Electronic Supplementary Material (ESI) for ChemComm.

Concise Synthesis of Cyclic Carbonyl Compounds from Haloarenes Using Phenyl Formate as the Carbonyl Source

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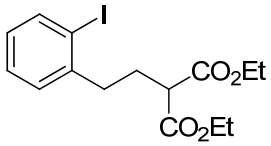
1. General method and materials

General. All reactions were performed in oven-dried or flame-dried glassware under argon atmosphere. Reactions were monitored by TLC on Merck silica gel 60 F254 plates visualized by UV lump at 254 nm. Column chromatography was performed on Merck silica gel 60 and preparative TLC was performed on Merck silica gel 60 F254 0.5 mm plates. NMR spectra were measured on a JEOL AL-400 NMR spectrometer (400 MHz for ^{1}H spectra and 100 MHz for ^{13}C spectra) or a JEOL ECX-500 NMR spectrometer (500 MHz for ^{1}H spectra and 125 MHz for ^{13}C spectra). For ^{1}H NMR, tetramethylsilane (TMS) ($\delta = 0$) in $CDCl_3$ served as an internal standard. For ^{13}C NMR, $CDCl_3$ ($\delta = 77.0$) served as an internal standard. Infrared spectra were measured on a SHIMADZU IR Prestige-21 spectrometer (ATR). High-resolution mass spectra (HRMS) were measured on a Bruker MicrOTOF time-of-flight mass spectrometer (ESI). Melting point was measured using a YAZAWA MICRO MELTING POINT BY-1.

Materials. $Pd(OAc)_2$, all ligands, **15a-b**, and **17** were purchased from TCI, Wako, Kanto, and Aldrich, and used as received. Triethylamine, 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), and all solvents except DMSO were purified by distillation prior to use. Anhydrous DMSO ("Super Dehydrated" grade) was purchased from Wako and used as received. Compounds **1a-b**,[1] **2**,[2] **7**,[3] **9**,[4] **13**,[5] **19** and **21**,[6] and **23**[7] were synthesized according to known methods, whose analytical data was identical to those reported in precedent literature.

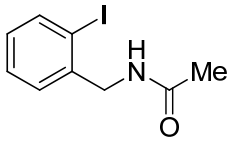
2. Preparation of substrates



Diethyl 2-(2-iodophenethyl)malonate (5)

5 was synthesized by similar method to that of **1a** using 1-(2-bromoethyl)-2-iodobenzene and diethyl malonate. Yield: 49%

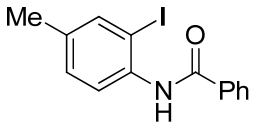
Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.49-7.21 (m, 2H), 6.89 (t, *J* = 7.6 Hz, 1H), 4.28-4.19 (m, 4H), 3.39 (t, *J* = 7.6 Hz, 1H), 2.78 (t, *J* = 8.0 Hz, 2H), 2.23-2.16 (m, 2H), 1.29 (t, *J* = 7.2 Hz, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.0, 143.3, 139.5, 129.5, 128.3, 128.0, 100.3, 61.3, 51.2, 38.1, 28.8, 14.0 ppm; IR (ATR) 1726, 1465, 1367, 1147, 1010, 750cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{15}H_{19}INaO_4$: 413.0220; found 413.0220.



Diethyl 2-(2-iodophenethyl)malonate (11)

A solution of (2-iodophenyl)methanamine[8] (1.86 g, 8.00 mmol) in pyridine (15 mL) was cooled to 0 °C. Acetic anhydride (756 μL, 8.00 mmol, 1.00 equiv) was added to the solution, and the mixture was stirred at rt for 10 h. After the reaction mixture was concentrated under reduced pressure, it was diluted with AcOEt and H_2O. The aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was recrystallized from hexane/CH_2Cl_2 to afford **11** (1.03 g, 3.74 mmol, 47%) as colorless needles.

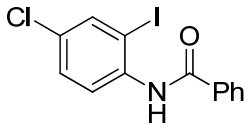
M.p. 130–132 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.35-7.28 (m, 2H), 6.89 (t, *J* = 7.2 Hz, 1H), 6.30 (br s, 1H), 4.41 (d, *J* = 6.0 Hz, 2H), 2.00 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 140.3, 139.3, 129.5, 129.2, 128.5, 98.9, 48.2, 23.1 ppm; IR (ATR) 1616, 1556, 1435, 1373, 1296, 1016, 748 cm^{-1}; HRMS (ESI) $[M+H]^+$ calcd for $C_9H_{11}IO$: 275.9880; found 275.9881.



***N*-(2-Iodo-4-methylphenyl)benzamide (25)**

To a solution of 2-iodo-4-methylaniline (1.17 g, 5.00 mmol) in Et_2O (30 mL) at 0 °C was added triethylamine (730 μL, 5.25 mmol, 1.05 equiv) and benzoyl chloride (610 μL, 5.25 mmol, 1.05 equiv), and the reaction mixture was stirred at rt for 12 h. The reaction mixture was diluted with AcOEt and H_2O. The aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was recrystallized from MeOH to afford **25** (1.46 g, 4.32 mmol, 86%) as colorless needles.

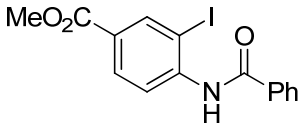
M.p. 160–161 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.4 Hz, 1H), 8.20 (br s, 1H), 7.98-7.95 (m, 2H), 7.66 (s, 1H), 7.61-7.49 (m, 3H), 7.21 (d, *J* = 8.0 Hz, 1H), 2.31 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 138.9, 136.0, 135.7, 134.5, 132.0, 129.9, 128.8, 127.1, 121.7, 90.5, 20.3 ppm; IR (ATR) 1645, 1485, 1298, 1026, 817cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{14}H_{12}INNaO$: 359.9856; found 359.9856.



***N*-(4-Chloro-2-iodophenyl)benzamide (27)**

27 was synthesized by similar method to that of **25** using 4-chloro-2-iodoaniline. Yield: 85%.

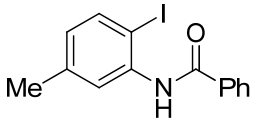
Pale yellow needles; M.p. 138–140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 9.2 Hz, 1H), 8.26 (br s, 1H), 7.98-7.94 (m, 2H), 7.81 (d, *J* = 2.4 Hz, 1H), 7.63-7.51 (m, 3H), 7.39 (dd, *J* = 9.6, 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 137.9, 137.1, 134.3, 132.4, 129.9, 129.5, 129.0, 127.2, 122.0, 89.8 ppm; IR (ATR) 1643, 1510, 1465, 1298, 1097, 815 cm^{-1}; HRMS (ESI) $[M+H]^+$ calcd for $C_{13}H_9ClINO$: 379.9310; found 379.9309.



Methyl 4-benzamido-3-iodobenzoate (29)

29 was synthesized by similar method to that of **25** using methyl 4-amino-3-iodobenzoate. Yield: 50%.

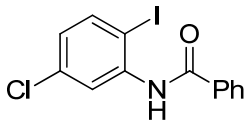
Colorless needles; M.p. 140–141 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.8 Hz, 1H), 8.53 (br s, 1H), 8.51 (d, *J* = 2.0 Hz, 1H), 8.08 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.00-7.97 (m, 2H), 7.65-7.52 (m, 3H), 3.93 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 165.1, 142.1, 140.1, 134.0, 132.5, 130.9, 129.0, 127.2, 127.0, 120.0, 88.7, 52.3 ppm; IR (ATR) 1714, 1651, 1517, 1384, 1269, 1226, 1112, 763cm^{-1}; HRMS (ESI) $[M+H]^+$ calcd for $C_{15}H_{13}INO_3$: 381.9935; found 381.9934.



***N*-(2-Iodo-5-methylphenyl)benzamide (31)**

31 was synthesized by similar method to that of **27** using 2-iodo-5-methylaniline. Yield: 76%.

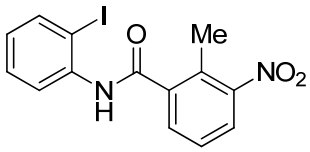
White solid; M.p. 132–133 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 8.25 (br s, 1H), 7.97 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.61-7.50 (m, 3H), 6.73 (d, *J* = 8.4 Hz, 1H), 2.37 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 139.6, 138.2, 137.8, 134.4, 132.0, 128.8, 127.0, 122.5, 86.3, 21.2 ppm (one carbon signal is missing); IR (ATR) 1645, 1516, 1463, 1300, 1018, 798 cm^{-1}; HRMS (ESI) $[M+H]^+$ calcd for $C_{14}H_{13}INO$: 338.0036; found 338.0037.



***N*-(5-Chloro-2-iodophenyl)benzamide (33)**

33 was synthesized by similar method to that of **27** using 5-chloro-2-iodoaniline. Yield: 46%.

Colorless needles; M.p. 142–143 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 2.4 Hz, 1H), 8.30 (br s, 1H), 8.01-7.95 (m, 2H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.63-7.51 (m, 3H), 6.89 (dd, *J* = 8.4, 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 139.1, 135.5, 134.0, 132.4, 128.9, 127.1, 125.9, 121.4, 86.9 ppm (one carbon signal is missing); IR (ATR) 1649, 1564, 1514, 1398, 1290, 1016, 798 cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{13}H_9ClINNaO$: 379.9310; found 379.9308.



***N*-(2-Iodophenyl)-2-methyl-3-nitrobenzamide (35)**

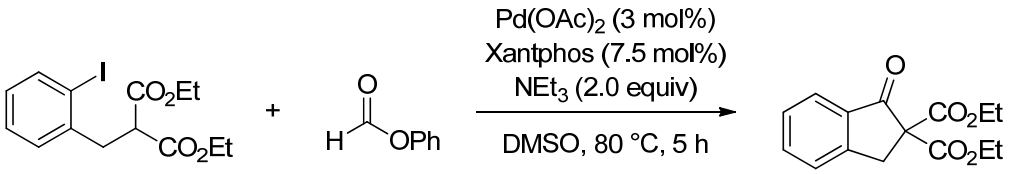
A solution of 2-methyl-3-nitrobenzoic acid (1.81 g, 10.0 mmol) in thionyl chloride (15 mL) was stirred at 60 °C for 25.5 h. Evaporation of the reaction mixture under reduced pressure afforded 2-methyl-3-nitrobenzoyl chloride (1.98 g, 9.92 mmol, 99%).

To a solution of 2-methyl-3-nitrobenzoyl chloride (1.98 g, 9.92 mmol) in Et_2O (40 mL) was added 2-iodoaniline (1.97 g, 9.00 mmol, 0.91 equiv) and triethylamine (1.39 mL, 10.0 mmol, 1.01 equiv) at 0 °C and the reaction mixture was stirred at rt for 24 h. The reaction mixture was diluted with AcOEt and H_2O. The aqueous layer was extracted with AcOEt. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was recrystallized from MeOH to afford **35** (3.02 g, 7.90 mmol, 88%) as colorless needles.

M.p. 166–168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.6 Hz, 1H), 7.93 (d, *J* = 7.6 Hz, 1H), 7.85-7.75 (m, 3H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 6.95 (t, *J* = 8.0 Hz, 1H), 2.65 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 151.2, 139.4, 139.0, 137.7, 130.9, 130.5, 129.4, 127.1, 127.0, 125.8, 122.5, 90.7, 16.2 ppm; IR (ATR) 1647, 1519, 1348, 1305, 1018, 748 cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{14}H_{11}IN_2NaO_3$: 404.9707; found 404.9706.

3. Experimental procedures

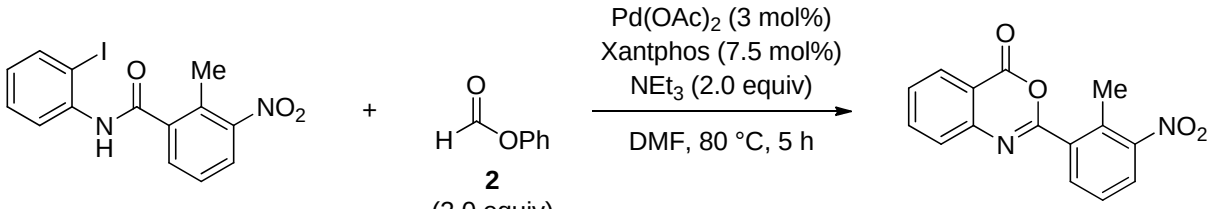
General experimental procedure of cyclocarbonylation (Table 3 and 4).



1a **2** (2.0 equiv) **3**

$Pd(OAc)_2$ (2.0 mg, 9.00 μmmol, 3 mol%) and Xantphos (13.0 mg, 22.5 μmmol, 7.5 mol%) were added in a screw-capped 10-mL test tube containing a magnetic stirring bar. The tube was evacuated and backfilled with Ar three times. DMSO (1.0 mL), **1a** (113 mg, 0.300 mmol), **2** (65.4 μL, 0.600 mmol, 2.0 equiv), and NEt_3 (83.3 μL, 0.600 mmol, 2.0 equiv) were added to the tube and the tube was equipped with screw cap. The tube was warmed to 80 °C in an oil bath and stirred for 5 h. The reaction mixture was cooled to rt and was diluted with EtOAc, washed with H_2O three times, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was purified by preparative TLC (hexane/EtOAc 5/1) to afford **3** (69.6 mg, 0.252 mmol, 84%) as a colorless oil.

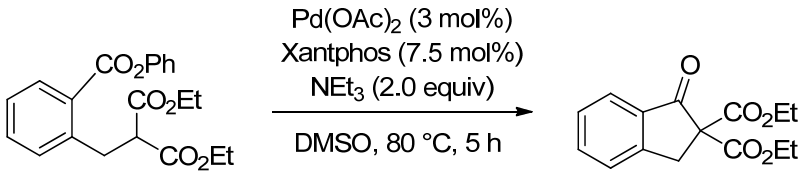
Large-scale synthesis of compound 36.



35 **2** (2.0 equiv) **36**

$Pd(OAc)_2$ (60.6 mg, 0.270 mmol, 3 mol%) and Xantphos (391 mg, 0.675 mmol, 7.5 mol%) were added in 100-mL round-bottomed flask containing a magnetic stirring bar. The tube was evacuated and backfilled with Ar three times. DMF (30 mL), **35** (3.44 g, 9.00 mmol), **2** (1.96 mL, 18.0 mmol, 2.0 equiv), and NEt_3 (2.50 mL, 18.0 mmol, 2.0 equiv) were added to the flask and the tube was equipped with an Ar balloon through a septum cap. The flask was warmed to 80 °C in an oil bath and stirred for 5 h. The reaction mixture was cooled to rt and was diluted with CH_2Cl_2, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was triturated with MeOH to afford **36** (1.77 g, 6.26 mmol, 70%) as a pale yellow solid.

Conversion of 4 to 3.



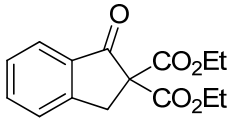
4 **3**

$Pd(OAc)_2$ (1.30 mg, 0.00582 mmol, 3 mol%), Xantphos (8.42 mg, 0.0146 mmol, 7.5 mol%), and **4** (71.9 mg, 0.194 mmol) were added in a screw-capped 10-mL test tube containing a magnetic stirring bar. The tube was evacuated and backfilled with Ar three times. DMSO (0.633 mL) and NEt_3 (53.9 μL, 0.388 mmol, 2.0 equiv) were added to the tube and the tube was equipped with screw cap. The tube was warmed to 80 °C in an oil bath and stirred for 5 h. The reaction mixture was cooled to rt and was diluted with EtOAc, washed with H_2O three times, dried over Na_2SO_4, filtered, and concentrated. The obtained residue was purified by preparative TLC (hexane/EtOAc 5/1) to afford **3** (48.0 mg, 0.174 mmol, 90%) as a colorless oil.

In another batch, conversion of **4** to **3** without using $Pd(OAc)_2$ and Xantphos was conducted with similar reaction procedures. Yield : 90%.

5. Analytical data of products

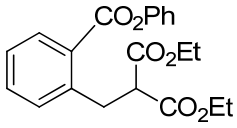
Analytical data of **3**,[9] **8**,[10] **10**,[11] **12**,[12] **14**,[13] **16**,[14] **20**,[15] **22**,[16] **24**,[17] **26**,[18] **28** and **34**,[19] **30**,[20] and **32**[21] were identical to those reported in literature. Yield of **18**[21] was determined by ^{1}H NMR analysis of crude reaction mixture using mesitylene as an internal standard.



