Organic Syntheses

Preparation of 6*H*-Benzo[c]chromen-6-one

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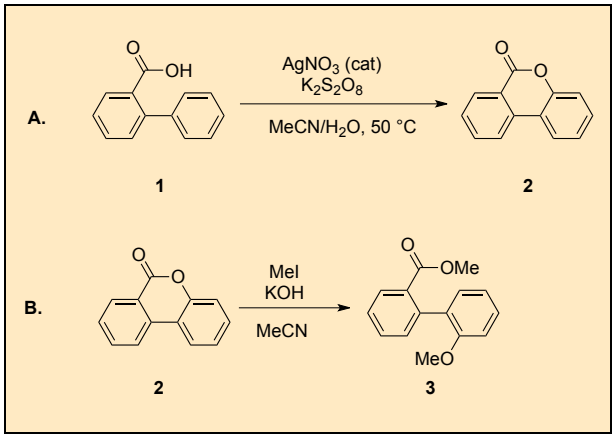
Checked by Evan R. Darzi and Neil K. Garg



Procedure (Note 1)

A. *6H-Benzo[c]chromen-6-one* (**2**). A 1000 mL, double-necked, round-bottomed flask is charged with a Teflon-coated magnetic stir bar (3 cm × 1 cm). To this flask are added biphenyl-2-carboxylic acid **1** (7.93 g, 40 mmol, 1 equiv) (Note 2), potassium peroxydisulfate (21.6 g, 80 mmol, 2 equiv) (Note 3), and silver nitrate (68 mg, 0.01 equiv) (Note 4), followed by water (200 mL) (Note 5) and acetonitrile (200 mL) (Note 6) under an air atmosphere. The flask is equipped with a water-cooling condenser (Note 7) (Figure 1) and a glass stopper. After stirring at 50 °C for 27 h (600 rpm) (Note 8), the reaction mixture is cooled to 23 °C (Figure 2). The reaction mixture is extracted with dichloromethane (3 x 200 mL) (Note 9)

DOI: 10.15227/orgsyn.95.0097

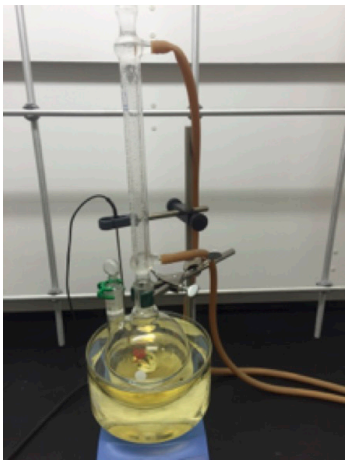
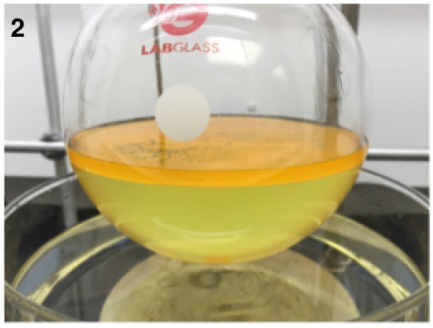


Figure 1. Reaction apparatus used in Step A



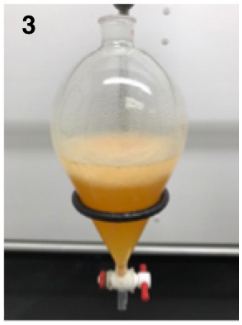


Figure 2. Reaction mixture of Step A after cooled down
Figure 3. Extraction in Step A with dichloromethane

(Figure 3) and the combined organic extracts are concentrated on a rotary evaporator under reduced pressure to obtain an orange solid (Figure 4). The residue is dissolved in ethyl acetate (50 mL) (Note 10), and the suspension is filtered through a short pad of silica gel (Note 11) (Figures 5 and 6) with the aid of ethyl acetate (100 mL). The organic solution is washed with 1 M NaOH solution (2 x 75 mL), followed by saturated NaCl solution (75 mL) (Figures 7, 8 and 9). The combined aqueous layers are back-extracted with ethyl acetate (100 mL). The combined organic extracts are dried over Na_2SO_4 (50 g) and filtered through cotton wool. The filtrate is concentrated on a

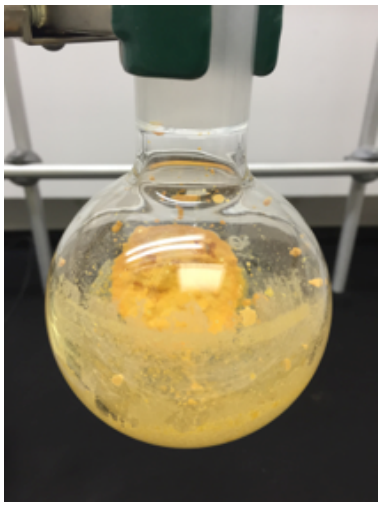
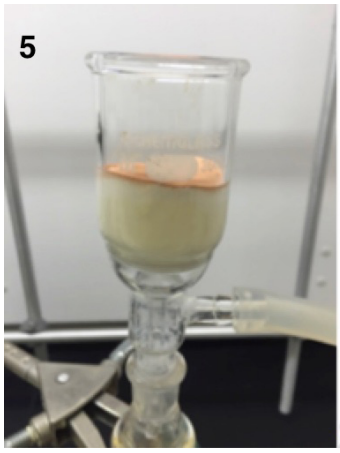


Figure 4. Crude product of Step A



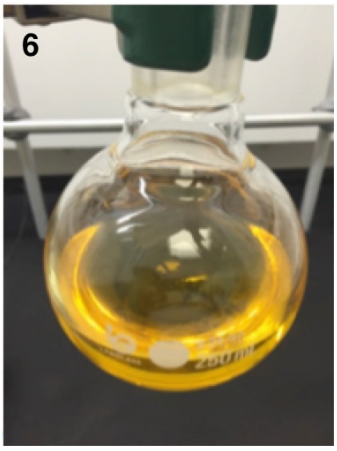


Figure 5. Experimental setup for filtration used in Step A

Figure 6. Filtrate of Step A

rotary evaporator under reduced pressure. The residual solid is dried in vacuo for 12 h at ambient temperature (23 °C) to yield 6*H*-benzo[c]chromen-6-one **2** as a pale-yellow solid (6.40 g, 82%) (Notes 12 and 13) (Figure 10).

