Eight-Membered Ring Construction by [4+2+2] Annulation Involving β -Carbon Elimination

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Supporting Information

General. All reactions were carried out with standard Schlenk and glove box techniques under a nitrogen atmosphere. Preparative thin-layer chromatography was performed with silica gel 60 PF₂₅₄ (Merck). ¹H and ¹³C NMR spectra were recorded on a Varian Gemini 2000 (¹H at 300.07 Hz and ¹³C at 75.46 Hz) spectrometer or a JEOL JNM-A400 (¹H at 399.65 MHz and ¹³C at 100.40 MHz) spectrometer. Proton chemical shifts were referenced to residual solvent signals in CDCl₃ (δ 7.26). Carbon chemical shifts were referenced to the deuterated solvent signals in CDCl₃ (δ 77.00) and C₆D₆ (δ 128.00). High resolution mass spectra (EI) were recorded on a JOEL JMS-SX102A spectrometer. X-ray crystallographic data were measured on a Rigaku RAXIS-RAPID imaging plate diffractometer with Mo-K α (λ = 0.71069 Å). The structure was solved by Rigaku CrystalStructure™.

Materials. 1,6-Diynes **1a**,¹ **1b**,¹ **1c**,² **1d**,³ **1e**,² **1f**,⁴ **1g**,⁵ and 1,3-bis(2,6-diisopropylphenyl)-imidazol-2-ylidene ⁶ (IPr) were prepared according to the literature methods. Cyclobutanones **2a–e** were prepared by [2+2] cycloaddition of the corresponding olefins

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with dichloroketene and the subsequent dechlorination with zinc dust in acetic acid.⁷ Cyclobutanone **2f** was prepared from cyclopropylidenation of benzaldehyde⁸ and the subsequent *m*CPBA oxidation.⁹ Cyclobutanone **2g** was prepared according to the literature method.¹⁰ Toluene was distilled over sodium/benzophenone ketyl prior to use. All other commercially available chemical resources were used without further purifications.

General Procedure A (GP-A)

A toluene solution (0.80 mL) of Ni(cod)₂ (5.5 mg, 0.020 mmol), P(*n*-Bu)₃ (8.1 mg, 0.040 mmol), and cyclobutanone **1** (0.20 mmol) was stirred at 100 °C for a few minutes. To the solution was added dropwise a toluene solution (0.20 mL) of diyne **2** (0.30 mmol) via syringe over 2.5 h. After being stirred for a further 0.5 h, the reaction mixture was concentrated. The residue was purified by preparative thin-layer chromatography of silica gel to afford product **3**.

General Procedure B (GP-B)

A toluene solution (0.50 mL) of Ni(cod)₂ (5.5 mg, 0.020 mmol) and IPr (7.8 mg, 0.020 mmol) was stirred for at least 6 h and added to a mixture of cyclobutanone **1** (0.20 mmol) and diyne **2** (0.24 mmol) in toluene (1.50 mL). After being stirred for 1–3 h at room temperature, the reaction mixture was filtered through a pad of Florisil®. The filtrate was concentrated, and the residue was purified by preparative thin-layer chromatography of silica gel to afford product **3**.

Dimethyl 2,5,7-trimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3a). According to GP-B, **3a** (76.1 mg, 92%) was prepared from **1a** (56.7

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mg, 0.24 mmol) and **2a** (33.4 mg, 0.21 mmol). **3a**: ^1H NMR δ 1.18 (s, 3H), 1.41 (s, 3H), 1.96 (s, 3H), 2.31-2.42 (m, 3H), 2.96 (d, J = 15.3 Hz, 1H), 3.11-3.17 (m, 3H), 3.54 (d, J = 9.9 Hz, 1H), 3.72 (s, 3H), 3.75 (s, 3H), 7.20-7.34 (m, 5H); ^{13}C NMR (CDCl_3) δ 16.0, 23.0, 29.0, 38.9, 42.0, 47.4, 49.6, 50.4, 52.9, 53.0, 55.7, 125.4, 126.2, 128.2, 131.8, 135.3, 141.3, 147.1, 148.8, 171.6, 171.7, 201.7; HRMS calcd for $\text{C}_{24}\text{H}_{28}\text{O}_5$ 396.1937, found 396.1938.