Diethyl 1-oxo-1*H*-indene-2,2(3*H*)-dicarboxylate (3)[9]

3 was obtained from **1a** or **1b** as a colorless oil. Yield: 84% from **1a**, 93% from **1b**.

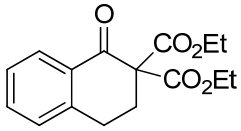
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.0 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 4.32-4.20 (m, 4H), 3.82 (s, 2H), 1.29 (t, *J* = 7.2 Hz, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.4., 166.9, 151.8, 135.6, 134.3, 128.0, 126.2, 125.2, 67.2, 62.5, 36.1, 13.9 ppm.



Diethyl 2-(2-(phenoxycarbonyl)benzyl)malonate (4)

4 was obtained from **1a** as a colorless oil. Yield: 38% (table 1, entry 2).

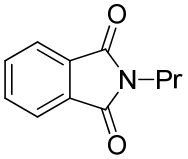
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 7.6 Hz, 1H), 7.52-7.21 (m, 8H), 4.17-4.06 (m, 4H), 3.94 (t, *J* = 8.4 Hz, 1H), 3.61 (d, *J* = 8.0 Hz, 2H), 1.16 (t, *J* = 7.2 Hz, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 165.4, 150.8, 140.6, 132.8, 132.4, 131.7, 129.5, 128.6, 127.2, 126.0, 121.8, 61.3, 52.9, 33.7, 13.9 ppm; IR (ATR) 1728, 1242, 1188, 1161, 1041, 746 cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{21}H_{22}NaO_6$: 393.1309; found 393.1308.



Diethyl 1-oxo-3,4-dihydronaphthalene-2,2(1*H*)-dicarboxylate (6)

6 was obtained from **5** as a colorless oil. Yield: 54%.

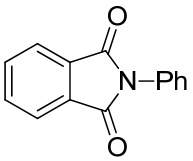
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.0 Hz, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 1H), 4.27 (q, *J* = 7.5 Hz, 4H), 2.98 (d, *J* = 6.5 Hz, 2H), 2.76 (t, *J* = 6.5 Hz, 2H), 1.26 (t, *J* = 7.0 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.2, 167.5, 142.5, 133.8, 131.3, 128.7, 128.0, 126.9, 66.7, 62.1, 29.9, 25.6, 13.9 ppm; IR (ATR) 1726, 1687, 1298, 1259, 1236, 1219, 1176, 1066, 918, 738 cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{16}H_{18}NaO_5$: 313.1046; found 313.1046.



2-Propylisoindoline-1,3-dione (8)[10]

8 was obtained from **7** as a white solid. Yield: 86%.

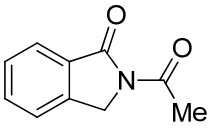
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.82 (m, 2H), 7.73-7.69 (m, 2H), 3.66 (t, *J* = 7.6 Hz, 2H), 1.71 (sext, *J* = 7.2 Hz, 2H) 0.96 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 133.8, 132.1, 123.1, 39.6, 21.9, 11.3 ppm.



2-Phenylisoindoline-1,3-dione (10)[11]

10 was obtained from **9** as a white solid. Yield: 93%.

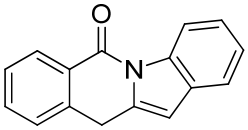
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.95 (m, 2H), 7.82-7.78 (m, 2H), 7.54-7.40 (m, 5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 134.3, 131.7, 131.6, 129.0, 128.0, 126.5, 123.7 ppm.



2-Acetylisoindolin-1-one (12)[12]

12 was obtained from **11** as a white solid. Yield: 89%.

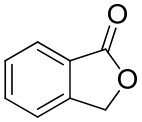
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 8.8 Hz, 1H), 7.54-7.50 (m, 2H), 4.82 (s, 2H), 2.69 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 167.7, 141.1, 134.1, 131.2, 128.6, 125.2, 123.5, 48.1, 24.8 ppm.



Indolo[1,2-*b*]isoquinolin-6(11*H*)-one (14)[13]

14 was obtained from **13** as a white solid. Yield: 88%.

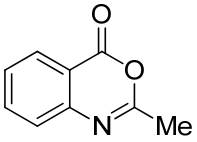
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (d, *J* = 8.0 Hz, 1H), 8.52 (d, *J* = 7.6 Hz, 1H), 7.65-7.61 (m, 1H), 7.51-7.39 (m, 4H), 7.25 (t, *J* = 7.6 Hz, 1H), 6.68 (s, 1H), 4.19 (s, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.2, 142.9, 141.7, 136.5, 132.3, 128.7, 128.1, 127.8, 126.1, 125.63, 125.61, 125.5, 124.3, 117.7, 102.5, 34.0 ppm.



Isobenzofuran-1(3*H*)-one (16)[14]

16 was obtained from **15a** or **15b** as a white solid. Yield: 60% from **15a**, 70% from **15b**.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.6 Hz, 1H), 7.70 (t, *J* = 8.0 Hz, 1H), 7.54 (dd, *J* = 8.0, 7.6 Hz, 2H), 5.33 (s, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 146.5, 133.9, 128.9, 125.63, 125.61, 122.1, 69.6 ppm.

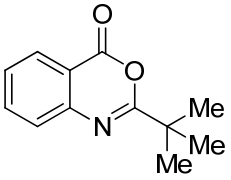


20

2-Methyl-4*H*-benzo[*d*][1,3]oxazin-4-one (20)[15]

20was obtained from **19** as a white solid. Yield: 63%.

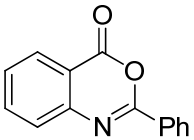
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.80 (dt, *J* = 7.6, 1.2 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 2.47 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 159.6, 146.4, 136.5, 128.4, 128.1, 126.4, 116.6, 21.3 ppm.



2-(*tert*-Butyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (22)[16]

22 was obtained from **21** as a white solid. Yield: 75%.

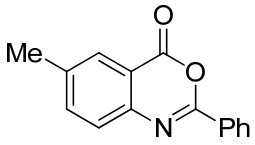
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.0 Hz, 1H), 7.78 (t, *J* = 8.0 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 1.41 (s, 9H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2, 160.0, 146.5, 136.2, 128.2, 128.0, 126.9, 116.7, 37.9, 27.6 ppm.



2-Phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (24)[17]

24 was obtained from **23** as a white solid. Yield: 77%.

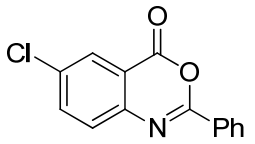
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (dd, *J* = 7.6, 1.2 Hz, 2H), 8.22 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.80 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.58-7.54 (m, 1H), 7.59-7.46 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 157.0, 146.9, 136.4, 132.5, 130.1, 128.7, 128.5, 128.2, 128.1, 127.1, 116.9 ppm.



6-Methyl-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (26)[18]

26 was obtained from **25** as a white solid. Yield: 81%.

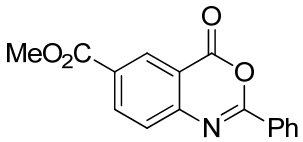
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28-8.26 (m, 2H), 8.00 (s, 1H), 7.60-7.47 (m, 5H), 2.47 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 156.3, 144.7, 138.6, 137.7, 132.3, 130.3, 128.6, 128.12, 128.08, 126.9, 116.6, 21.2 ppm.



6-Chloro-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (28)[19]

28 was obtained from **27** as colorless needles. Yield: 80%.

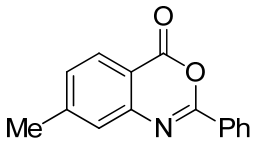
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, J = 7.6 Hz, 2H), 8.21 (d, J = 2.4 Hz, 1H), 7.77 (dd, J = 7.6, 2.4 Hz, 1H), 7.65 (d, J = 8.4 Hz, 1H), 7.60 (t, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 157.3, 145.5, 136.9, 133.9, 132.9, 129.9, 128.82, 128.78, 128.4, 128.0, 118.1 ppm.



Methyl 4-oxo-2-phenyl-4*H*-benzo[*d*][1,3]oxazine-6-carboxylate (30)[20]

30 was obtained from **29** as a white solid. Yield: 76%.

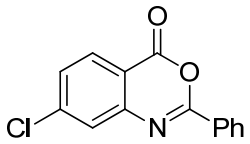
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, J = 2.0 Hz, 1H), 8.46 (dd, J = 8.4, 2.0 Hz, 1H), 8.34 (dd, J = 7.2, 1.6 Hz, 2H), 7.75 (d, J = 8.4 Hz, 1H), 7.62-7.52 (m, 3H), 3.97 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 158.8, 158.7, 150.1, 137.0 ,133.2 ,130.7, 129.8, 128.9, 128.7, 127.5, 121.7, 116.9, 52.6 ppm.



7-Methyl-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (32)[21]

32 was obtained from **31** as a white solid. Yield: 61%.

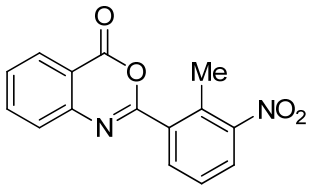
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33-8.30 (m, 2H), 8.13 (d, J = 8.4 Hz, 1H), 7.58-7.50 (m, 4H), 7.34 (d, J = 8.0 Hz, 1H), 2.52 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 157.2, 148.1, 147.0, 132.5, 130.4, 129.6, 128.7, 128.4, 128.3, 127.2, 114.4, 22.1 ppm.



7-Chloro-2-phenyl-4*H*-benzo[*d*][1,3]oxazin-4-one (34)[19]

34 was obtained from **33** as a pale yellow solid. Yield: 76%.

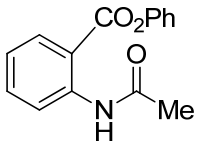
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30-8.28 (m, 2H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 1.6 Hz, 1H), 7.62-7.46 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 158.3, 148.1, 143.0, 133.0, 129.89, 129.85, 128.83, 128.78, 128.5, 127.0, 115.4 ppm.



2-(2-Methyl-3-nitrophenyl)-4*H*-benzo[*d*][1,3]oxazin-4-one (36)

36 was obtained from **35** as a white solid. Yield: 70%.

M.p. 167–168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 7.6 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.93-7.87 (m, 2H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 2.75 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 156.5, 152.1, 146.1, 136.8, 133.7, 133.3, 132.6, 129.3, 128.6, 127.5, 126.7, 126.4, 116.8, 16.9 ppm; IR (ATR) 1752, 1602, 1521, 1359, 1008, 769 cm^{-1}; HRMS (ESI) $[M+H]^+$ calcd for $C_{15}H_{11}N_2O_4$: 283.0713; found 283.0713.

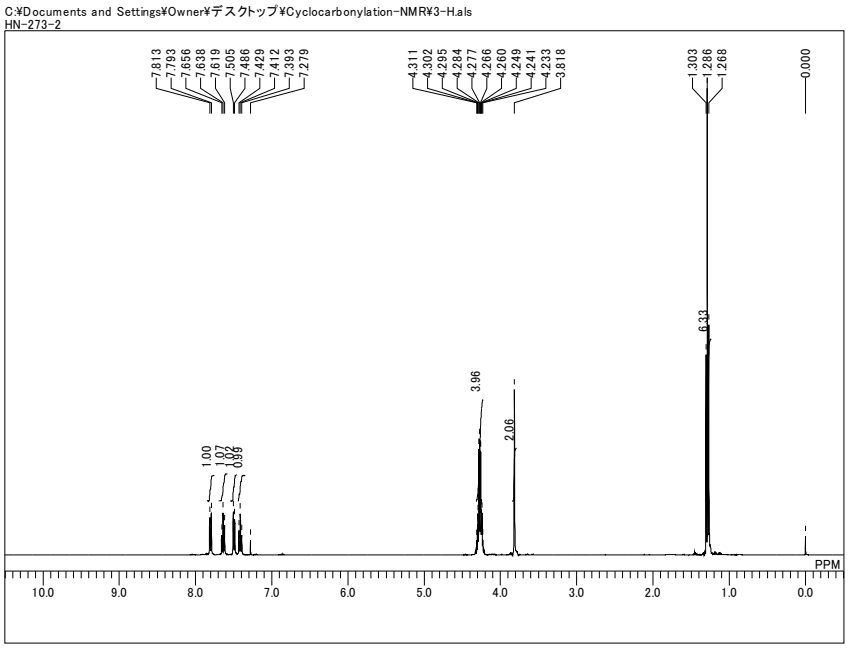


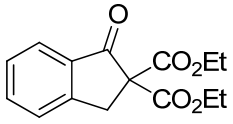
Phenyl 2-acetamidobenzoate (37)

37 was obtained in DMSO for 4 h from **19** as colorless needles. Yield: 72%.

M.p. 80–81 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.90 (br s, 1H), 8.78 (d, *J* = 8.0 Hz, 1H), 8.29 (d, *J* = 8.0 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.33 (dd, *J* = 7.2, 6.0 Hz, 1H), 7.19-7.15 (m, 3H), 2.19 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 167.2, 150.3, 142.3, 135.5, 131.3, 129.7, 126.4, 122.6, 121.7, 120.5, 113.9, 25.4 ppm; IR (ATR) 3284, 1708, 1687, 1587, 1529, 1261, 1232, 1192, 1161, 1062, 748 cm^{-1}; HRMS (ESI) $[M+Na]^+$ calcd for $C_{15}H_{13}NNaO_3$: 278.0788; found 278.0790.

6. NMR spectra of obtained compounds





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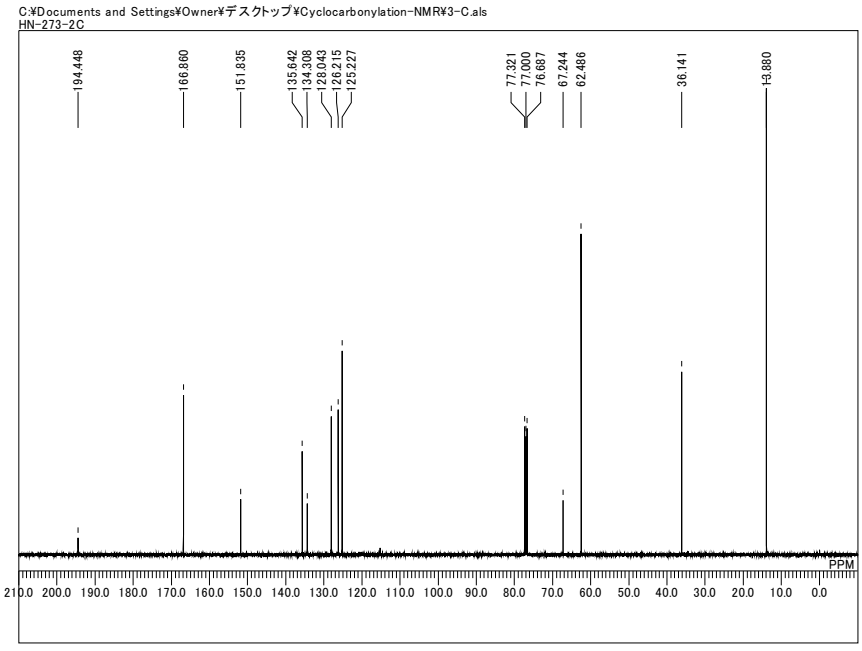

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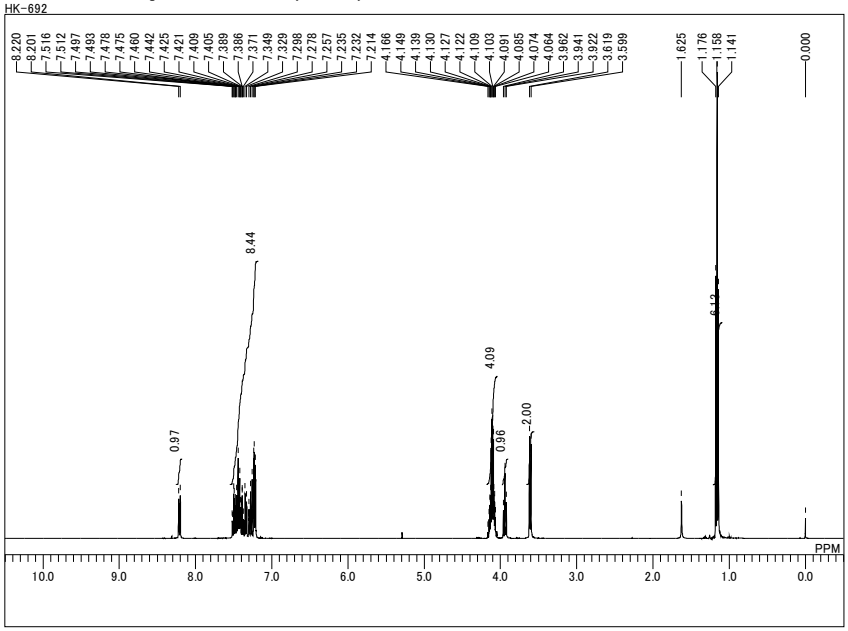
HK-692