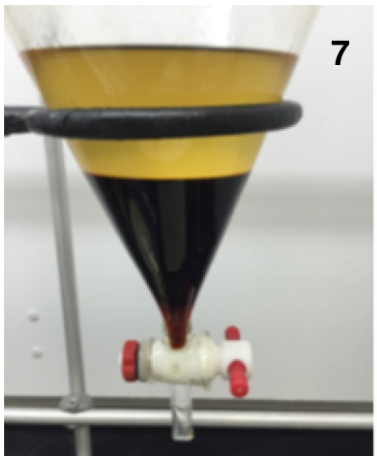


Figure 7. Filtrate of Step A washed with NaOH solution (first wash)

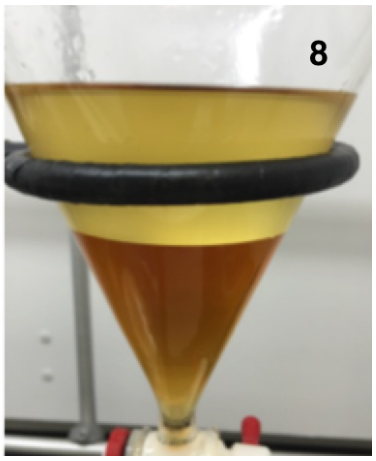


Figure 8. Filtrate of Step A washed with NaOH solution (second wash)

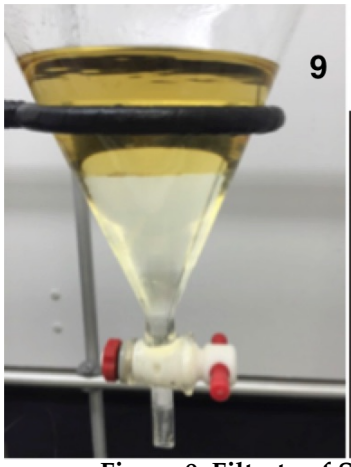


Figure 9. Filtrate of Step A washed with NaCl solution

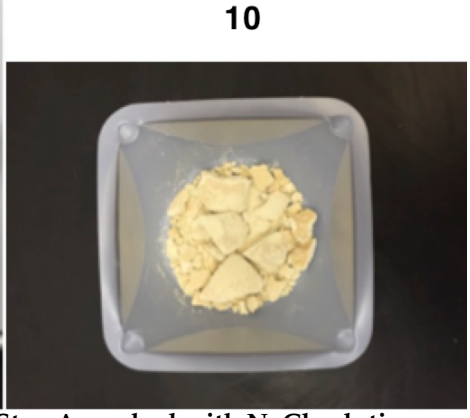
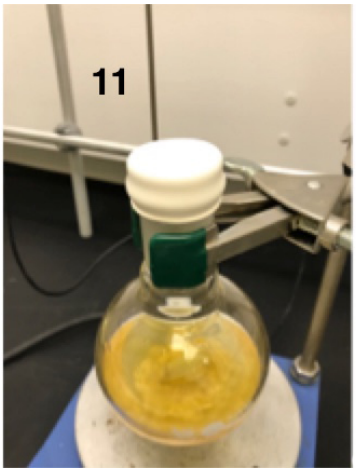


Figure 10. Product of Step A

B. *Methyl 2'-methoxy-[1,1'-biphenyl]-2-carboxylate* (**3**). A 500 mL, single-necked, round-bottomed flask is charged with a Teflon-coated magnetic stir bar (3 cm × 1 cm). Acetonitrile (60 mL), 6*H*-benzo[c]chromen-6-one **2** (5.89 g, 30 mmol, 1 equiv), and iodomethane (42.6 g, 18.7 mmol, 10 equiv) (Note 14) are added and stirred to form a solution. Potassium hydroxide (KOH)

pellets (9.9 g, 150 mmol, 5 equiv) (Note 15) are added to the flask under an atmosphere of air. The flask is sealed with a rubber septum (Figure 11), in which is inserted a needle that is connected to air (Note 16) (Figure 12). After stirring for 24 h (600 rpm) at 23 °C (Note 17) (Figure 13), the reaction mixture is concentrated on a rotary evaporator under reduced pressure to remove solvent and excess of iodomethane (Note 18) (Figure 14). Dichloromethane (50 mL) and deionized water (50 mL) are added to the residue (Figure 15). After the mixture is partitioned, the aqueous layer is extracted with dichloromethane (3 x 50 mL, and the combined organic extracts are washed with saturated NaCl solution (100 mL) (Figure 16). The aqueous phase is back extracted with dichloromethane (50 mL). The combined organic extracts are dried over Na_2SO_4 (50 g) and filtered through cotton wool. The filtrate is concentrated on a rotary evaporator under reduced pressure. The residual yellow oil is purified by column chromatography (Note 19) to yield methyl 2'-methoxy-[1,1'-biphenyl]-2-carboxylate **3** as a light yellow oil (6.50 g, 89%) (Notes 20 and 21) (Figure 17).



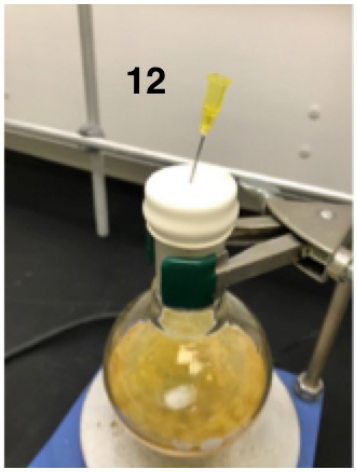
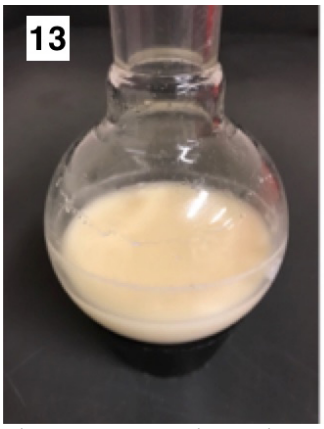


