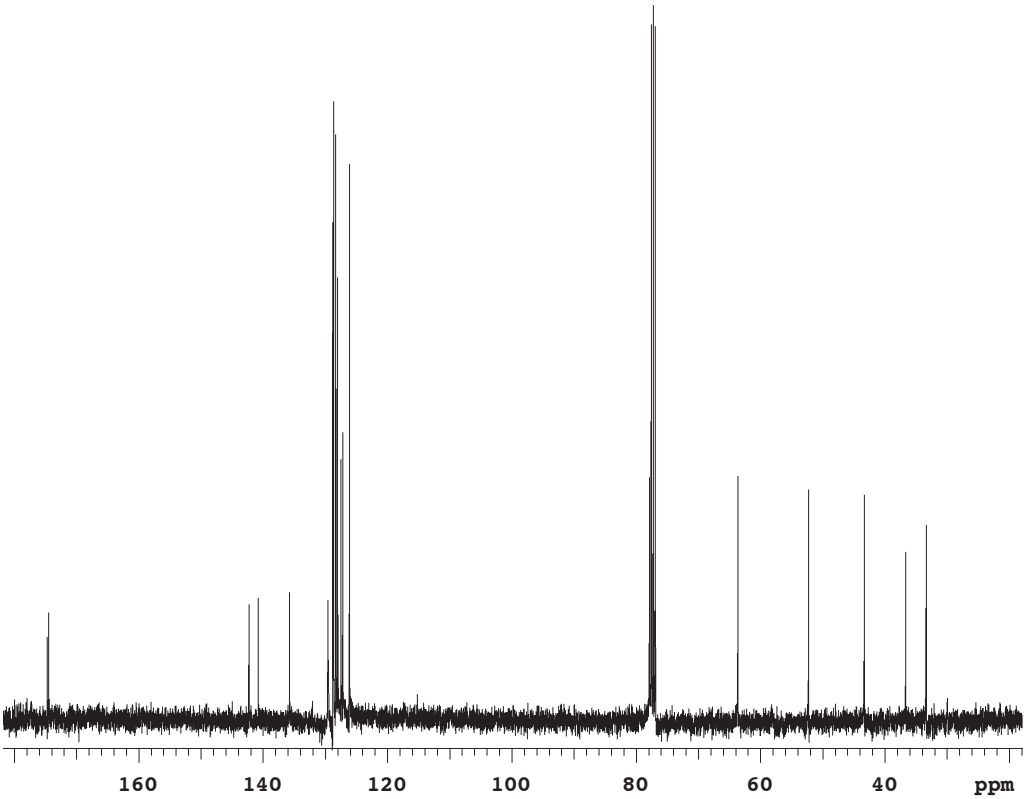
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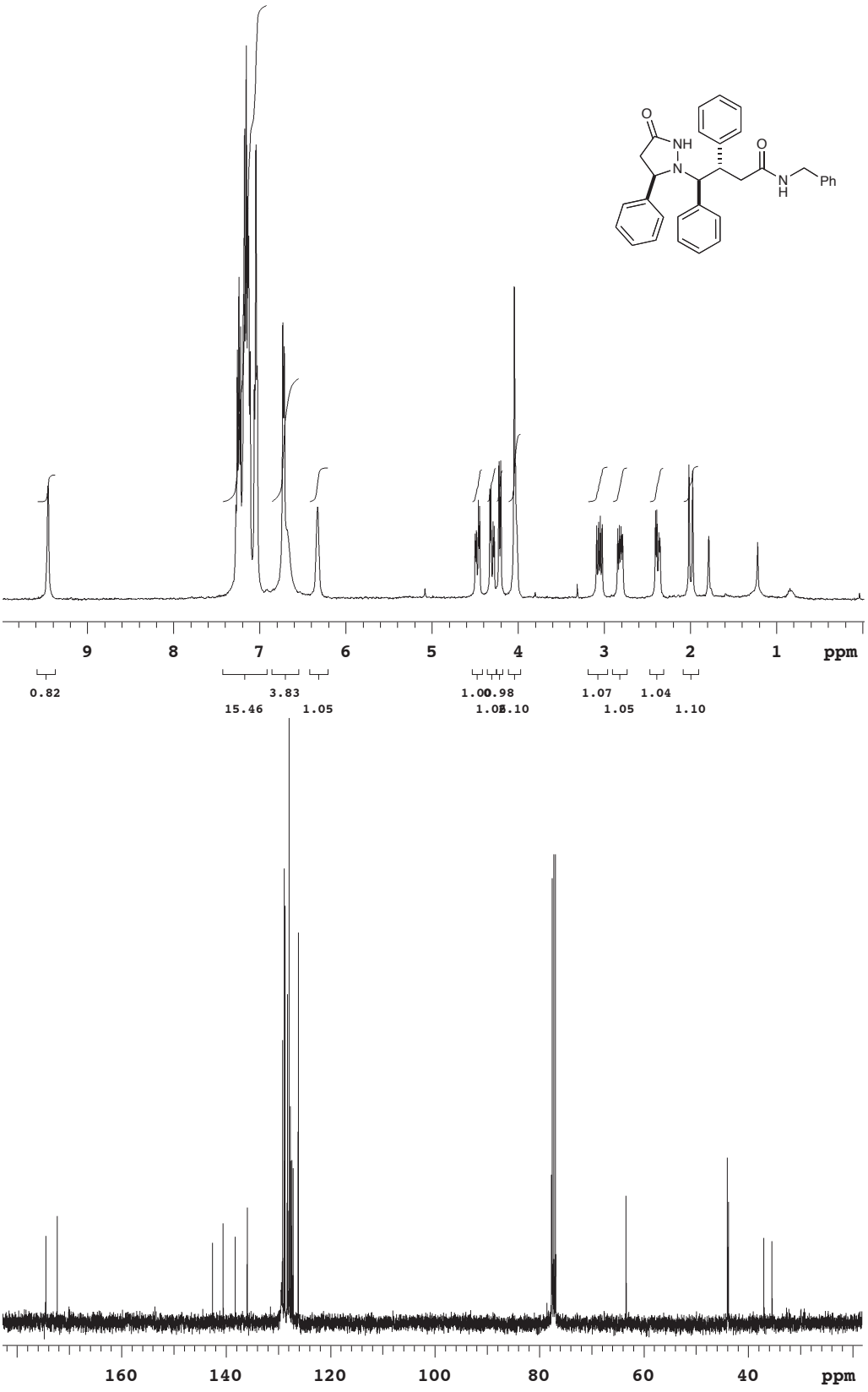