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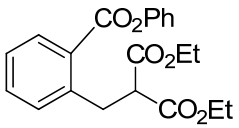

4

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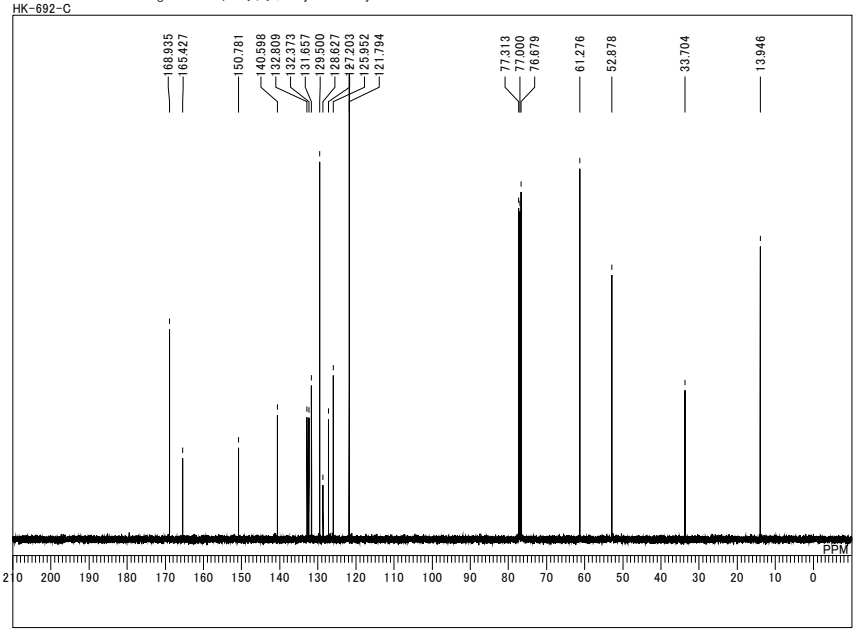

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PD	1.7920 sec
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CTEMP	24.3 c
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EXREF	77.00 ppm
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RGAIN	27

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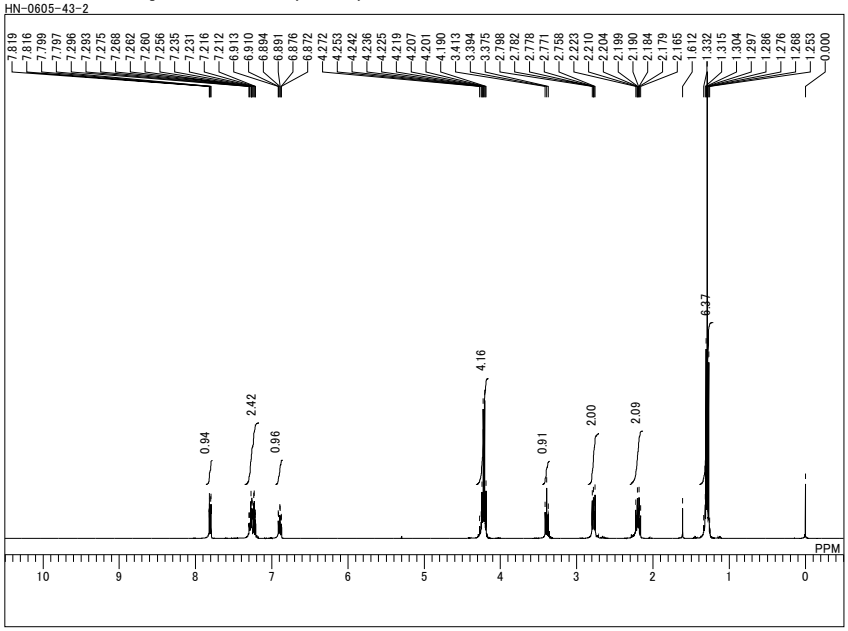
HN-0605-43-2



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EXREF	0.00 ppm
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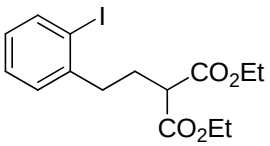
RGAIN	13



5

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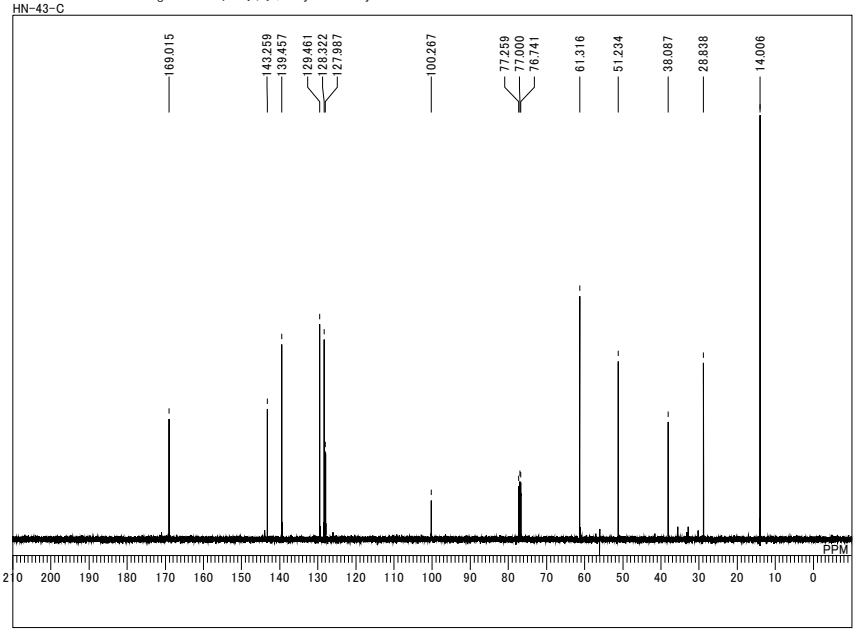
HN-43-C



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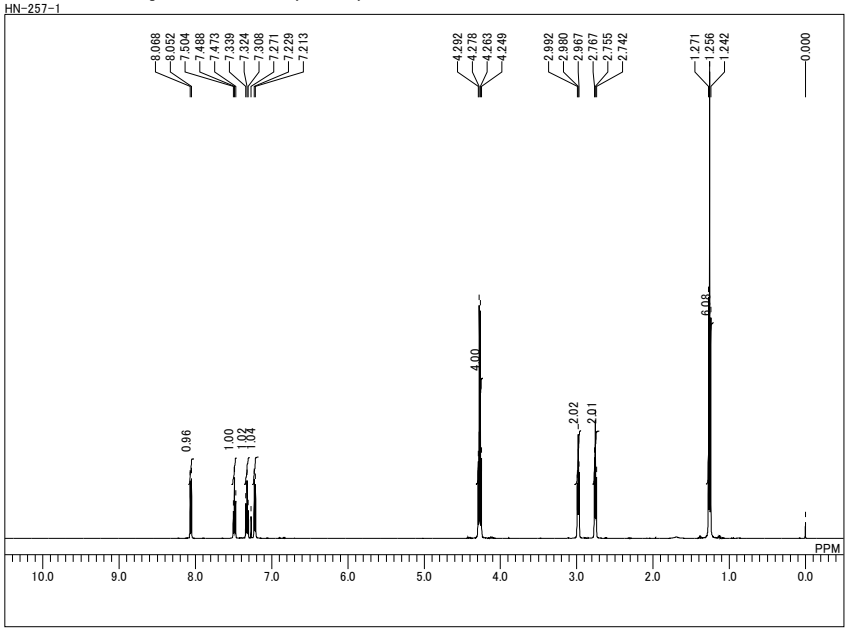
HN-257-1



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CTEMP 24.0 c
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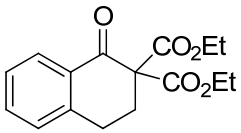
RGAIN 30



6

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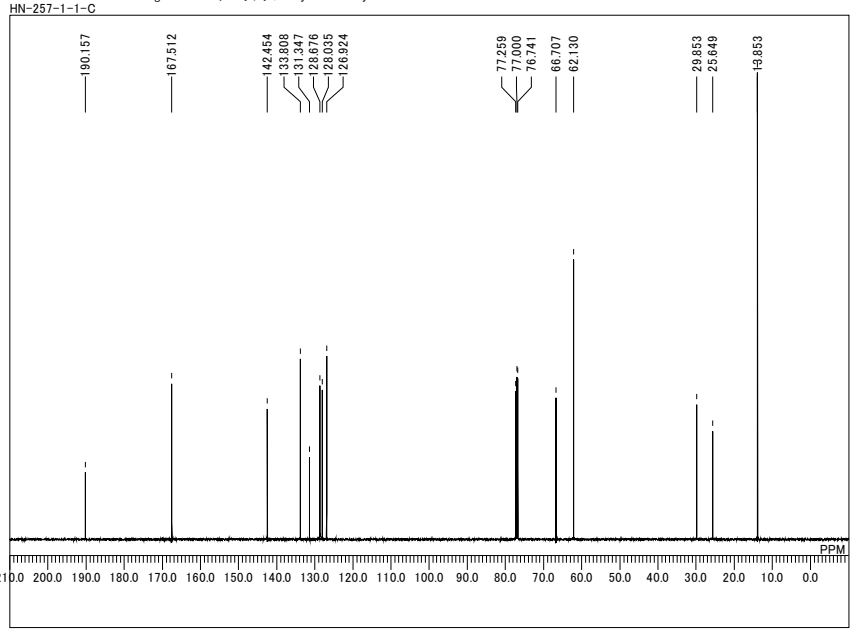

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SLVNT CDCL3
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RGAIN 54

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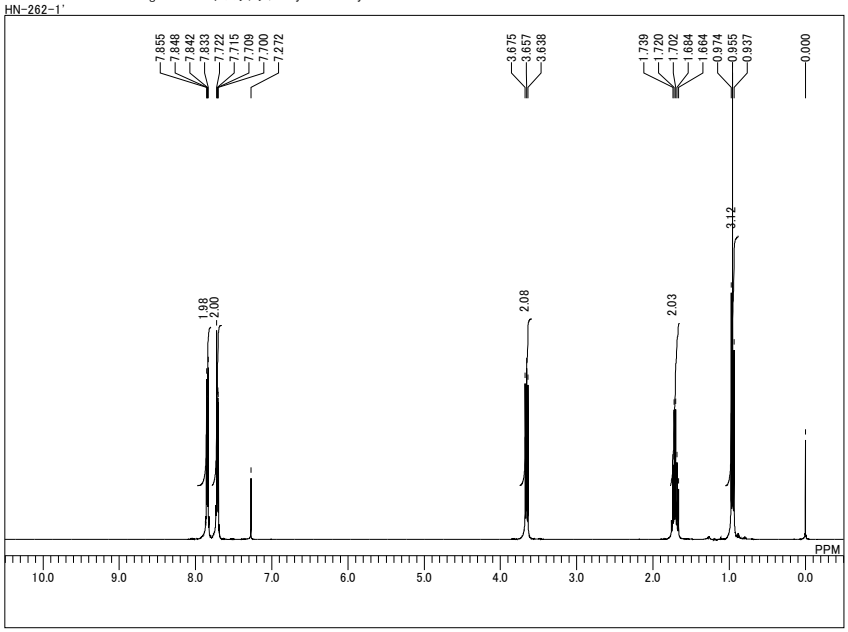
HN-262-1'



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ACQTM	2.0500 sec
PD	4.9500 sec
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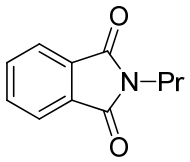
RGAIN	13



8

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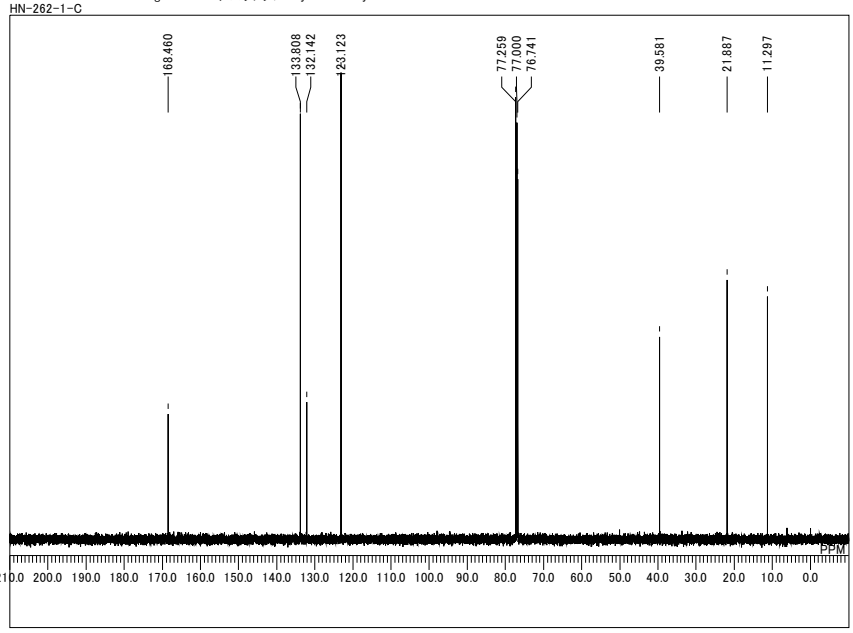
HN-262-1-C



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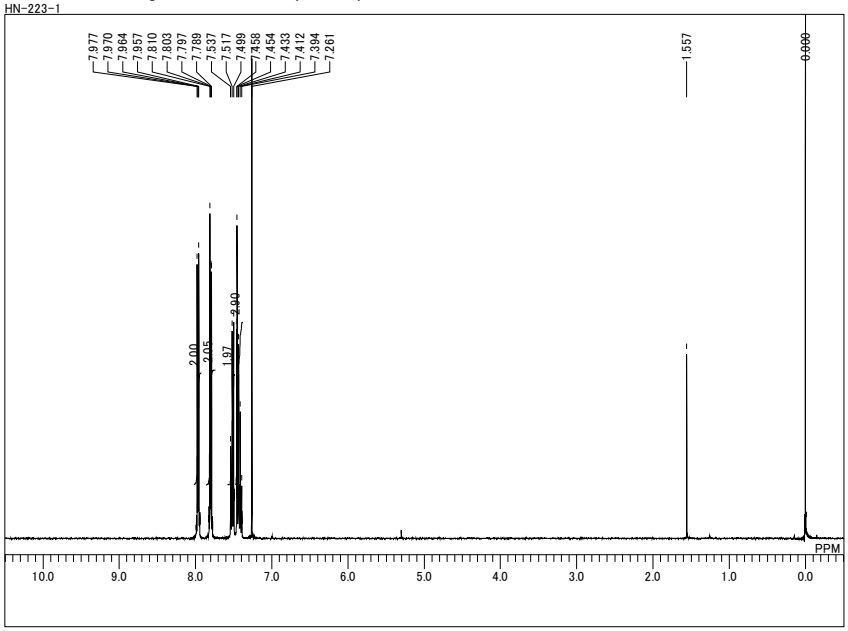
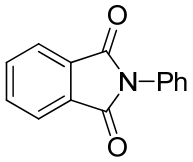
COMNT	HN-262-1-C
DATIM	29-04-2014 20:28:52
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	1200
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥10-H.als
HN-223-1