Dimethyl 2,7-dimethyl-5,5-diphenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3b). According to GP-A, **3b** (76.6 mg, 83%) was prepared from **1a** (70.9 mg, 0.30 mmol) and **2b** (44.5 mg, 0.20 mmol). **3b**: ^1H NMR δ 1.16 (s, 3H), 1.85 (s, 3H), 2.71-2.77 (m, 2H), 3.03 (d, J = 15.6 Hz, 1H), 3.16-3.123 (m, 4H), 3.75 (s, 3H), 3.77 (s, 3H), 3.92 (d, J = 9.9 Hz, 1H), 7.10-7.30 (m, 10H); ^{13}C NMR (CDCl_3) δ 15.9, 23.0, 39.1, 41.9, 46.6, 47.8, 53.0, 55.7, 58.2, 126.3, 127.1, 127.9, 128.2, 132.1, 136.0, 140.6, 147.0, 147.2, 148.1, 171.6, 171.7, 199.9 [Some sp^2 carbon chemical shifts are observed as nonequivalent signals due to barrier to inversion of the cyclooctadienone ring]; HRMS calcd for $\text{C}_{29}\text{H}_{30}\text{O}_5$ 458.2093, found 458.2097. Anal. Calcd for $\text{C}_{29}\text{H}_{30}\text{O}_5$: C, 75.96; H, 6.59. Found: C, 75.72; H, 6.71.

Dimethyl 5,5-diethyl-2,7-dimethylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3c). According to GP-A, **3c** (50.2 mg, 70%) was prepared from **1a** (70.9 mg, 0.30 mmol) and **3c** (25.1 mg, 0.20 mmol). **3c**: ^1H NMR δ 0.82 (t, J = 7.1 Hz, 6H), 1.27 (br s, 4H), 1.77-2.08 (m, 3H), 1.87 (s, 3H), 1.95 (s, 3H), 2.54 (br s, 1H), 3.05-3.21 (m, 4H), 3.73 (s, 6H); ^{13}C NMR (CDCl_3) δ 7.4, 16.0, 24.4, 27.9, 39.2, 42.1, 44.7, 47.5, 51.3, 53.0,

55.7, 131.8, 135.4, 140.8, 148.4, 171.6, 201.7; HRMS calcd for C₂₁H₃₀O₅ 362.2093, found 362.2094.

Dimethyl 2,7-dimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3d). According to GP-B, **3d** (62.7 mg, 84%) was prepared from **1a** (56.7 mg, 0.24 mmol) and **3d** (28.7 mg, 0.20 mmol). **3d**: ¹H NMR δ 1.51 (br s, 3H), 1.93 (s, 3H), 2.38 (br s, 1H), 2.56 (br s, 2H), 3.10-3.24 (m, 5H), 3.65 (br s, 1H), 3.72 (s, 3H), 3.75 (s, 3H), 7.12-7.30 (m, 5H); ¹³C NMR (CDCl₃) δ 16.0, 23.5, 39.0, 41.9 [two carbons], 42.4, 50.4, 52.98, 53.04, 55.7, 126.6, 126.8, 128.3, 131.1, 135.1, 139.5, 143.4, 148.4, 171.6, 171.7, 203.5; HRMS calcd for C₂₃H₂₆O₅ 382.1780, found 382.1773.

Dimethyl 2,7-dimethyl-5-octylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3e). According to GP-B, **3e** (76.3 mg, 91%) was prepared from **1a** (56.7 mg, 0.24 mmol) and **2e** (36.4 mg, 0.20 mmol). **3e**: ¹H NMR δ 0.84 (t, *J* = 6.6 Hz, 3H), 1.23-1.40 (m, 14H), 1.85 (s, 3H), 1.91 (s, 3H), 1.96-2.85 (m, 5H), 3.11(dd, *J* = 21.9, 17.4 Hz, 4H), 3.71 (s, 6H); ¹³C NMR (CDCl₃) δ 14.0, 15.8, 22.6, 23.2, 27.4, 29.2, 29.5, 29.6, 31.8, 34.7, 38.9, 40.3, 41.8, 43.2, 46.2, 52.9 [two carbons], 55.7, 131.3, 134.4, 140.2, 148.2, 171.6 [two carbons], 203.5; HRMS calcd for C₂₅H₃₈O₅ 418.2719, found 418.2721.