Figure 11. Reaction apparatus used in Step B

Figure 12. Reaction mixture of Step B during the reaction



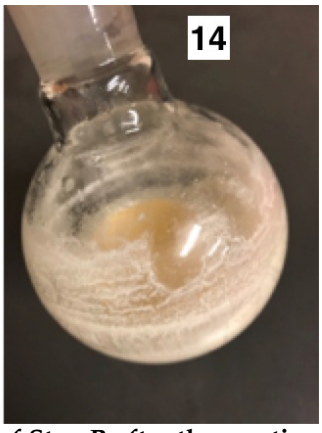
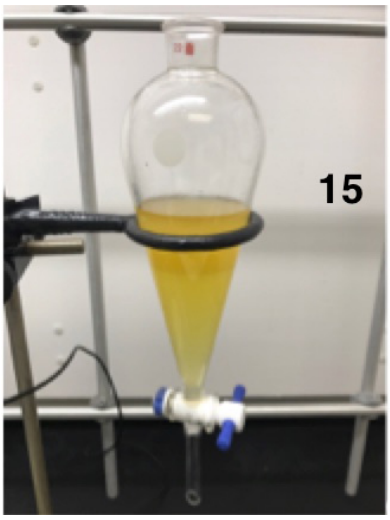


Figure 13. Reaction mixture of Step B after the reaction

Figure 14. Reaction mixture of Step B after removal of solvent and iodomethane



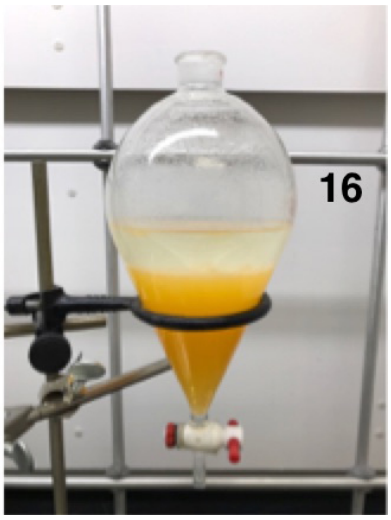


Figure 15. Reaction mixture of Step B washed with dichloromethane

Figure 16. Reaction mixture of Step B washed with NaCl solution

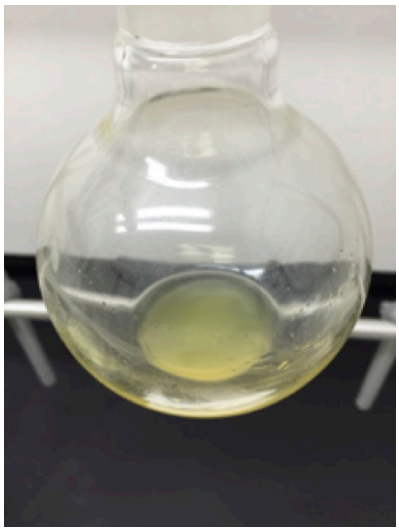


Figure 17. Product of Step B

Notes

1. Prior to performing each reaction, a thorough hazard analysis and risk assessment should be carried out with regard to each chemical substance and experimental operation on the scale planned and in the context of the laboratory where the procedures will be carried out. Guidelines for carrying out risk assessments and for analyzing the hazards associated with chemicals can be found in references such as Chapter 4 of "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at https://www.nap.edu/catalog/12654/prudent-practices-in-the-laboratory-handling-and-management-of-chemical). See also "Identifying and Evaluating Hazards in Research Laboratories" (American Chemical Society, 2015) which is available via the associated website "Hazard Assessment in Research Laboratories" at https://www.acs.org/content/acs/en/about/governance/committees/chemicalsafety/hazard-assessment.html. In the case of this procedure, the risk assessment should include (but not necessarily be limited to) an evaluation of the potential hazards associated with biphenyl-2-

carboxylic acid, potassium peroxydisulfate, silver nitrate, acetonitrile, dichloromethane,ethyl acetate, sodium hydroxide, sodium chloride, sodium sulfate, iodomethane, potassium hydroxide, hexanes, and silica gel.

2. [1,1'-Biphenyl]-2-carboxylic acid (98%) was purchased from Ark Pharm and used as received.
3. Potassium peroxydisulfate (99.9%) was purchased from Alfa Aesar and used as received.
4. Silver nitrate (99.9%) was purchased from Alfa Aesar and used as received.
5. Distilled water was used from tap distilled water.
6. Acetonitrile (99.9%, HPLC grade) was purchased from Fisher Scientific and used as received.
7. It is important to run the reaction under air. Sodium hydroxide (98%) was purchased from Alfa Aesar and used as received.
8. The temperature for the oil bath was set at 55 °C to ensure the reaction mixture's temperature is at 50 °C. It is important to maintain this reaction temperature. Reaction temperature was checked by immersing the tip of thermometer into the reaction mixture through the side neck of the flask. The reaction mixture was monitored by TLC analysis on silica using ethyl acetate/hexanes (1:3). R_f of **1** = 0.15, R_f of **2** = 0.4.