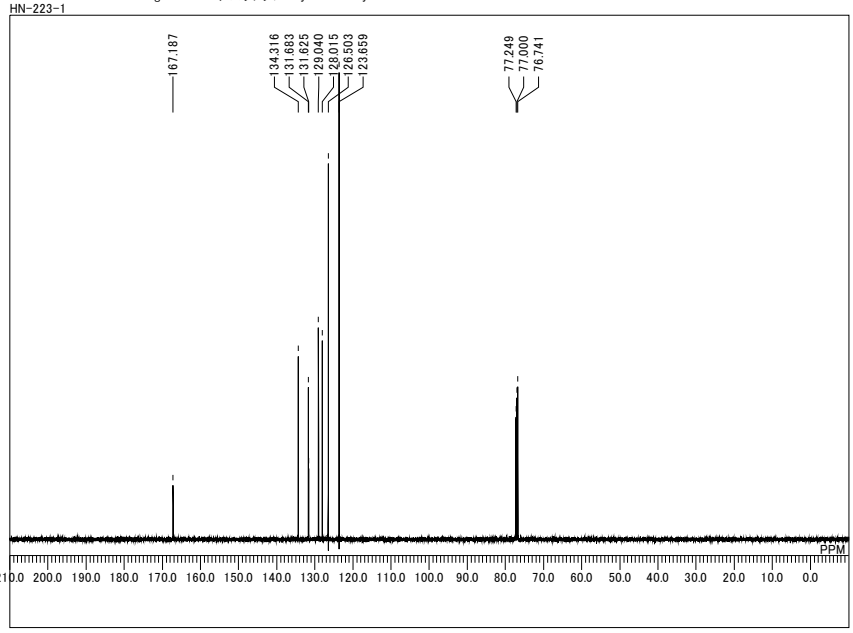


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-223-1
DATIM	Wed Dec 04 22:44:02 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	17



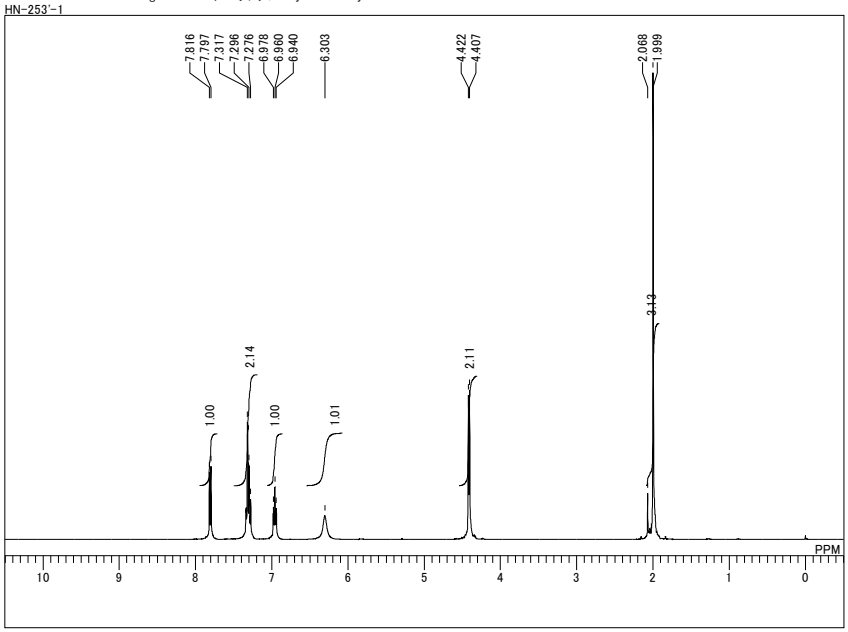
10

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥10-C.als
HN-223-1

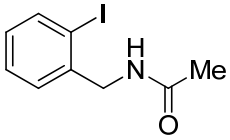


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-223-1
DATIM	22-04-2014 09:09:19
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	1000
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥11-H.als
HN-253'-1

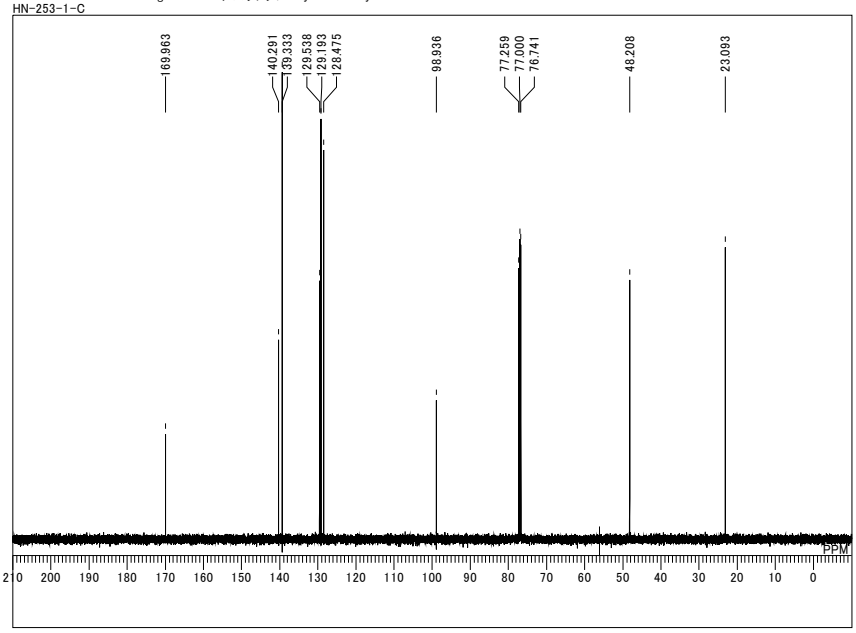


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-253'-1
DATIM	Tue Aug 26 13:07:51 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.14 Hz
RGAIN	10



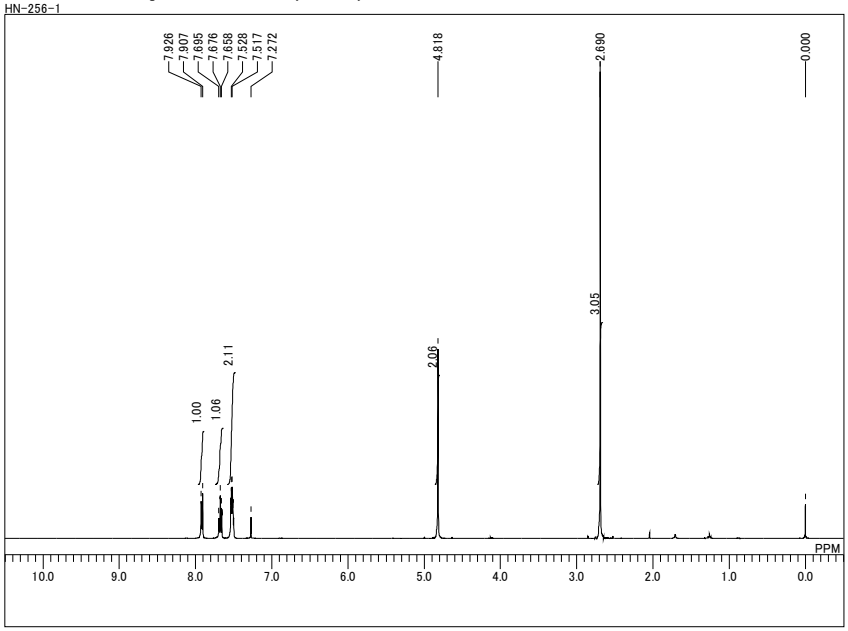
11

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥11-C.als
HN-253-1-C

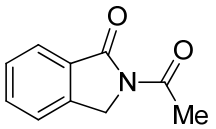


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-253-1-C
DATIM	26-08-2014 13:24:03
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	500
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.14 Hz
RGAIN	54

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥12-H.als
HN-256-1

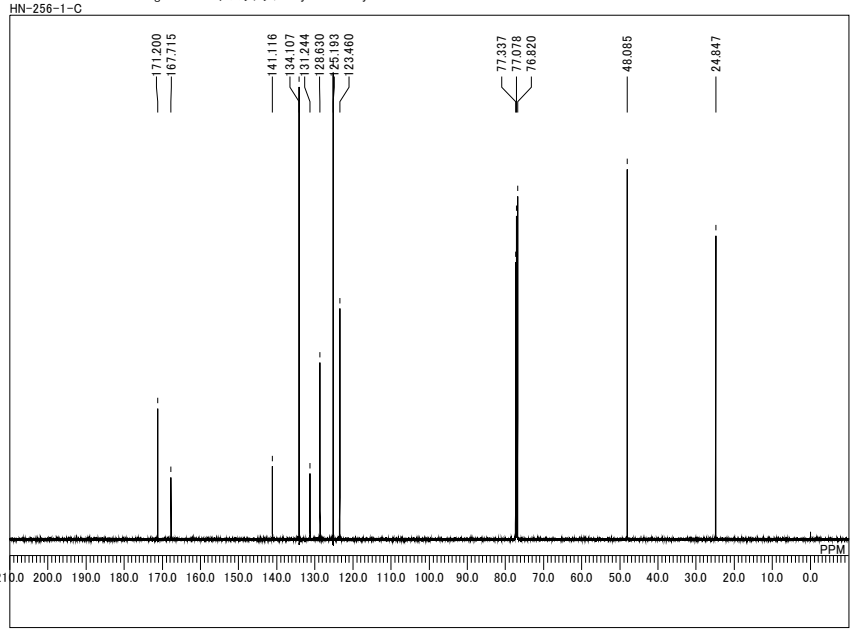


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-256-1
DATIM	Fri Jan 17 09:01:54 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	23.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	13



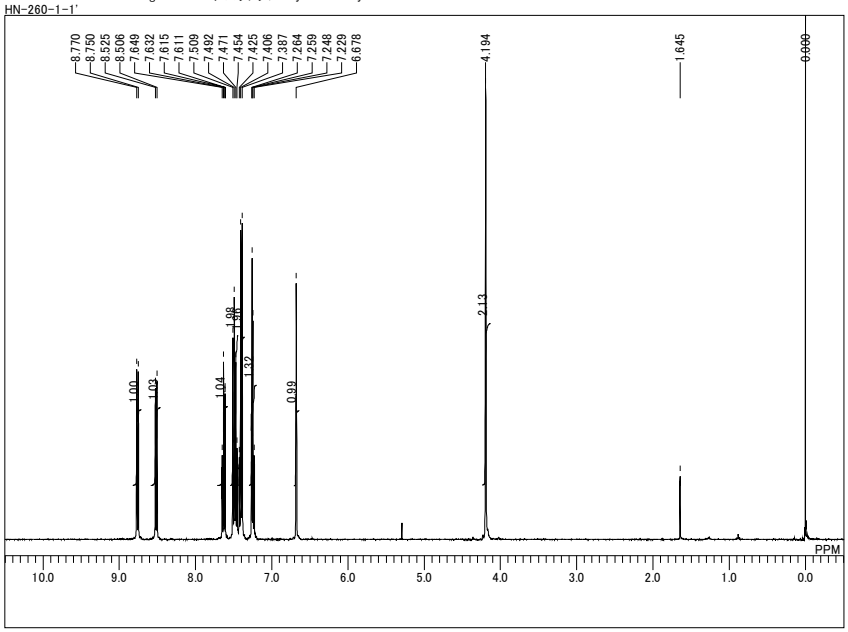
12

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥12-C.als
HN-256-1-C

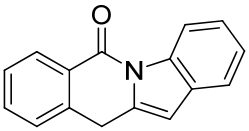


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-256-1-C
DATIM	22-04-2014 07:21:22
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	10000
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	54

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥14-H.als
HN-260-1-1'

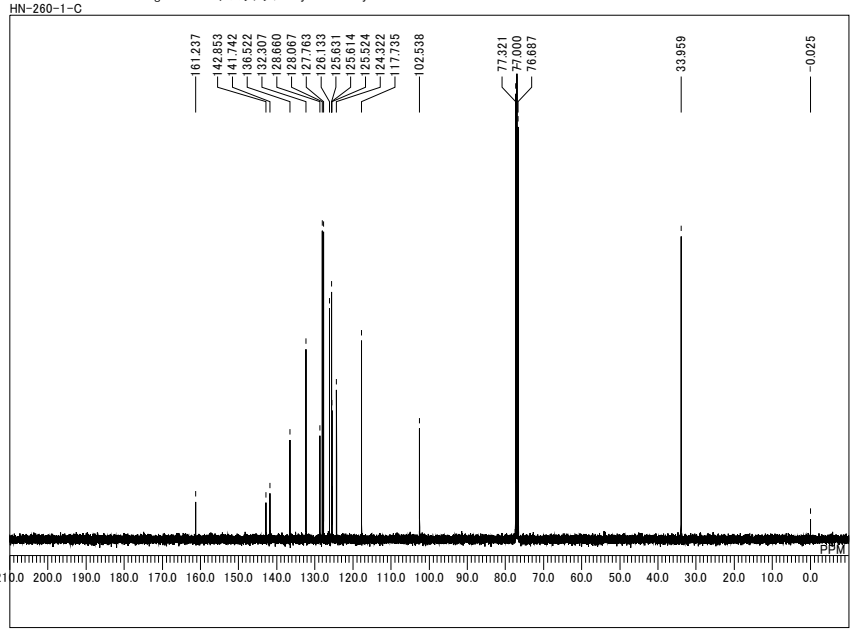


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-260-1-1'
DATIM	Tue Jan 21 01:04:11 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	16



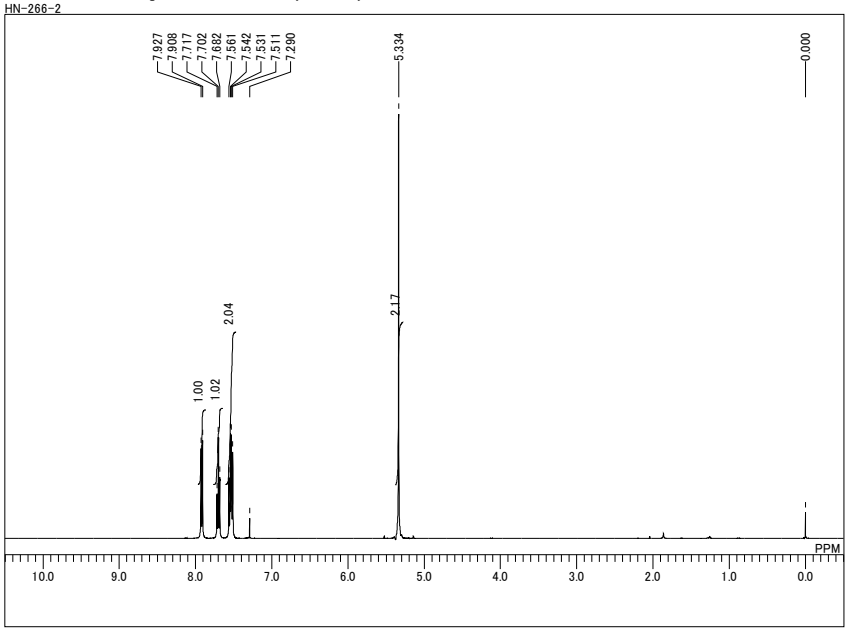
14

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥14-C.als
HN-260-1-C

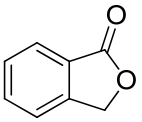


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-260-1-C
DATIM	Tue Jan 21 01:58:27 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥16-H.als
HN-266-2

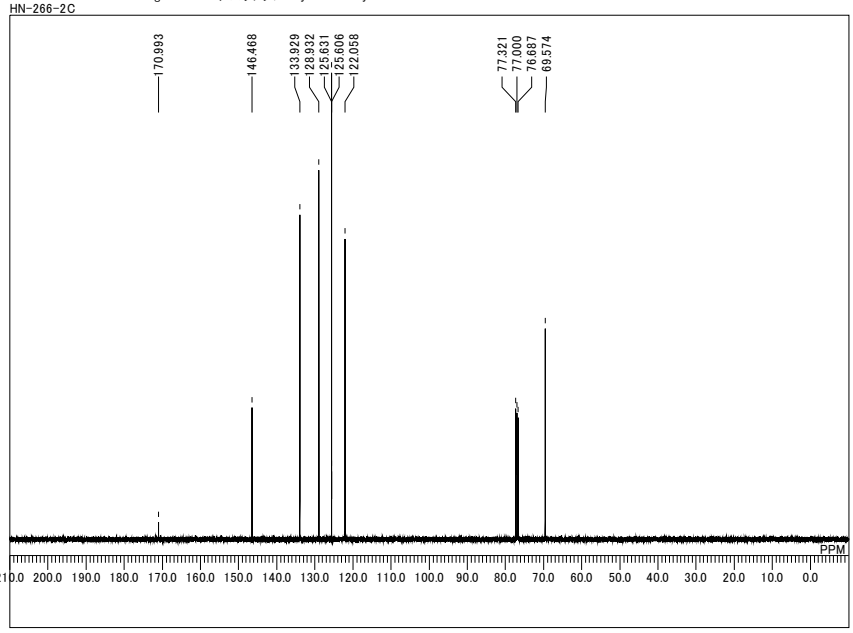


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-266-2
DATIM	Thu Jan 23 17:38:38 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	11