Dimethyl 5-methyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3f). A toluene solution (0.60 mL) of Ni(cod)₂ (5.5 mg, 0.020 mmol), P(*c*-Hex)₃ (11.2 mg,

0.040 mmol), and **2a** (31.4 mg, 0.20 mmol) was stirred at 100 °C for a few minutes. To the solution was added dropwise a toluene solution (0.90 mL) of **1b** (125.0 mg, 0.60 mmol) via syringe over 2.5 h. After being stirred for 0.5 h, the reaction mixture was concentrated. The residue was purified by preparative thin-layer chromatography of silica gel to afford **3f** (49.0 mg, 68%). **3f**: ^1H NMR δ 1.38 (s, 3H), 2.26-2.33 (m, 1H), 2.61 (br s, 1H), 2.71 (dd, J = 12.8, 8.0 Hz, 1H), 2.98-3.36 (m, 5H), 3.73 (s, 3H), 3.74 (s, 3H), 6.00 (s, 1H), 6.08 (br t, 1H), 7.19-7.26 (m, 1H), 7.31-7.34 (m, 4H); ^{13}C NMR (CDCl_3) δ 30.0, 40.8, 41.7, 42.2, 49.4, 51.6, 53.0 [two carbons], 56.2, 125.2, 126.3, 126.8, 128.4, 133.4, 140.2, 146.4, 150.7, 171.1, 171.2, 202.2; HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_5$ 368.1624, found 368.1628.

Dimethyl 2,7-diethyl-5-methyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3g). According to GP-A, **3g** (74.1 mg, 85%) was prepared from **1c** (79.3 mg, 0.30 mmol) and **2a** (32.8 mg, 0.20 mmol). **3g**: ^1H NMR δ 0.50-0.75 (m, 3H), 0.80-1.20 (m, 3H), 1.41 (s, 3H), 1.74-1.93 (m, 1H), 2.18-2.56 (m, 6H), 3.06-3.29 (m, 4H), 3.52 (d, J = 10.8 Hz, 1H), 3.72 (s, 3H), 3.75 (s, 3H), 7.17-7.33 (m, 5H); ^{13}C NMR (C_6D_6) δ 11.5, 13.7, 24.2, 29.0, 29.6, 38.7, 41.5, 46.4, 48.0, 50.0, 52.4, 52.5, 55.9, 125.7, 126.4, 128.5, 135.5, 138.9, 146.7, 147.6, 147.7, 171.5, 171.6, 200.1; HRMS calcd for $\text{C}_{26}\text{H}_{32}\text{O}_5$ 424.2250, found 424.2253.

2,5,7-Trimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one (3h). According to GP-B, **3h** (50.7 mg, 87%) was prepared from **1d** (28.8 mg, 0.24 mmol) and **2a** (33.4 mg, 0.21 mmol). **3h**: ^1H NMR δ 1.17 (s, 3H), 1.43 (s, 3H), 1.70-1.78 (2H), 1.97 (s, 3H), 2.31-2.70 (m, 7H), 3.59 (d, J = 9.6 Hz, 1H), 7.19-7.23 (m, 1H), 7.30-7.35 (m, 4H); ^{13}C NMR (C_6D_6) δ

16.7, 22.0, 23.3, 29.4, 31.9, 35.4, 47.8, 49.9, 50.2, 125.8, 126.3, 128.4, 131.4, 138.9, 139.5, 148.1, 152.6, 200.8; HRMS calcd for C₂₀H₂₄O 280.1827, found 280.1827

10,10-Bis(methoxymethyl)-2,5,7-trimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one (3i). According to GP-B, **3i** (64.3 mg, 91%) was prepared from **1e** (50.0 mg, 0.24 mmol) and **2a** (31.0 mg, 0.19 mmol). **3i**: ¹H NMR δ 1.14 (s, 3H), 1.42 (s, 3H), 1.93 (s, 3H), 2.20-2.57 (m, 7H), 3.20-3.35 (m, 10H), 3.54 (d, J = 9.9 Hz, 1H), 7.18-7.34 (m, 5H); ¹³C NMR (CDCl₃) δ 16.1, 23.2, 29.0, 37.3, 41.0, 43.0, 47.6, 49.7, 50.5, 59.26, 59.32, 75.7, 76.1, 125.4, 126.1, 128.2, 131.8, 137.8, 140.6, 147.4, 152.6, 201.9; HRMS calcd for C₂₄H₃₂O₃ 368.2351, found 368.2354.

2,5,7-Trimethyl-5-phenyl-10-oxabicyclo[6.3.0]undeca-1,7-dien-3-one (3j). According to GP-A, but using P(*c*-Hex)₃ instead of P(*n*-Bu)₃, **3j** (35.0 mg, 61%) was prepared from **1f** (36.6 mg, 0.30 mmol) and **2a** (32.4 mg, 0.20 mmol). **3j**: ¹H NMR δ 1.18 (br s, 3H), 1.47 (s, 3H), 1.86 (s, 3H), 2.30-2.49 (m, 3H), 3.61 (br s, 1H), 4.51-4.66 (m, 4H), 7.20-7.37 (m, 5H); ¹³C NMR (CDCl₃) δ 15.5, 23.4, 29.2, 47.5, 49.9, 51.7, 71.9, 73.4, 125.3, 126.4, 128.4, 129.4, 134.9, 141.0, 146.7, 147.3, 202.2; HRMS calcd for C₁₉H₂₂O₂ 282.1620, found 282.1617.