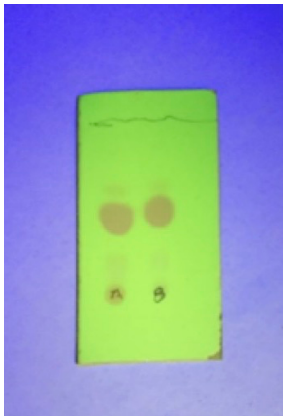


Figure 18: Crude product TLC after Step A

9. Dichloromethane (99.5%) was purchased from Sigma Aldrich and used as received.

10. Ethyl acetate (99.5%) was purchased from Sigma Aldrich and used as received.
11. The silica gel pad was pre-wetted by ethyl acetate (30 mL Filter Funnel, Büchner, fine Frit height ~3.5cm; diameter~3.5cm).
12. A second run on the identical scale provided 6.40 g (82%) of the product.
13. The product was dried under vacuum for 24 h to remove residue solvent. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.28–7.33 (m, 2H), 7.44 (ddd, *J* = 8.4, 7.1, 1.6 Hz, 1H), 7.54 (ddd, *J* = 8.0, 7.3, 1.1 Hz, 1H), 7.79 (ddd, *J* = 8.1, 7.3, 1.5 Hz, 1H), 7.98–8.02 (m, 1H), 8.04–8.08 (m, 1H), 8.35 (ddd, *J* = 8.0, 1.4, 0.60 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 117.8, 118.1, 121.3, 121.7, 122.8, 124.6, 128.9, 130.5, 130.6, 134.8, 134.9, 151.3, 161.2. IR (film): 3073, 1733, 1607, 1505, 1485, 1457 cm^{-1}. HRMS–APCI (*m*/*z*) calculated for $C_{13}H_9O_2$ [M + H] 197.05971, found 197.05817. Purity of **2** is 97.5%, which was determined by QNMR using ethylene carbonate as internal standard (15.6 mg ethylene carbonate and 34.7 mg **2**, NMR setting, d1 = 60 s, ns = 2).
14. Iodomethane (99%) was purchased from Spectrum Chemicals and used as received.
15. Potassium hydroxide (85%) was purchased from Alfa Aesar and used as received.
16. A needle connected to air was use to release the pressure from the flask. Inert atmosphere is not important for that step.
17. The reaction was stirred for 24 h. The submitters report that the reaction can be monitored by GC-MS.
18. Iodomethane is toxic, thus, its removal should be performed in a fume hood.
19. The crude product was diluted with dichloromethane (10 mL), loaded to a pre-wetted silica gel column, and purified by eluding with 1:3 ethyl acetate/hexanes. Column diameter ~3 cm, column height ~10 cm. Approximately 200 mL of the eluent was used, and the product was the only fraction collected.
20. A second reaction performed on the identical scale provided 6.40 (88%) of the same product.
21. The final product was dried under vacuum for 24 h to remove residual solvent. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 3.66 (s, 3H), 3.72 (s, 3H), 6.91 (dd, *J* = 8.2, 1.0 Hz, 1H), 7.05 (td, *J* = 7.5, 1.1 Hz, 1H), 7.25 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.32–7.36 (m, 2H), 7.40 (td, *J* = 7.6, 1.3 Hz, 1H), 7.55 (td, *J* = 7.6, 1.5 Hz, 1H), 7.87 (ddd, *J* = 7.8, 1.4, 0.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 51.8, 55.4, 110.2, 120.9, 127.3, 129.0, 129.5, 130.1, 130.7, 131.5,

131.7, 131.8, 138.9, 156.2, 168.8. IR (film): 3064, 2998, 1725, 1597, 1291, 1249 cm^{-1}. HRMS–APCI (*m*/*z*) calculated for $C_{15}H_{15}O_3$ [M + H] 243.10157, found 243.10011. Purity of **3** is 99.5%, which was determined by QNMR, using ethylene carbonate as internal standard (61.9 mg ethylene carbonate and 45.9 mg **3**, NMR setting, d1 = 30 s, ns = 4).

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

Discussion

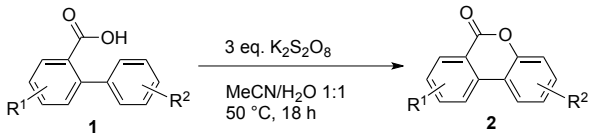
6*H*-Benzo[c]chromen-6-one motif presents in a variety of natural products, medicinal molecules, and functional organic compounds.[2] Due to the importance of this core, different synthetic methods were developed toward its synthesis. Existing methods include transition metal-catalyzed C–C bond formation[3] or intramolecular O–H/C–X coupling reaction.[4] Unfortunately, these methods suffer from limited availability of starting material and narrow reaction scope.

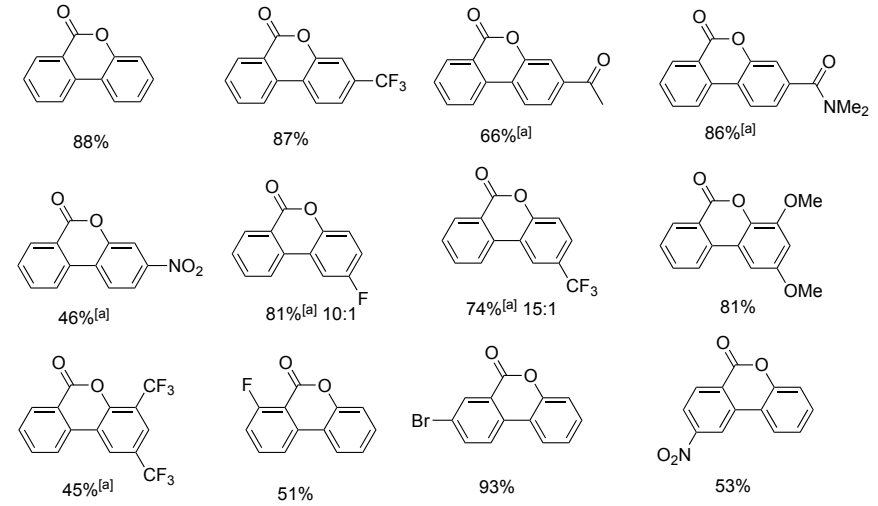
Biaryl-2-carboxylic acids are easily available materials and hence are ideal precursors for 6*H*-benzo[c]chromen-6-one synthesis. Thus, many research groups attempted to employ biaryl-2-carboxylic acids for synthesis of 6*H*-benzo[c]chromen-6-ones. The Thomson group reported a method to convert biaryl-2-carboxylic acid's silver salts into the corresponding 6*H*-benzo[c]chromen-6-ones via oxidation.[5] Similarly, Togo and Yokoyama described a *uv*-mediated procedure for synthesis of 6*H*-benzo[c]chromen-6-ones from unstable [bis(*o*-phenylphenylcarbonyloxy)iodo]benzene intermediates, which were produced from biaryl-2-carboxylic acids.[6] Another approach for the synthesis of 6*H*-benzo[c]chromen-6-ones from biaryl-2-carboxylic acids requires stoichiometric amounts of toxic Cr(VI) or Pb(IV) oxidants. Unfortunately, poor yields, narrow scope, and use of stoichiometric amounts of toxic metal reagents limit the utility of these transformations.[7] Recently, a Pd-catalyzed cyclization method was introduced by the Wang group,[8] where biaryl-2-carboxylic acids were converted into 6*H*-benzo[c]chromen-6-ones in good yields. Drawbacks of this method include employment of significant amount of expensive palladium catalyst, as well as the limitation of this method to electron-rich and -neutral substrates only. Later, the Martin group reported Cu-catalyzed remote C–H oxygenation reactions for synthesis of 6*H*-benzo[c]chromen-6-ones from biaryl-2-carboxylic acids. Independently, the Gevorgyan group reported both the Cu-catalyzed, as well as a metal-free method, for this transformation.[10] It deserves mentioning that the Cu-catalyzed method worked well with electron-rich and -neutral compounds, while less efficient for substrates bearing electron-withdrawing groups. In contrast, the metal-free O–H/C–H dehydrogenative coupling method[10] appeared to be insensitive to the electronics of the host aromatic ring.