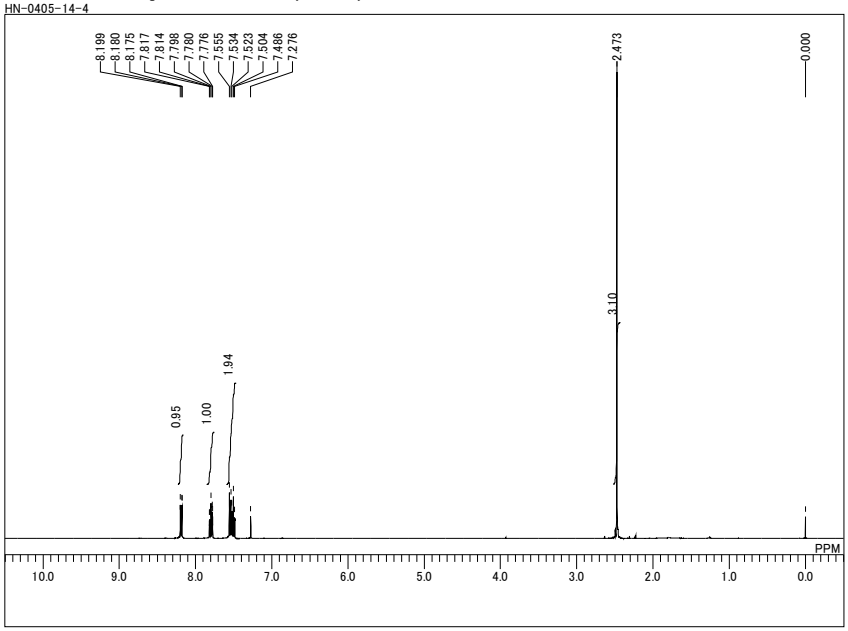
16

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥16-C.als
HN-266-2C

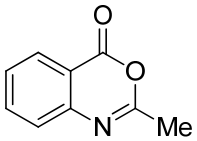


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-266-2C
DATIM	Thu Jan 23 18:25:40 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	512
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥20-H.als
HN-0405-14-4

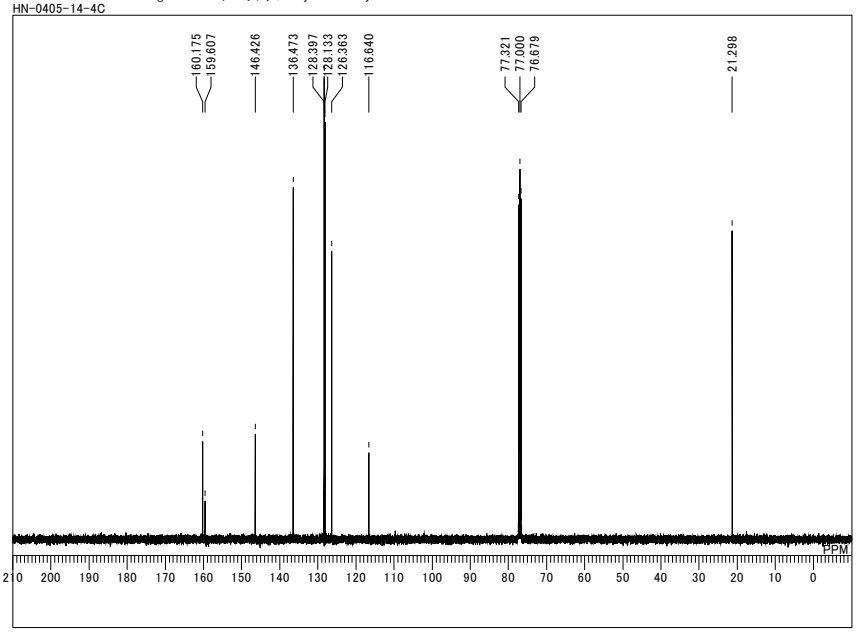


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-0405-14-4
DATIM	Fri Apr 05 15:52:36 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	24.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	14



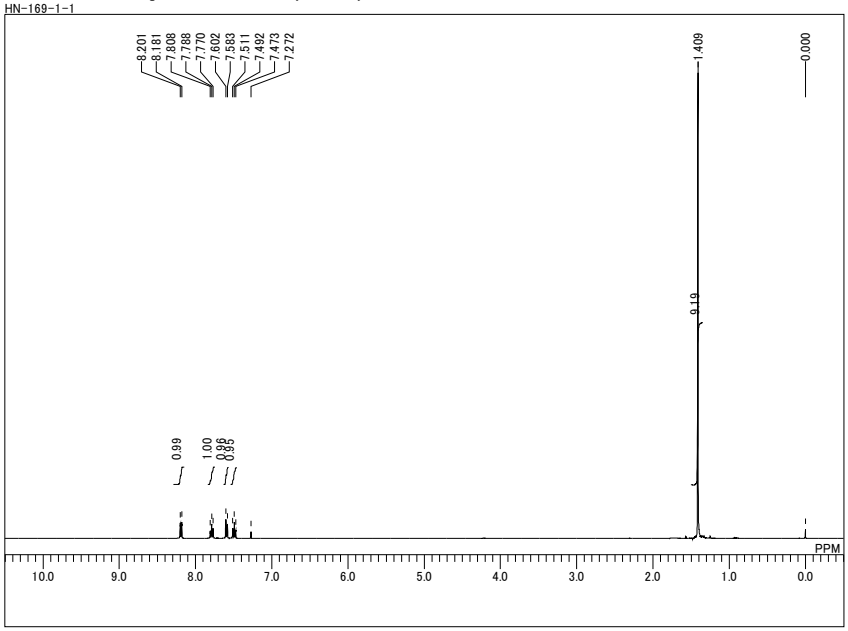
20

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥20-C.als
HN-0405-14-4C

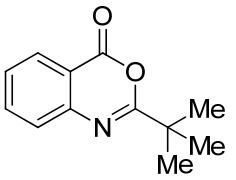


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-0405-14-4C
DATIM	Fri Apr 05 17:39:25 2013
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	26.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥22-H.als
HN-169-1-1

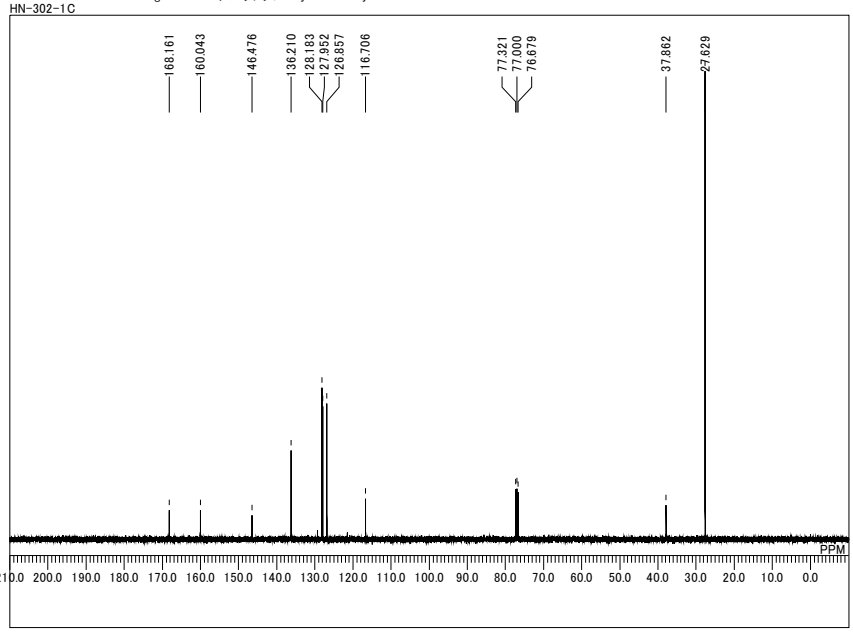


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-169-1-1
DATIM	Mon Oct 14 15:55:27 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	12



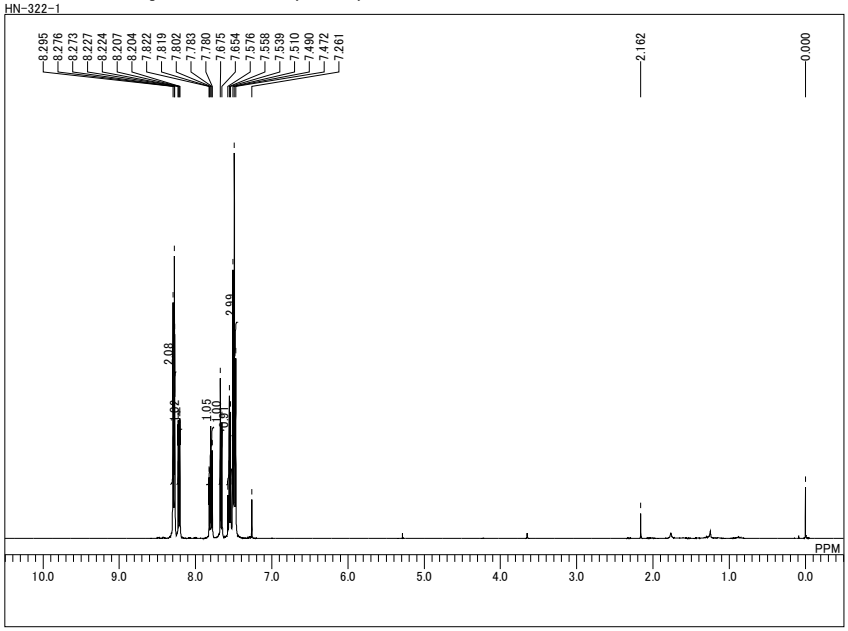
22

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥22-C.als
HN-302-1C

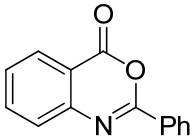


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-302-1C
DATIM	Fri Feb 21 08:24:33 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	512
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	21

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥24-H.als
HN-322-1

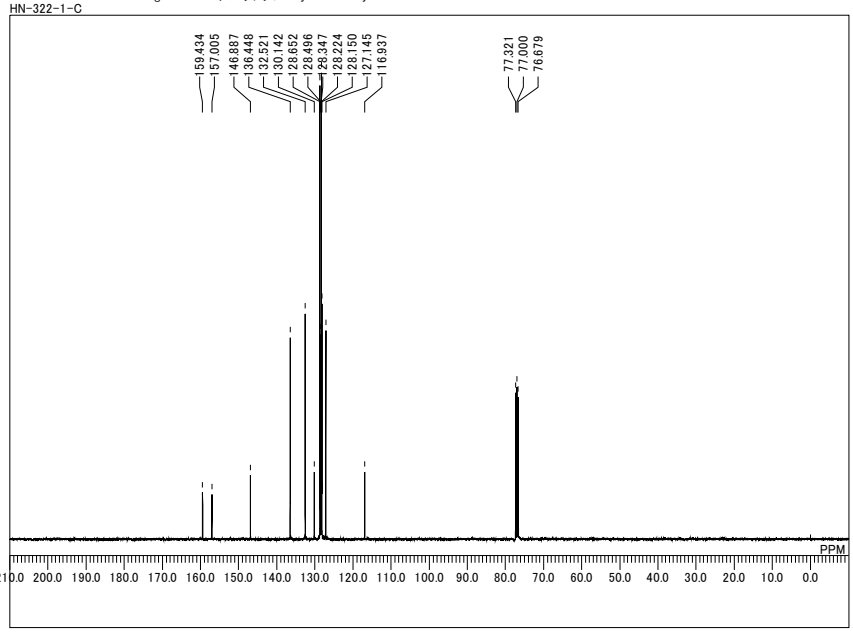


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-322-1
DATIM	Wed Mar 12 14:44:31 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	13



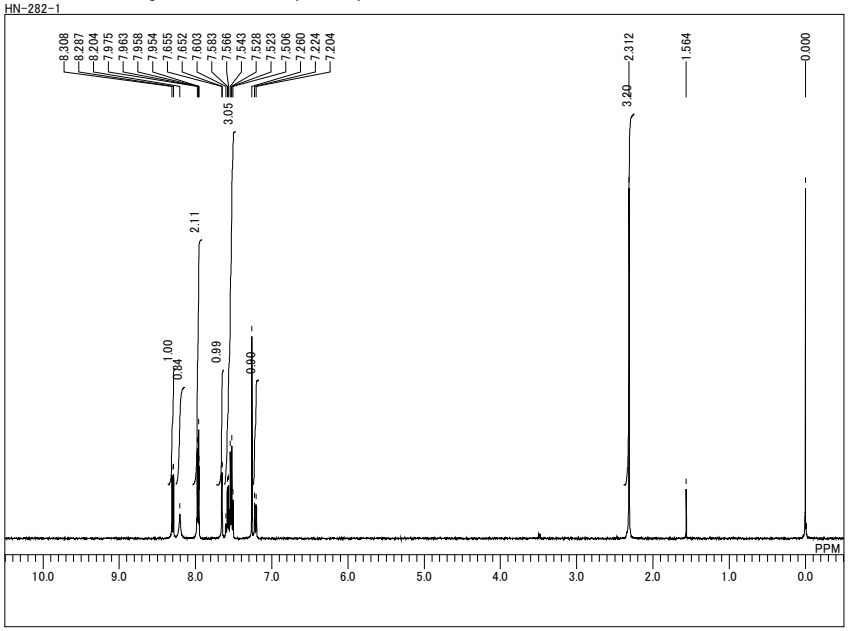
24

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥24-C.als
HN-322-1-C

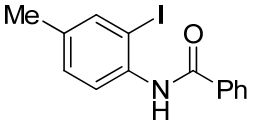


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-322-1-C
DATIM	Thu Mar 13 01:09:04 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	26

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥25-H.als
HN-282-1

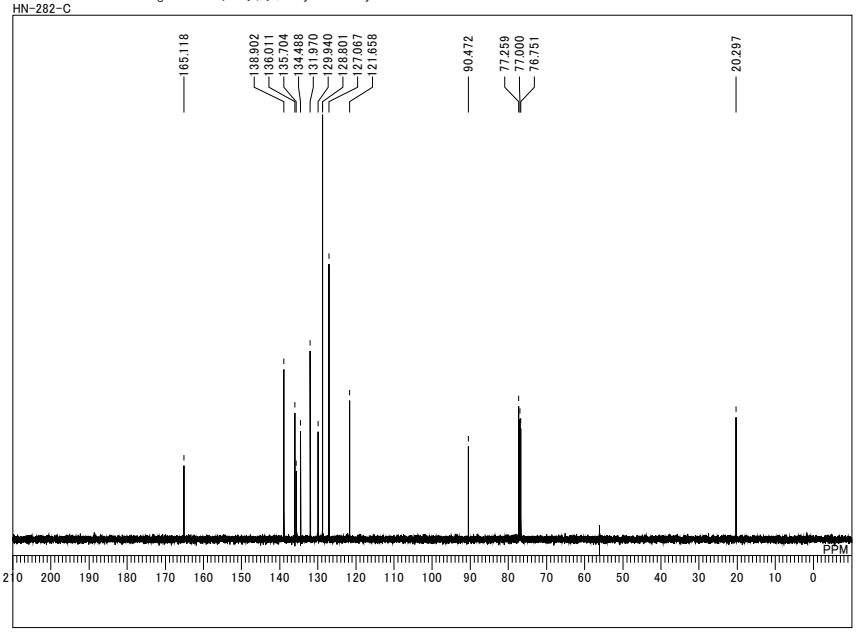


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-282-1
DATIM	Wed Feb 05 14:24:25 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	21