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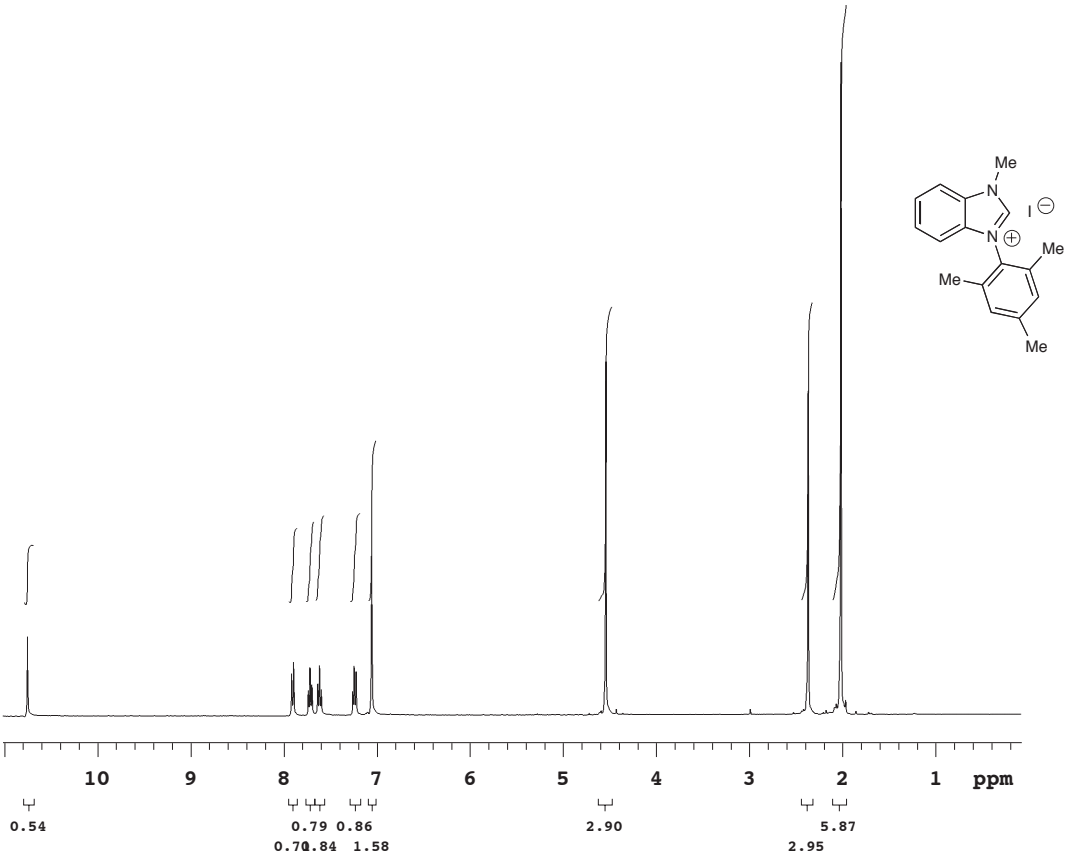