Herein, we describe a general metal-free method for synthesis of 6*H*-benzo[c]chromen-6-ones from biaryl-2-carboxylic acids,[8] which features a

general scope with respect to electronic nature of the substrate, broad functional group compatibility, and mild reaction conditions (Table 1). In addition, employment of environmental friendly and relative cheap solvent provides an economically feasible approach for the synthesis of 6*H*-benzo[c]chromen-6-ones.[10] Moreover, it was demonstrated that the 6*H*-benzo[c]chromen-6-one could be converted easily into methyl 2'-methoxy-[1,1'-biphenyl]-2-carboxylate, thus representing a remote C–H oxygenation reaction in biaryl system.[8-10]

Table 1. Selected Scope of 6H-benzo[c]chromen-6-ones





[a] add 10 mol% $AgNO_3$

References

1. Department of Chemistry, University of Illinois at Chicago, 845 W. Taylor St., Chicago, Illinois 60607 (USA). We thank National Science

Foundation (CHE-1362541) for financial support of this work. Yang Wang and Yi Shi contributed equally to this manuscript.

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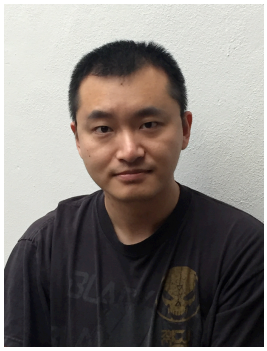
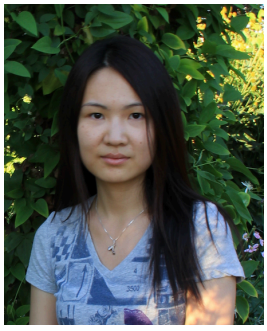
Appendix
Chemical Abstracts Nomenclature (Registry Number)

[1,1'-Biphenyl]-2-Carboxylic acid (947-84-2)
Potassium peroxydisulfate (7727-21-1)
Silver nitrate (7761-88-8)
6*H*-benzo[c]chromen-6-one (2005-10-9)
Potassium hydroxide (1310-58-3)
Iodomethane (74-88-4)



Yang Wang received his B.S. in 2008 from Wuhan University with Professor Qinghua Fan, and his M.S. in 2011 from The Chinese University of Hong Kong under the supervision of Professor Zuowei Xie. In 2011, he moved to Chicago and obtained his Ph.D. in 2017 with Professor Vladimir Gevorgyan. During his Ph.D. studies he was involved in development of novel transition metal-catalyzed, as well as metal-free, C–H functionalization methods. Currently, he is a postdoctoral fellow in the group of Prof. Tobin Marks at Northwestern University.



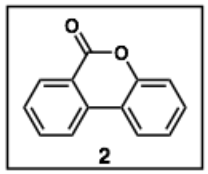
Dr. Yi Shi was born in Beijing in 1989. She received her B.S. degree from Peking University in 2011 under the supervision of Professor Jianbo Wang and Professor Yan Zhang. Then she received her Ph.D. degree from University of Illinois at Chicago in 2017 under the supervision of Professor Gevorgyan. Her graduate research focused on development of transition metal-catalyzed transformations of triazoles, metal carbine-involved coupling reactions and heterocycle synthesis. At present she is a postdoctoral fellow in the group of Prof. Fraser Stoddart at Northwestern University.

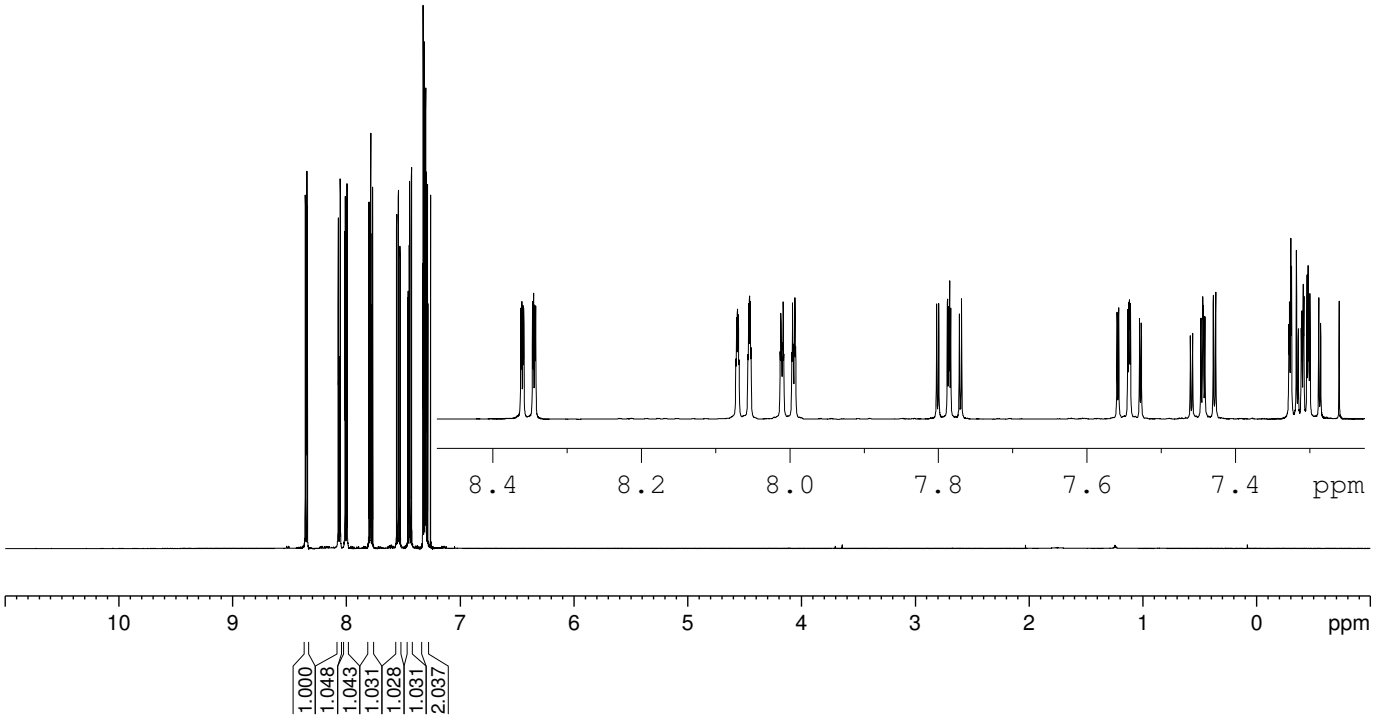
Vladimir Gevorgyan received his Ph.D. from the Latvian Institute of Organic Synthesis in 1984. After two years of postdoctoral research at Tohoku University (1992–1994), Japan, and a visiting professorship (1995) at CNR, Bologna, Italy, he joined the faculty at Tohoku University (Assistant Professor, 1996; Associate Professor, 1997–1999). He joined UIC as an Associate Professor in 1999 and was promoted to Professor in 2003. From 2012, he has been an LAS Distinguished Professor. His research interest is on the development of transition metal-catalyzed synthetic methodology for annulation and isomerization reactions, synthesis of carbo- and heterocycles, and C–H functionalization reactions.