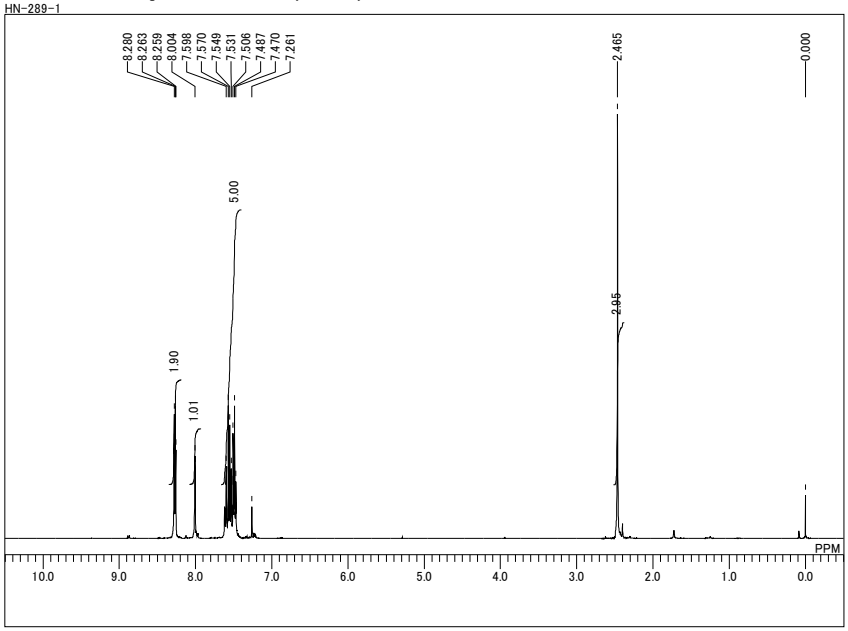
25

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥25-C.als
HN-282-C

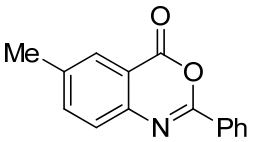


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-282-C
DATIM	28-08-2014 22:00:00
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	200
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.14 Hz
RGAIN	54

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥26-H.als
HN-289-1

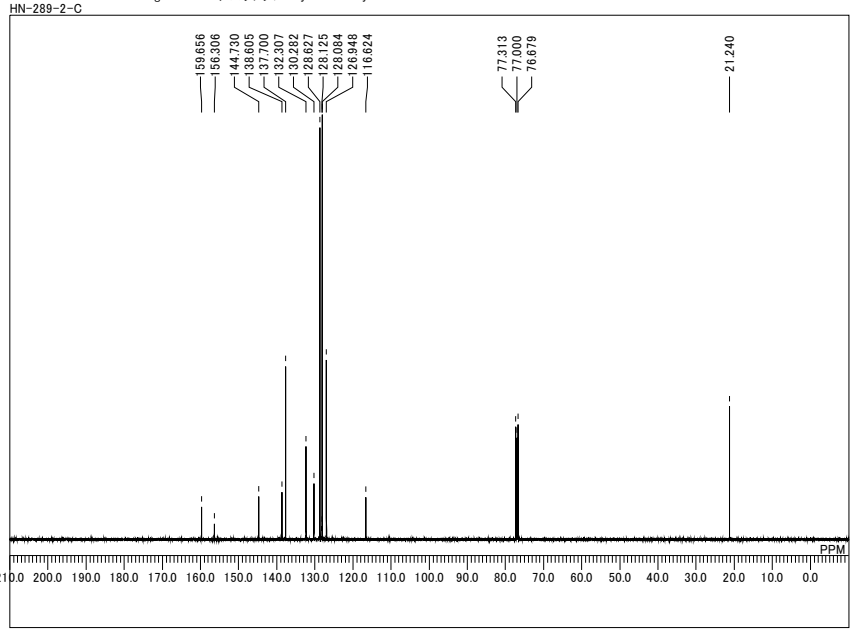


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-289-1
DATIM	Mon Feb 10 17:25:58 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	15



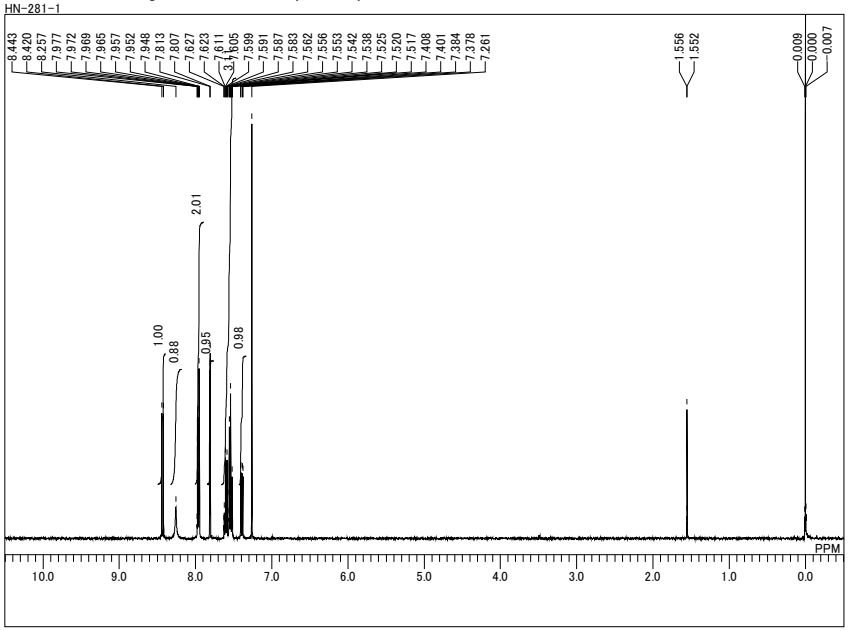
26

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥26-C.als
HN-289-2-C

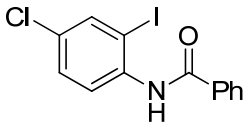


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-289-2-C
DATIM	Tue Feb 11 18:52:37 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥27-H.als
HN-281-1

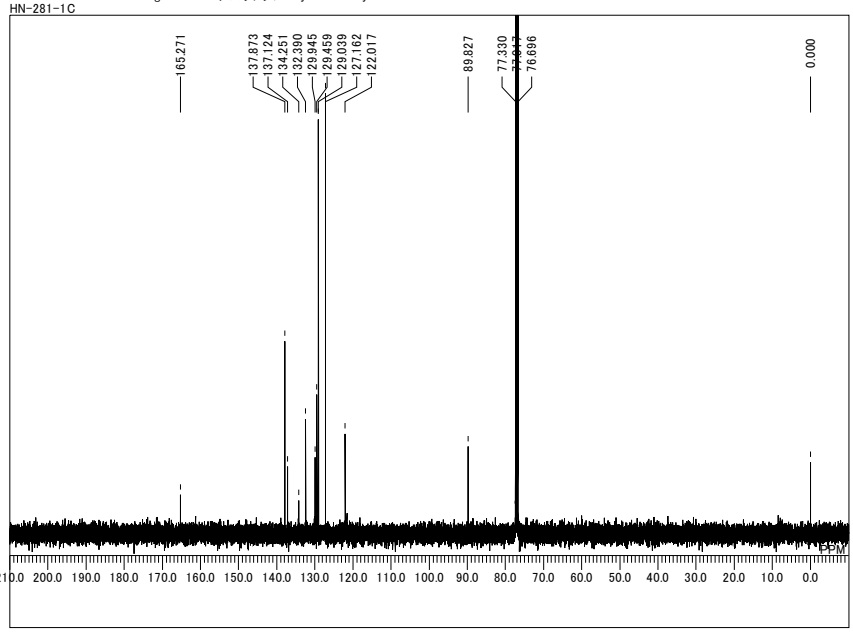


DFILE C:¥Documents and Settings¥Ow
COMNT HN-281-1
DATIM Tue Feb 04 18:07:07 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22



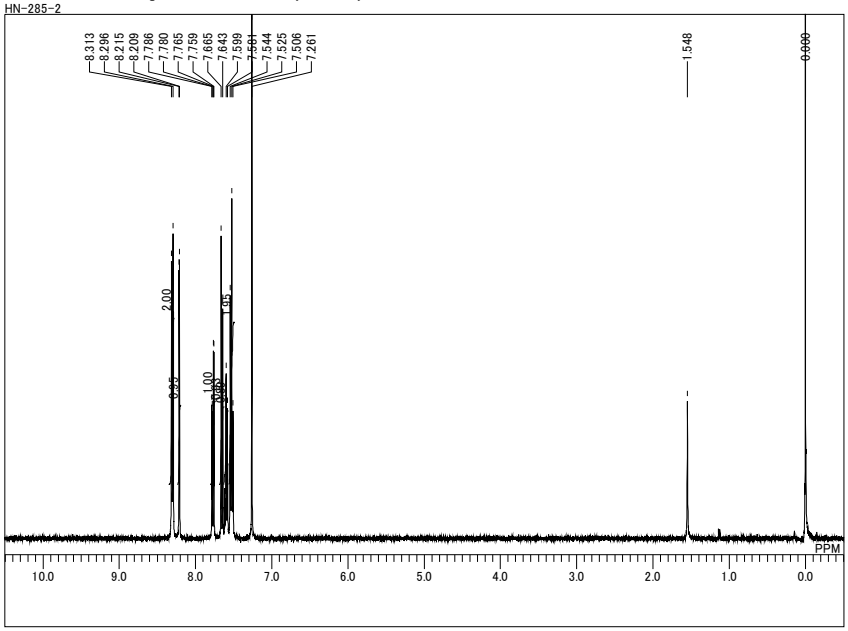
27

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥27-C.als
HN-281-1C

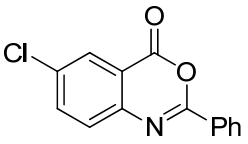


DFILE C:¥Documents and Settings¥Ow
COMNT HN-281-1C
DATIM Wed Feb 05 08:32:14 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥28-H.als
HN-285-2

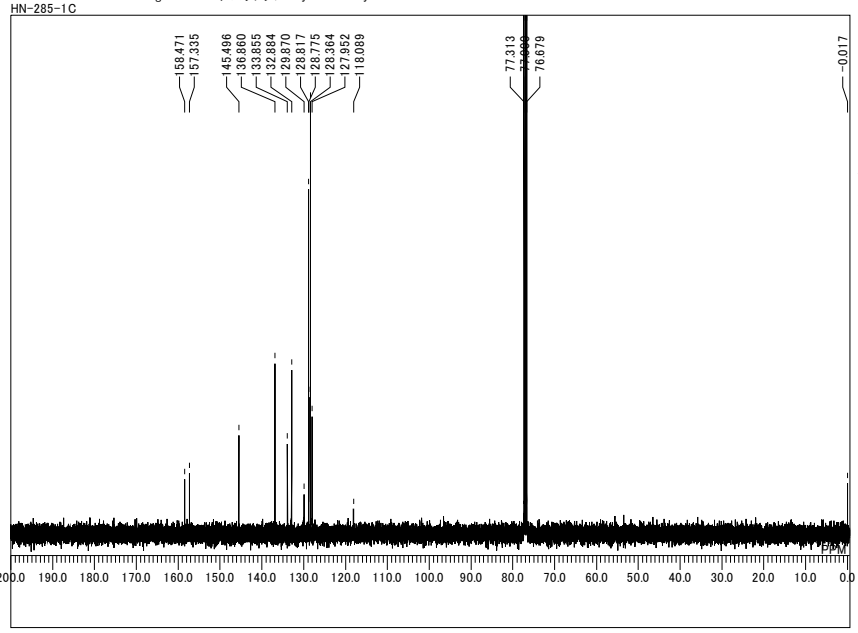


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-285-2
DATIM	Thu Feb 06 00:01:55 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	23



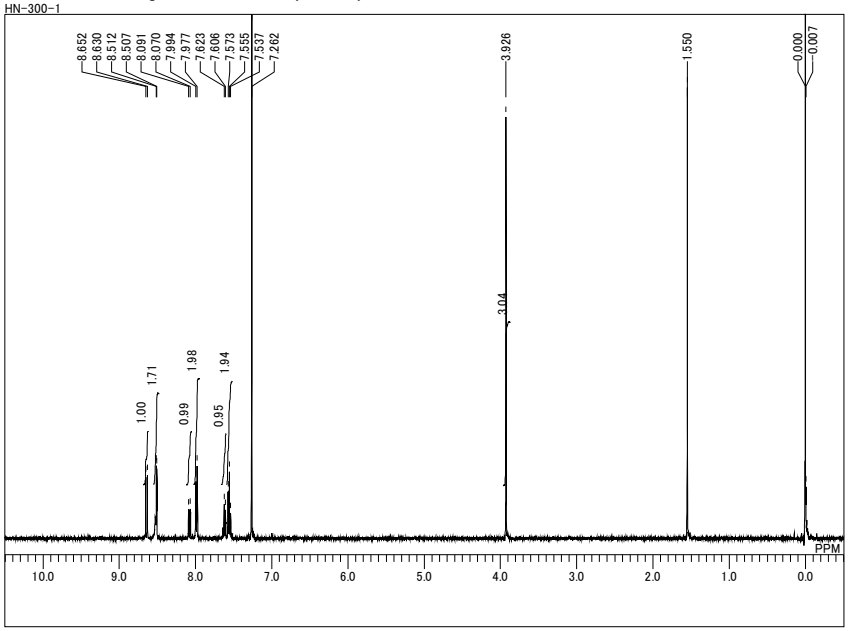
28

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥28-C.als
HN-285-1C

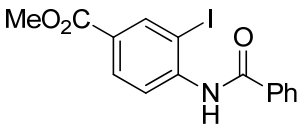


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-285-1C
DATIM	Thu Feb 06 00:58:28 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	26

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥29-H.als
HN-300-1

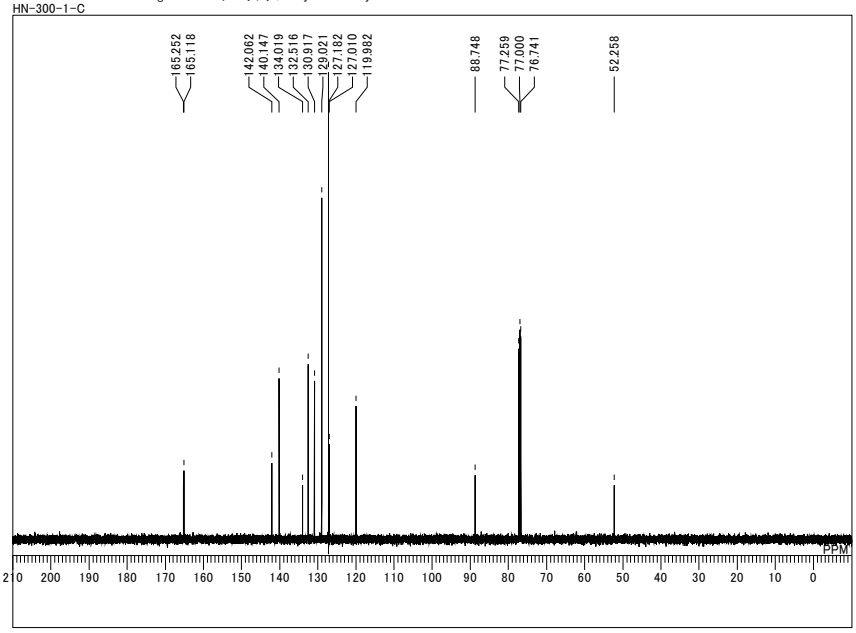


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-300-1
DATIM	Thu Feb 20 23:00:28 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	25

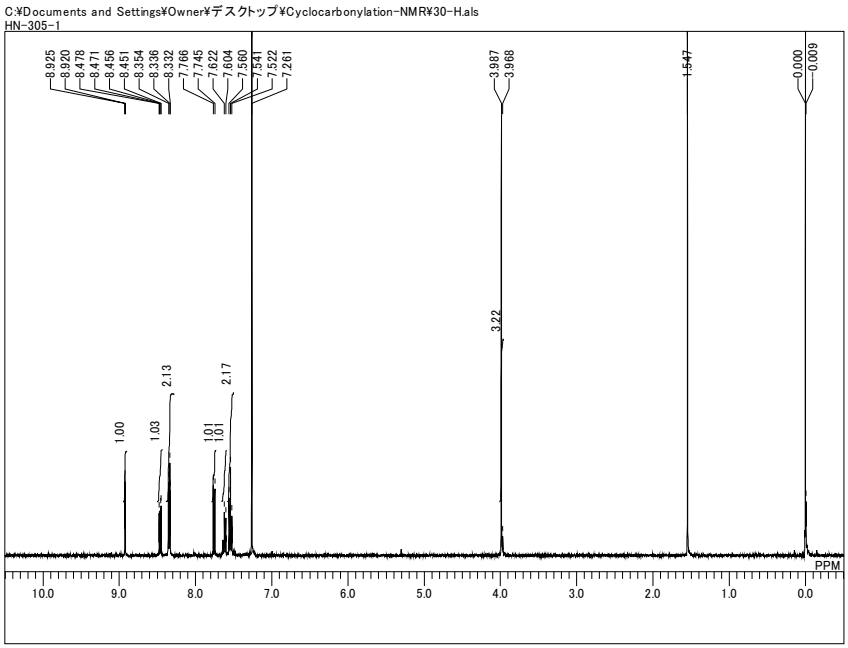


29

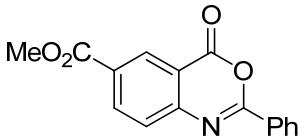
C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥29-C.als
HN-300-1-C