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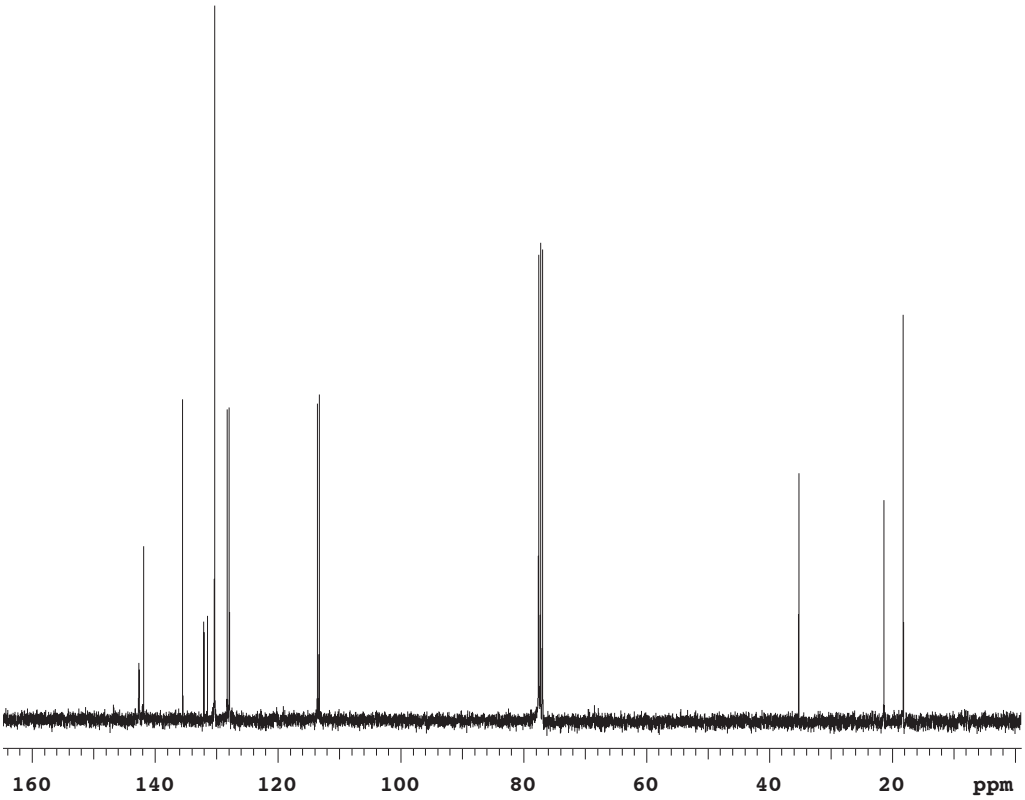
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