Evan R. Darzi received his B.S. in Medicinal Biochemistry from Arizona State University in Tempe, AZ, where he performed undergraduate research under Professor Edward Skibo on the synthesis of extended amidines. He received his Ph.D. from the University of Oregon in Eugene, OR, under the guidance of Professor Ramesh Jasti. There he developed syntheses of highly strained [*n*]Cycloparaphenylenes and other "nanohoops". He is currently a NIH postdoctoral fellow in Professor Neil K. Garg's laboratory at the University of California, Los Angeles. His postdoctoral studies are focused on the development of strained intermediates in synthetic methodology.

6H-Benzo[c]chromen-6-one

8.362 8.361 8.359 8.358 8.346 8.345 8.343 8.342 8.071 8.070 8.069 8.055 8.054 8.053 8.012 8.009 7.996 7.993 7.802 7.799 7.788 7.786 7.785 7.783 7.771 7.769 7.559 7.557 7.545 7.543 7.542 7.541 7.529 7.526 7.446 7.444 7.443 7.441 7.429 7.426 7.328 7.327 7.325 7.324 7.318 7.310 7.308 7.308 7.303 7.302 7.301 7.299 7.287 7.285





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PROCNO 1

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PROBHD Z119248_0002 (
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SOLVENT CDCl3
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DS 0
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FIDRES 0.305176 Hz
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RG 19.06
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
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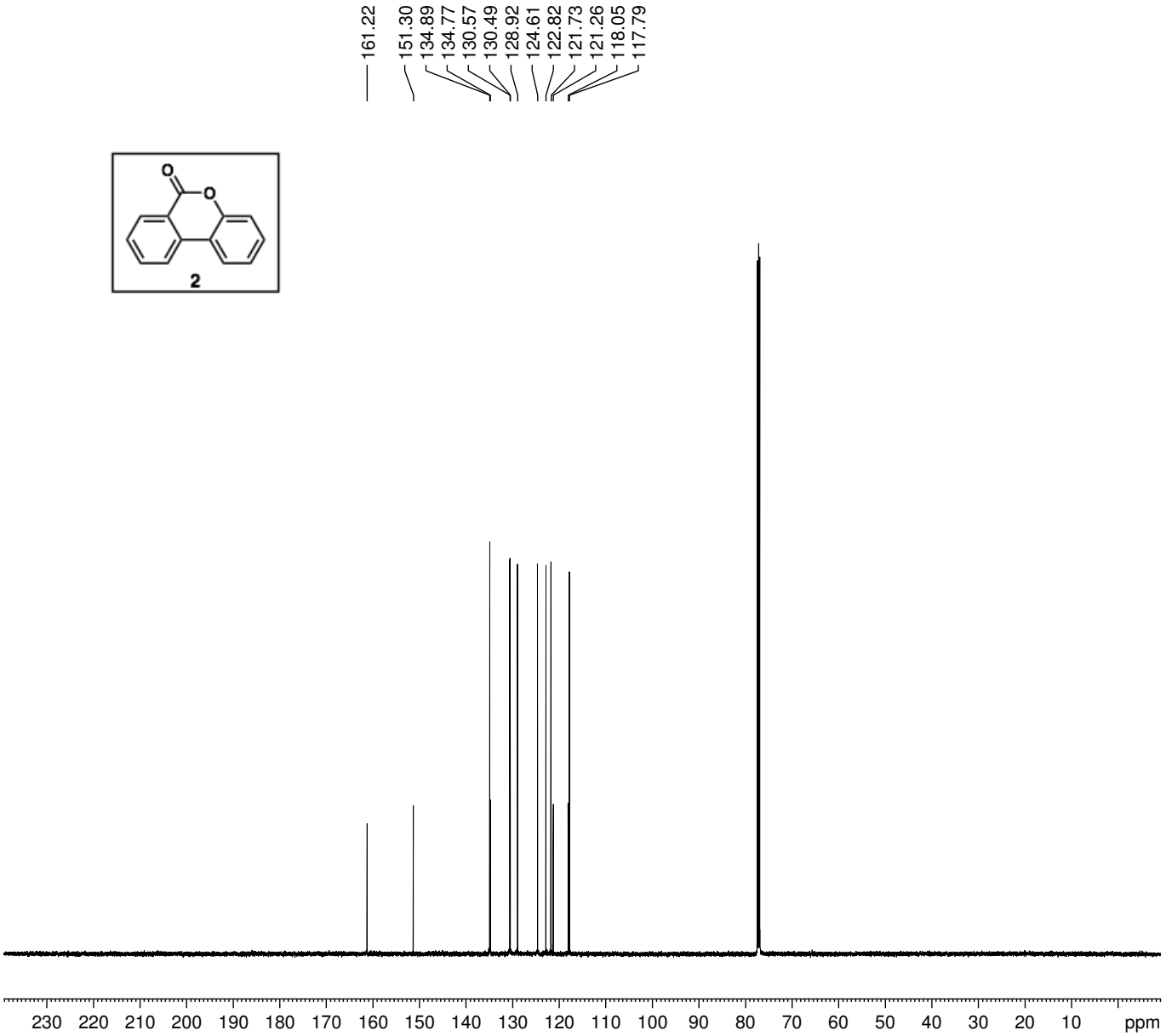
SF 500.1300119 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