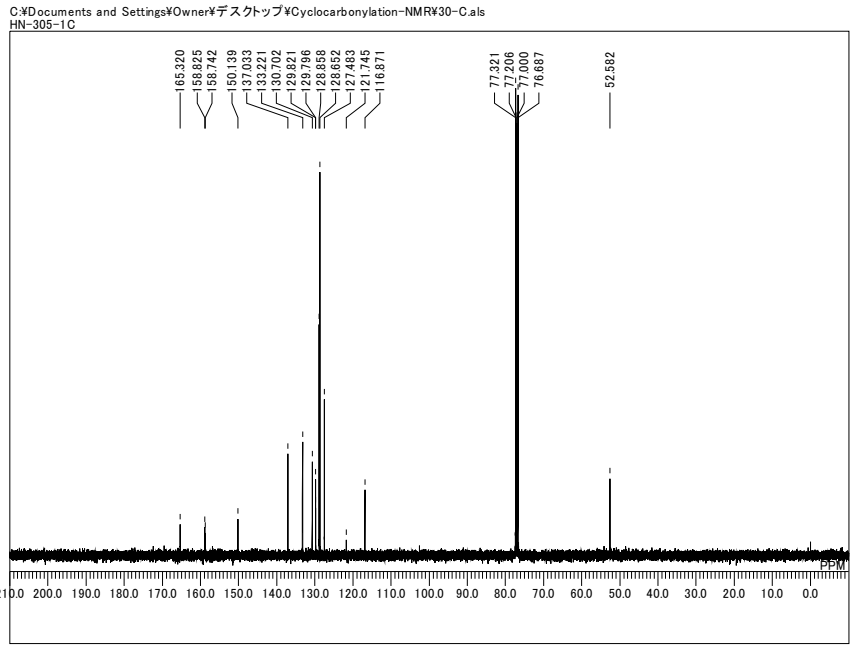
DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-300-1-C
DATIM	28-08-2014 18:09:46
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	400
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.14 Hz
RGAIN	54



DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-305-1
DATIM	Sat Mar 01 17:23:04 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	24

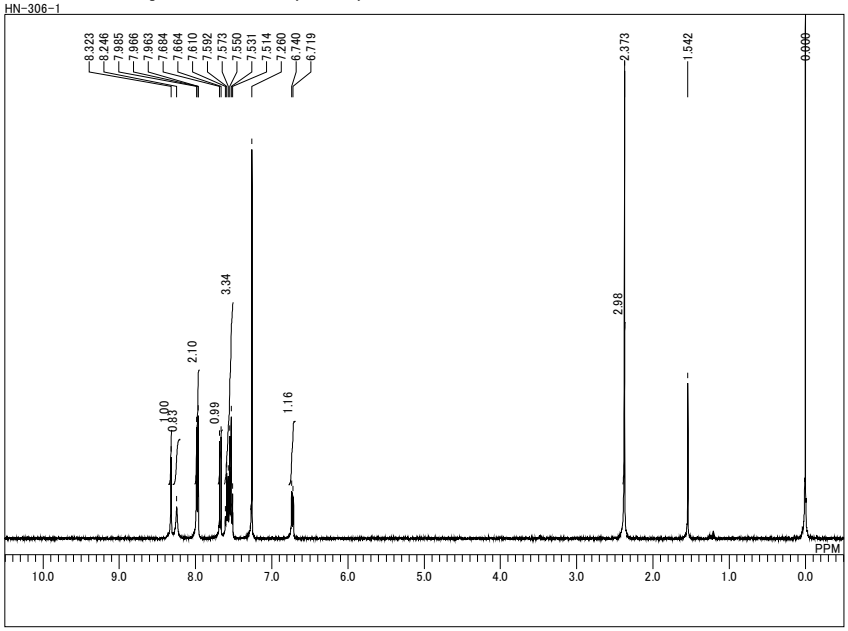


30

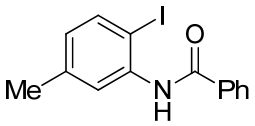


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-305-1C
DATIM	Sat Mar 01 18:40:11 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥31-H.als
HN-306-1

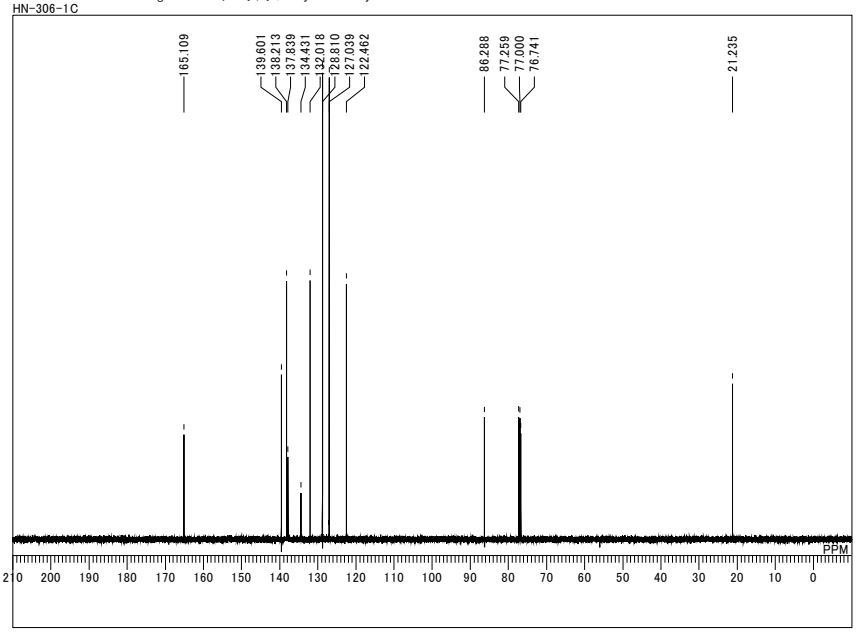


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-306-1
DATIM	Sun Mar 02 15:31:52 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	22



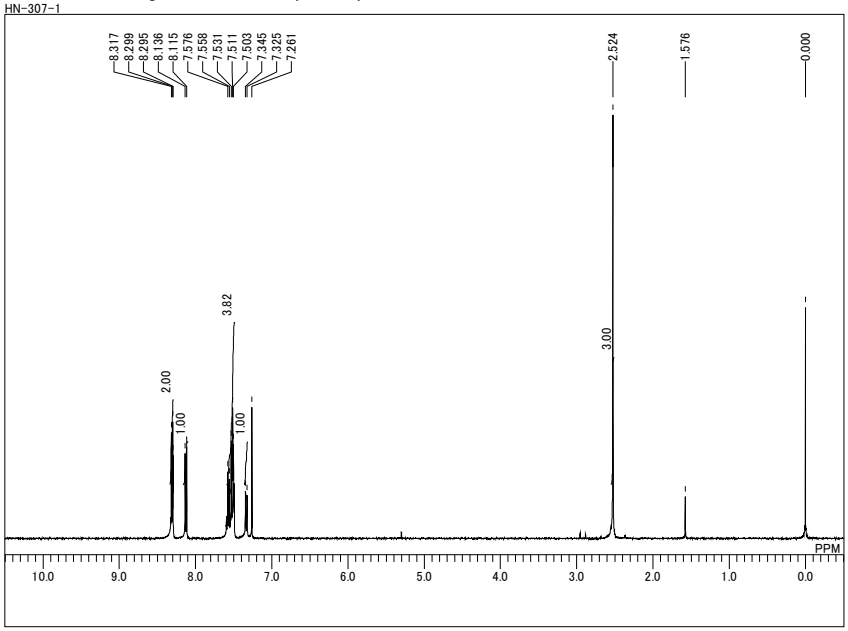
31

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥31-C.als
HN-306-1C

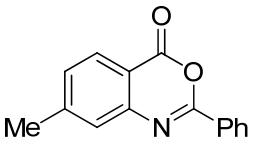


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-306-1C
DATIM	28-08-2014 18:26:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	200
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.14 Hz
RGAIN	48

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥32-H.als
HN-307-1

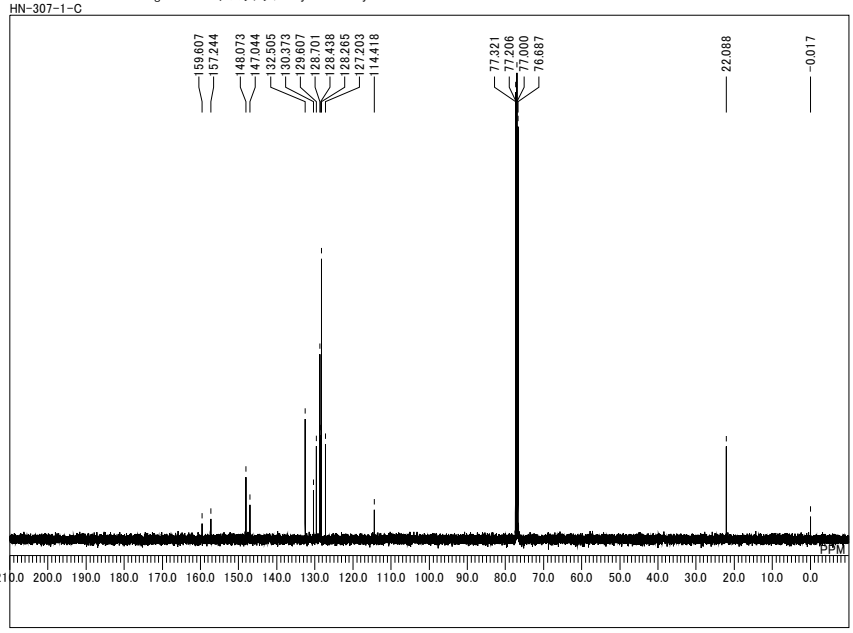


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-307-1
DATIM	Mon Mar 03 19:56:12 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20



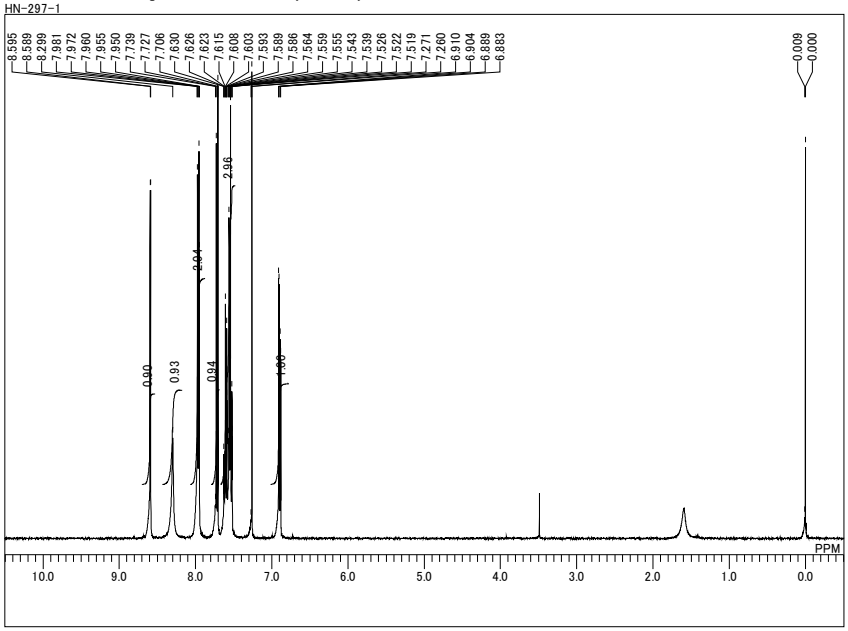
32

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥32-C.als
HN-307-1-C

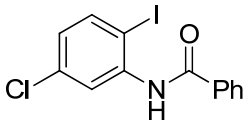


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-307-1-C
DATIM	Tue Mar 04 08:55:05 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥33-H.als
HN-297-1

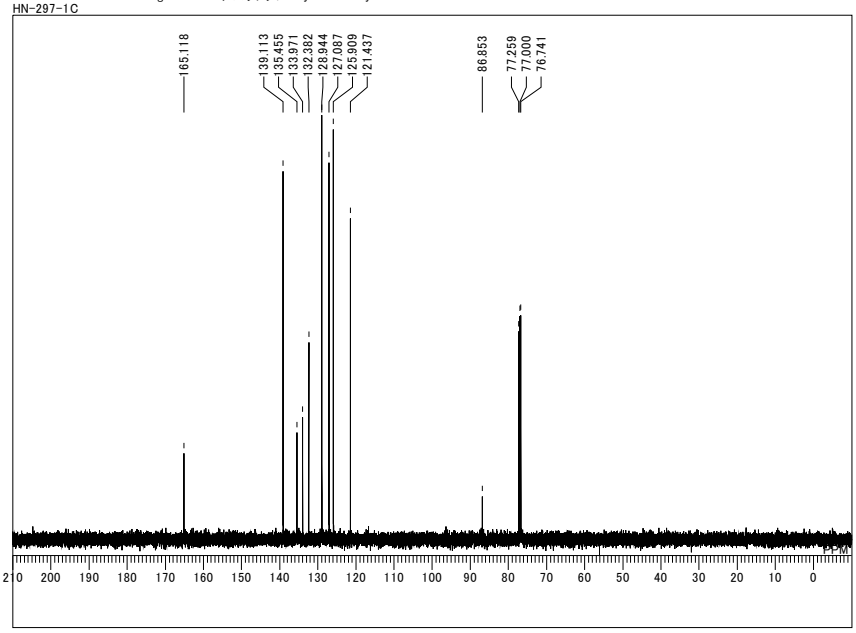


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-297-1
DATIM	Thu Sep 04 19:08:37 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	16



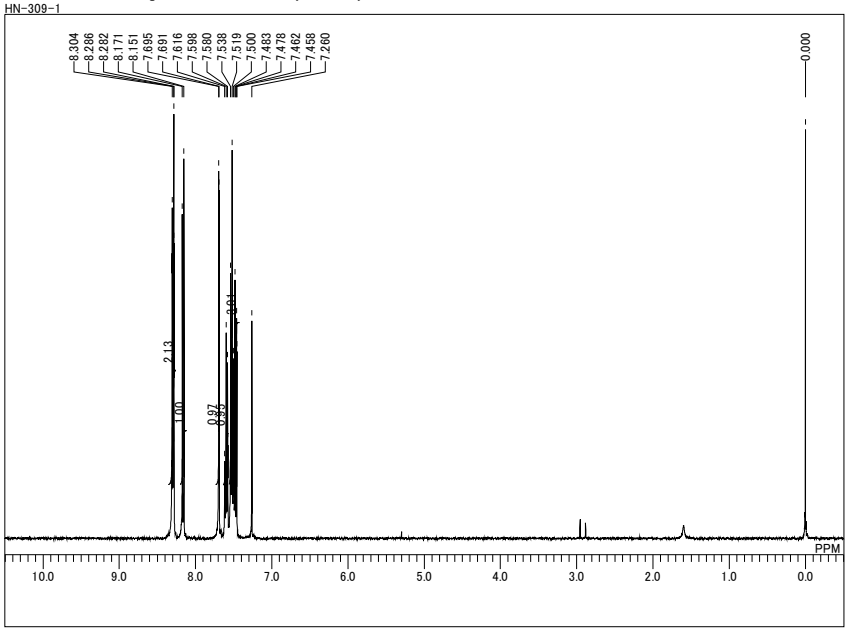
33

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥33-C.als
HN-297-1C

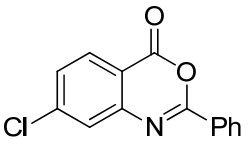


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-297-1C
DATIM	28-08-2014 18:46:49
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	124.51 MHz
OBSET	3.45 KHz
OBFIN	6.00 Hz
POINT	26214
FREQU	31249.52 Hz
SCANS	200
ACQTM	0.8389 sec
PD	2.0000 sec
PW1	5.20 usec
IRNUC	1H
CTEMP	24.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.14 Hz
RGAIN	48