6H-Benzo[c]chromen-6-one



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EXPNO 1
PROCNO 1

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SOLVENT CDCl3
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FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
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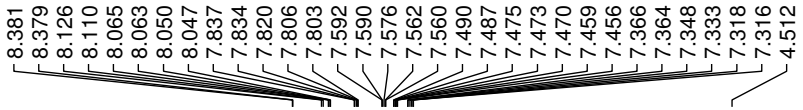
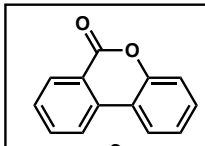
SF 125.7577803 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

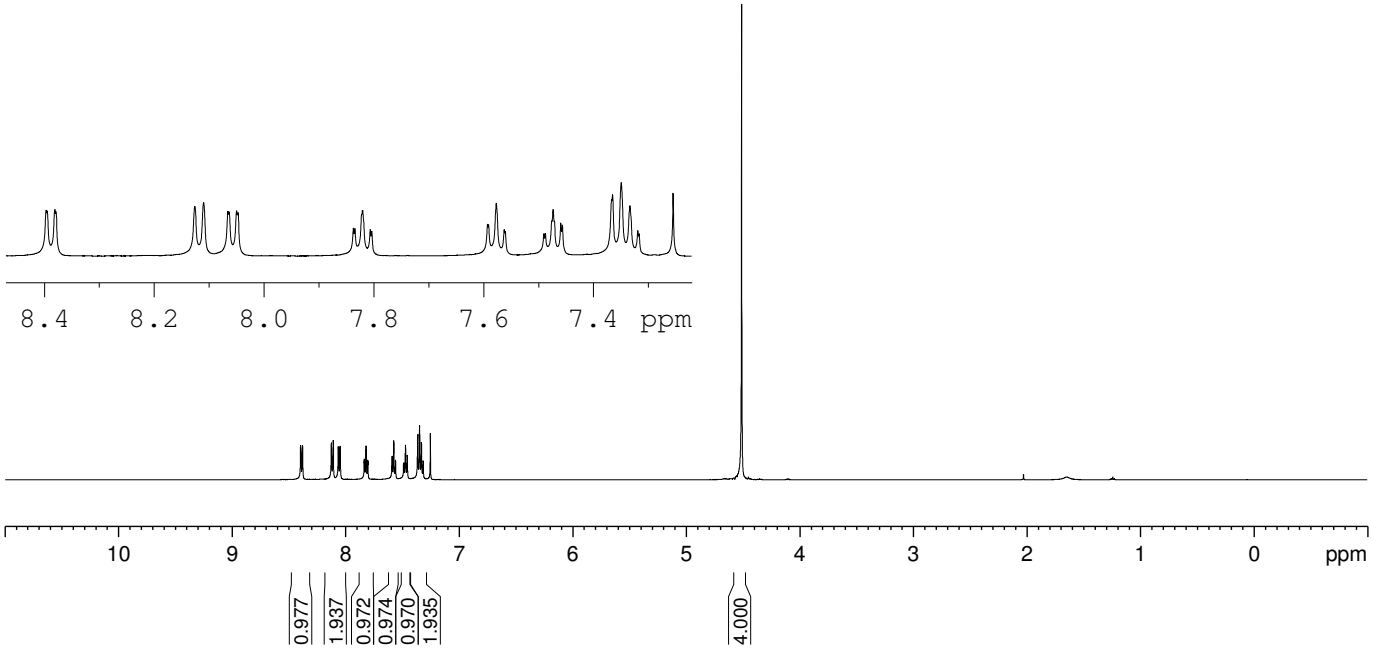
6H-Benzo[c]chromen-6-one





2

Note: Ethylene carbonate (δ 4.51 ppm) used to determine purity



Current Data Parameters
NAME ERD-2018-001-2-stan
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PROBHD Z119248_0002 (
PULPROG zg30
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SOLVENT CDCl3
NS 8
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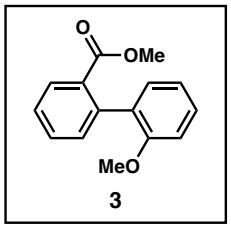
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

Methyl 2'-methoxy-[1,1'-biphen

7.546 7.534 7.531 7.416 7.413 7.401 7.398 7.386 7.383 7.357 7.354 7.350 7.349 7.347 7.346 7.342 7.341 7.339 7.337 7.335 7.334 7.332 7.331 7.326 7.322 7.266 7.262 7.251 7.248 7.247 7.061 7.059 7.046 7.044 7.031 7.029 6.917 6.915 6.900 6.898 3.722 3.656



3

Current Data Parameters
NAME ERD-2018-008-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
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SF 500.1300124 MHz
WDW no
SSB 0
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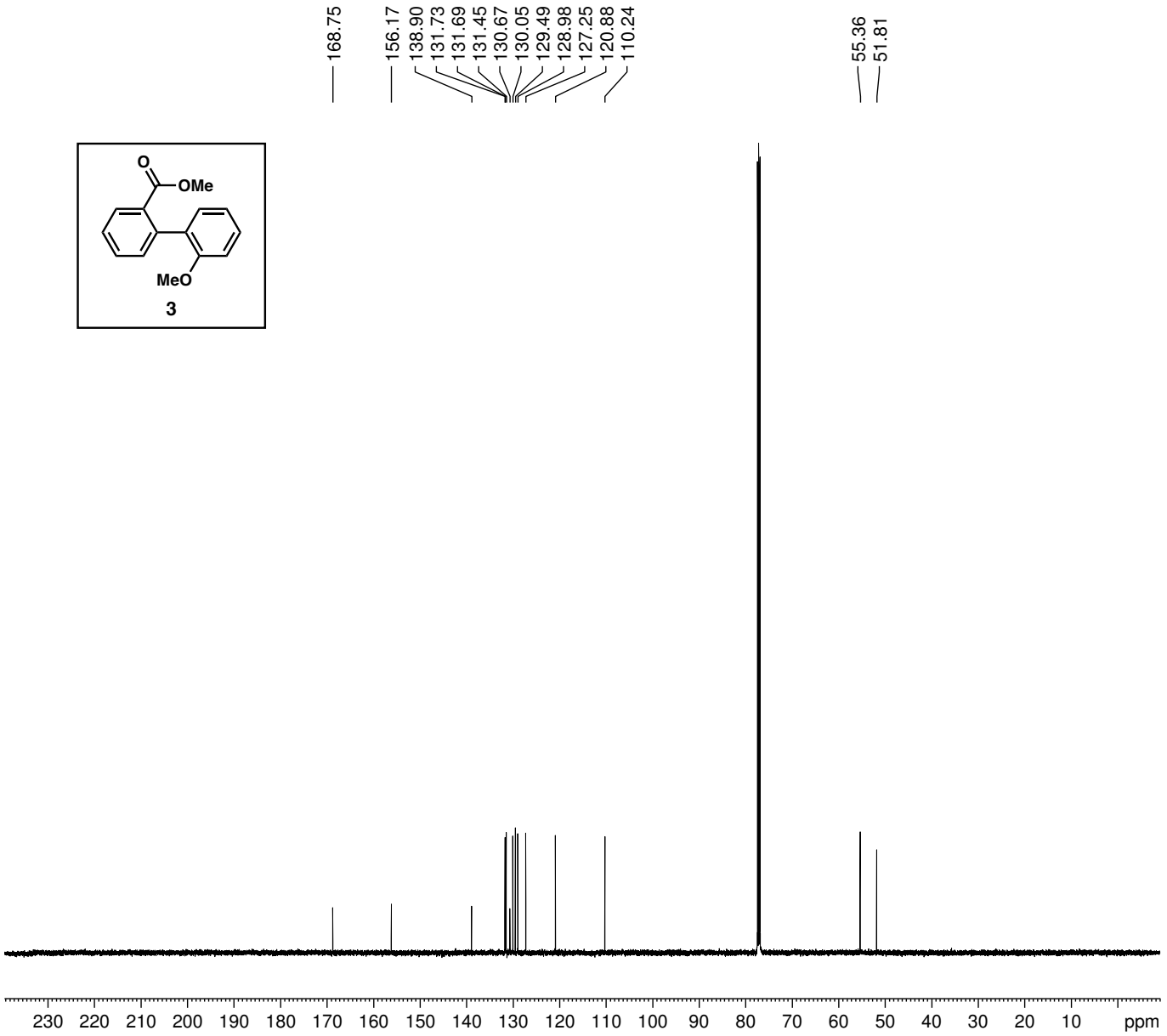
GB 0
PC 1.00

Methyl 2'-methoxy-[1,1'-biphen



Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 13.47 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
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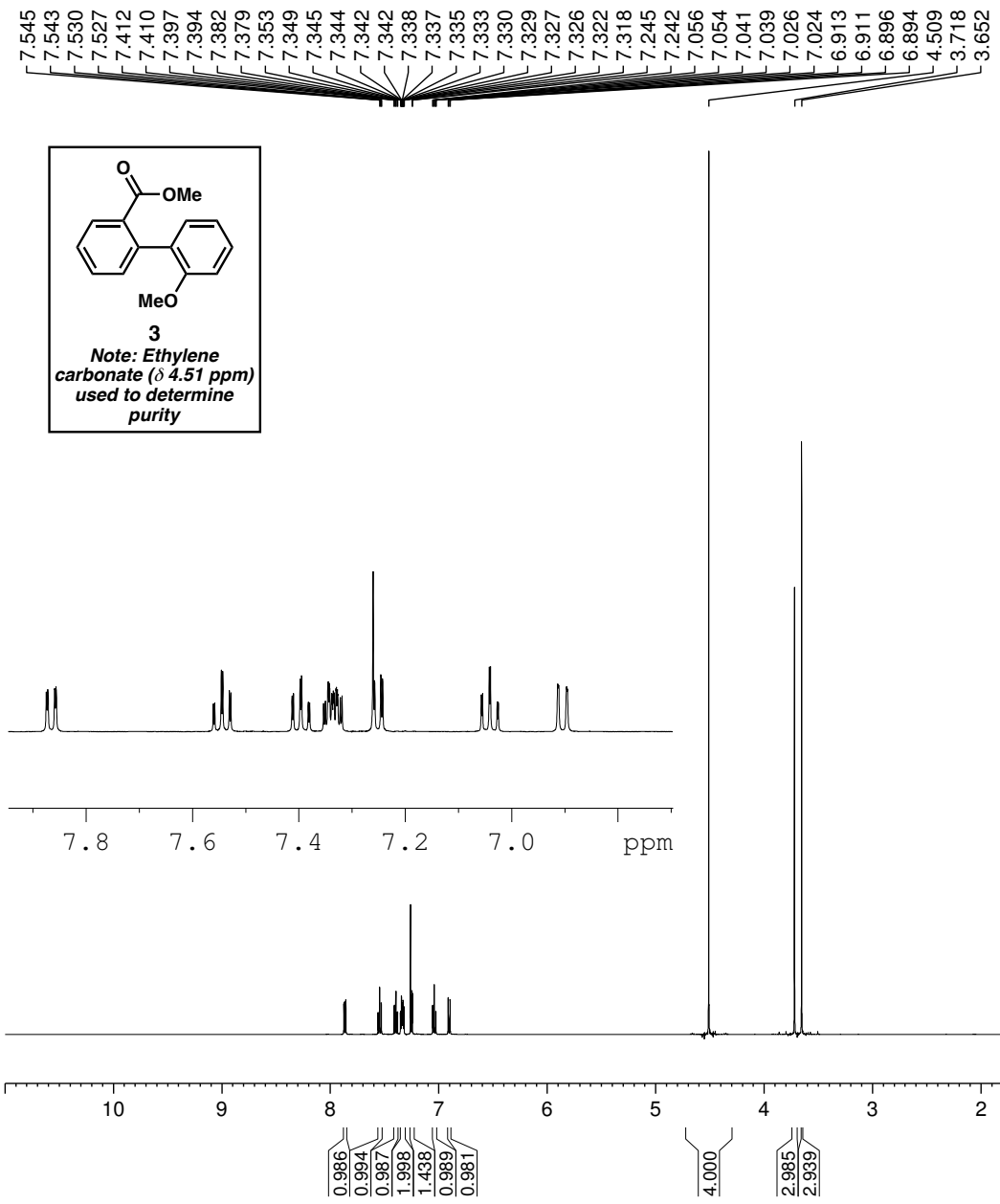
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577742 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Methyl 2'-methoxy-[1,1'-biphen



Current Data Parameters
NAME ERD-2018-008-2-refer
EXPNO 1
PROCNO 1

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PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
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FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300118 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00