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥34-H.als
HN-309-1

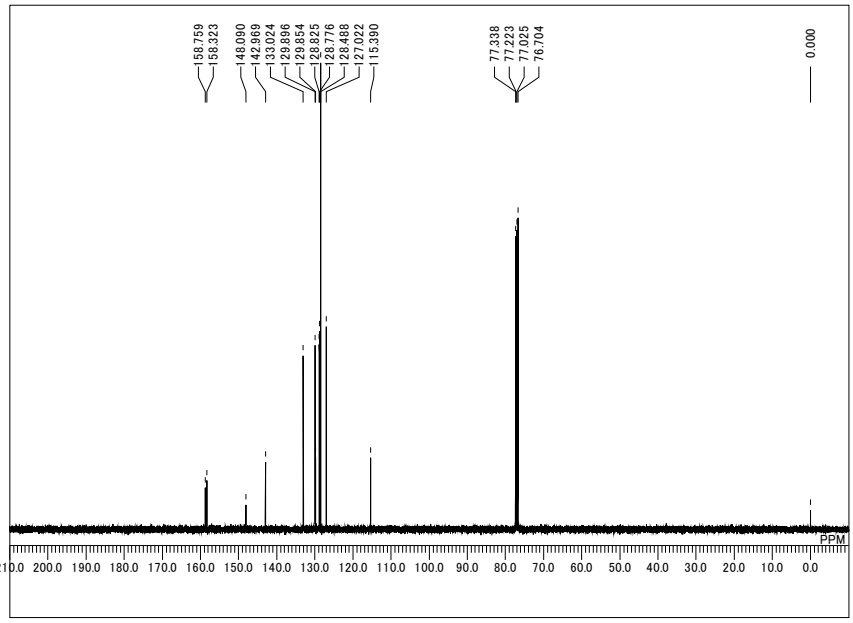


DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-309-1
DATIM	Tue Mar 04 20:35:09 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	18



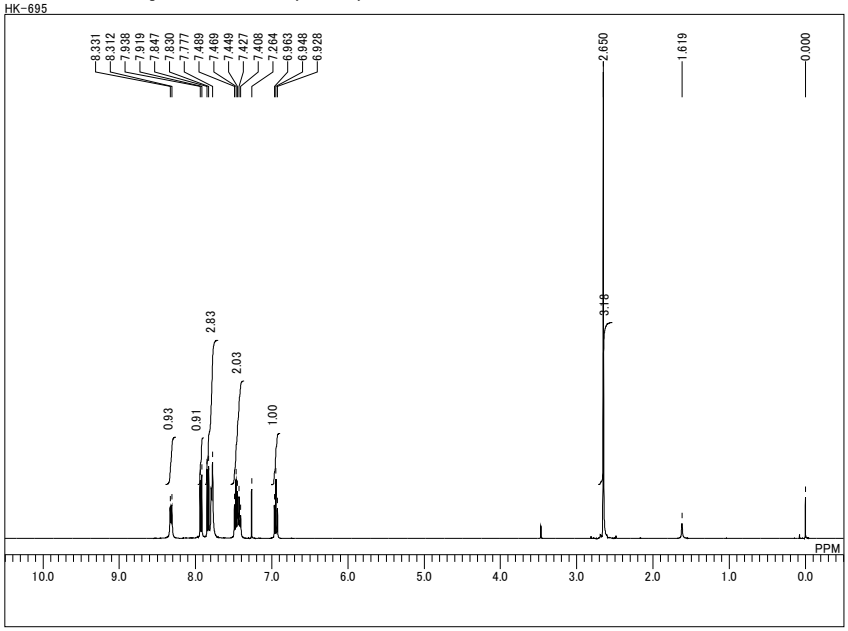
34

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥34-C.als

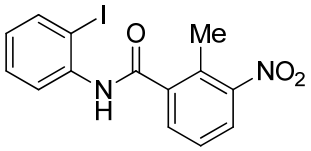


DFILE	C:¥Documents and Settings¥Ow
COMNT	
DATIM	Wed Mar 05 09:07:19 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥35-H.als
HK-695

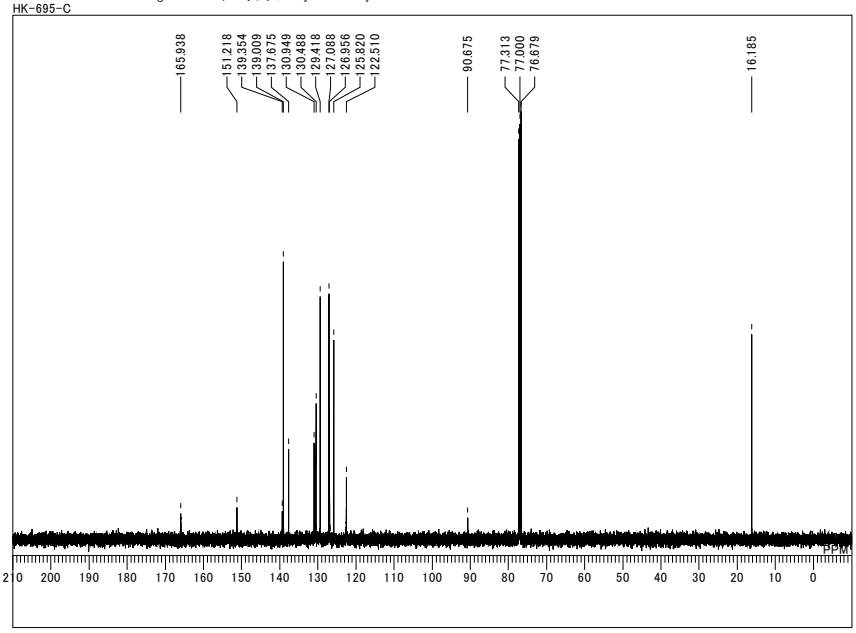


DFILE	C:¥Documents and Settings¥Ow
COMNT	HK-695
DATIM	Sat Sep 06 17:17:36 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	14

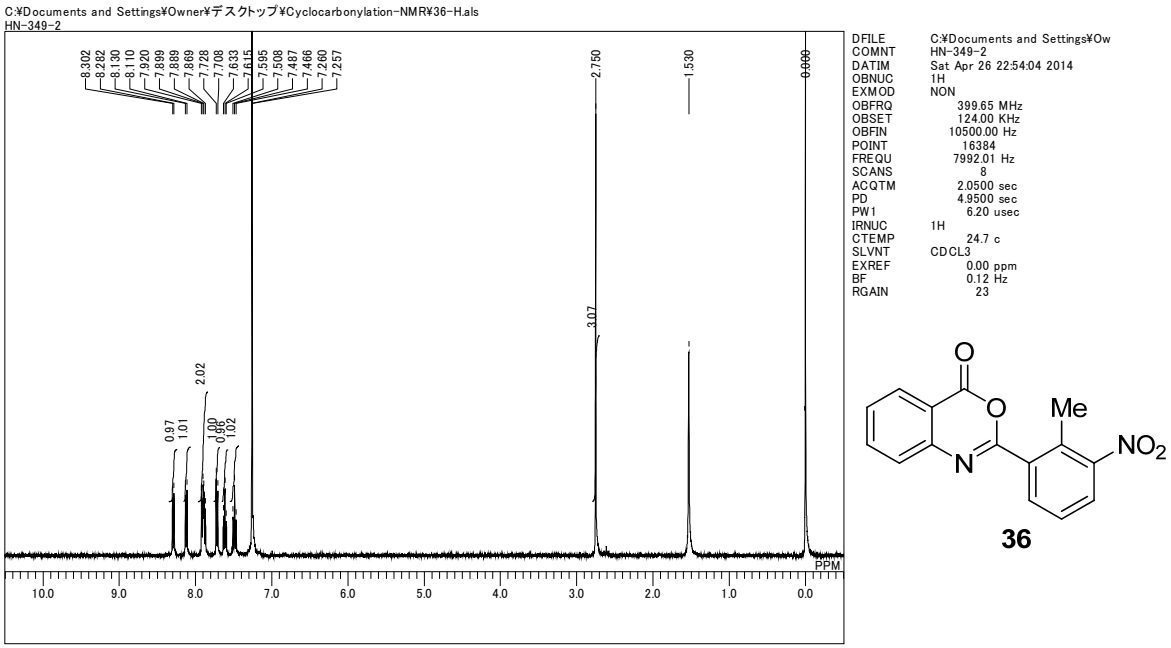


35

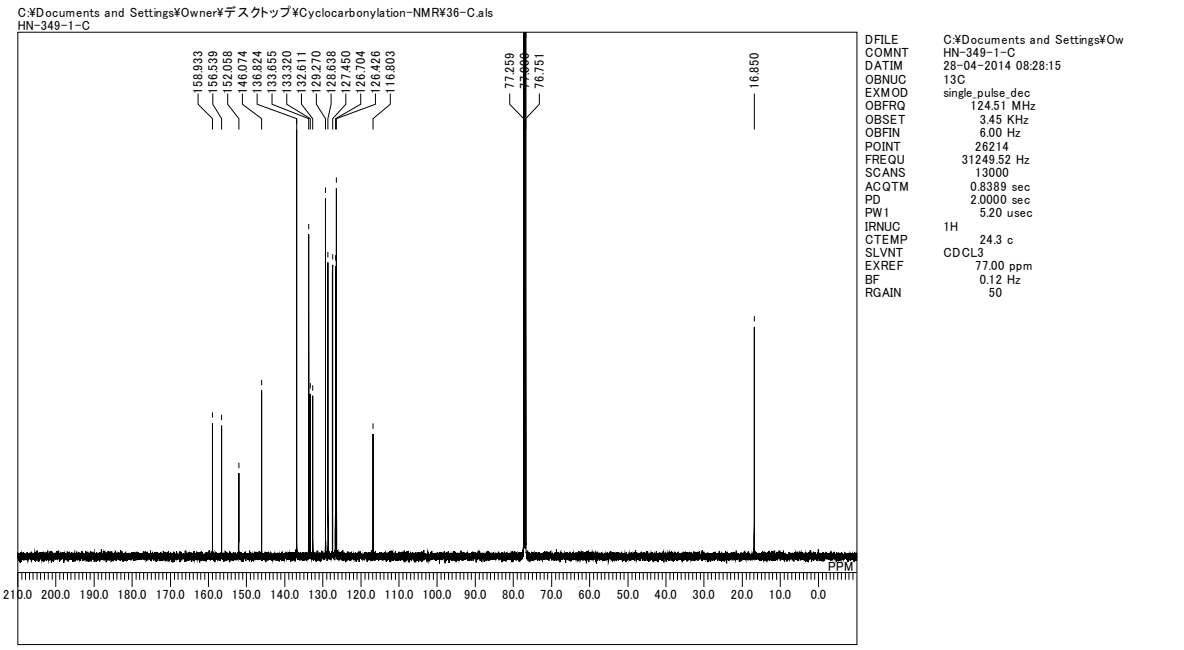
C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥35-C.als
HK-695-C



DFILE	C:¥Documents and Settings¥Ow
COMNT	HK-695-C
DATIM	Sat Sep 06 17:59:30 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	512
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27


C:¥Documents and Settings¥Owner¥デスクトップ¥Cyclocarbonylation-NMR¥36-H.als
HN-349-2
8.302
8.282
8.130
8.110
7.920
7.899
7.889
7.869
7.728
7.708
7.633
7.615
7.595
7.508
7.487
7.466
7.260
7.257
2.750
1.530
0.000
3.07
2.02
0.97
1.01
1.00
0.98
1.02
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
DFILE C:¥Documents and Settings¥Ow
COMNT HN-349-2
DATIM Sat Apr 26 22:54:04 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23
O
O
N
Me
NO2

36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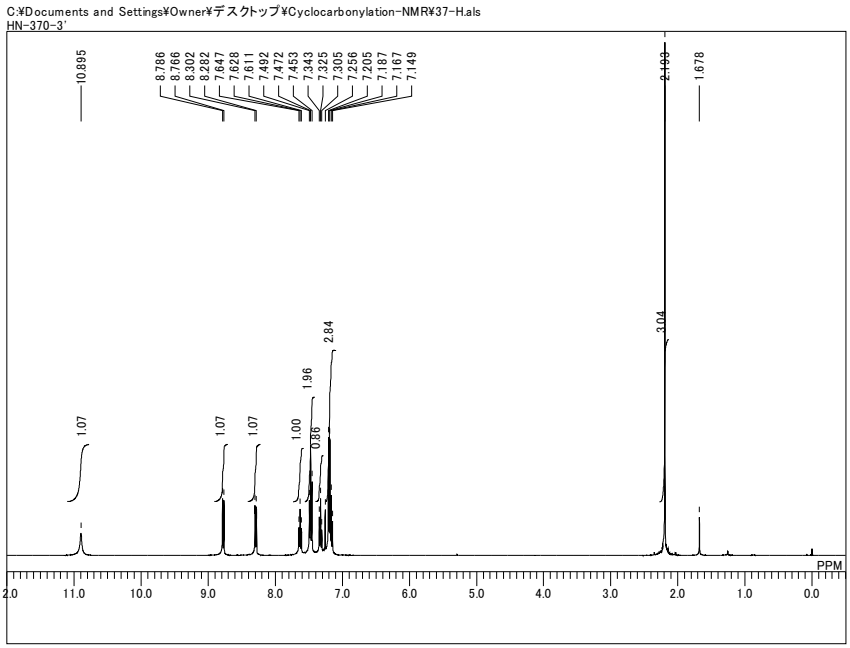
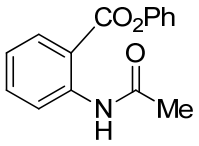
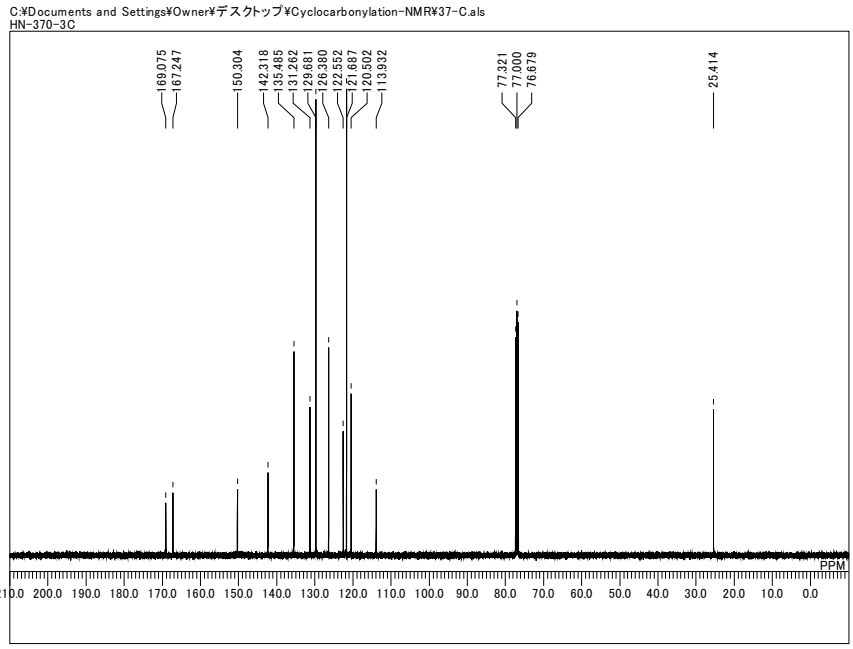
HN-349-1-C
158.933
156.539
152.058
146.074
136.824
133.655
133.320
132.611
129.270
128.638
127.450
126.704
126.426
116.803
77.259
77.008
76.751
16.850
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
DFILE C:¥Documents and Settings¥Ow
COMNT HN-349-1-C
DATIM 28-04-2014 08:28:15
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26214
FREQU 31249.52 Hz
SCANS 13000
ACQTM 0.8389 sec
PD 2.0000 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-370-3'
DATIM	Thu Nov 20 09:42:19 2014
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	4.9500 sec
PW1	6.20 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	15



37



DFILE	C:¥Documents and Settings¥Ow
COMNT	HN-370-3C
DATIM	Thu Nov 20 09:11:16 2014
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	1024
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27

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