10-(4-Toluenesulfonyl)-2,5,7-trimethyl-5-phenyl-10-azabicyclo[6.3.0]undeca-1,7-dien-3-one (3k). According to GP-B, **3k** (60.2 mg, 71%) was prepared from **1g** (66.1 mg, 0.24 mmol) and **2a** (31.2 mg, 0.19 mmol). **3k**: ¹H NMR δ 1.11 (s, 3H), 1.44 (s, 3H), 1.86 (s, 3H),

2.15-2.42 (m, 3H), 2.43 (s, 3H), 3.27 (br s, 1H), 3.99-4.28 (m, 4H), 7.20-7.35 (m, 7H), 7.72 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 16.0, 21.5, 23.4, 29.0, 47.2, 49.7, 51.1, 52.0, 54.1, 125.2, 126.5, 127.7, 128.4, 129.8, 131.4, 132.0, 133.1, 143.0, 144.0, 144.1, 146.4, 201.2; HRMS calcd for $\text{C}_{26}\text{H}_{29}\text{NO}_3\text{S}$ 435.1868, found 435.1868.

5,5-Diphenylbicyclo[6.4.0]dodeca-1,7-dien-3-one (3l). A toluene solution (0.80 mL) of $\text{Ni}(\text{cod})_2$ (11.0 mg, 0.040 mmol), $\text{P}(c\text{-Hex})_3$ (22.4 mg, 0.080 mmol), and **2b** (44.4 mg, 0.20 mmol) was stirred at 100 °C for a few minutes. To the solution was added dropwise a toluene solution (0.20 mL) of **1h** (63.6 mg, 0.60 mmol) via syringe over 2.5 h. After being stirred for 0.5 h, the reaction mixture was concentrated, and the residue was purified by preparative thin-layer chromatography of silica gel to afford **3l** (30.5 mg, 46%). **3l**: ^1H NMR δ 1.45-1.71 (m, 2H), 1.86-1.99 (m, 2H), 2.25-2.46 (m, 4H), 2.79 (dd, $J = 12.3, 6.5$ Hz, 1H), 2.91 (d, $J = 10.7$ Hz, 1H), 3.21 (dd, $J = 12.3, 9.9$ Hz, 1H), 3.79 (d, $J = 10.7$ Hz, 1H), 5.56 (ddd, $J = 9.9, 6.5, 1.5$ Hz, 1H), 5.91 (s, 1H), 7.15-7.32 (m, 10H); ^{13}C NMR (CDCl_3) δ 26.9, 27.2, 36.7, 37.7, 38.6, 49.5, 54.9, 126.1, 126.3, 126.9, 127.0, 127.2, 128.0, 128.2, 129.1, 141.4, 146.3, 146.7, 152.9, 200.7; HRMS calcd for $\text{C}_{24}\text{H}_{24}\text{O}$ 328.1827, found 328.1828.

Dimethyl 2,5-dimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3m) and dimethyl 5,7-dimethyl-5-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3n). According to GP-B, **3m** (32.4 mg, 43%) was prepared from **1i** (55.3 mg, 0.24 mmol) and **2a** (31.4 mg, 0.20 mmol). **3m**: ^1H NMR δ 1.37 (s, 3H),

1.92 (s, 3H), 2.17 (br s, 1H), 2.62 (dd, $J = 12.1, 8.0$ Hz, 2H), 2.98-3.33 (m, 5H), 3.72 (s, 3H), 3.74 (s, 3H), 5.97 (s, 1H), 7.19-7.35 (m, 5H); ^{13}C NMR δ 15.9, 30.0, 39.8, 41.6, 43.0, 48.7, 52.3, 53.0 [two carbons], 55.7, 125.3, 126.1, 128.2, 131.0, 133.1, 140.8, 146.0, 146.5, 171.3, 171.5, 202.3; HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_5$ 382.1780, found 382.1780.

According to GP-A, but using $\text{P}(c\text{-Hex})_3$ instead of $\text{P}(n\text{-Bu})_3$, **3m** (51.5 mg, 66%) and **3n** (13.6 mg, 18%) were prepared from **1i** (66.7mg, 0.30 mmol) and **2a** (32.5 mg, 0.20 mmol). **3n**: ^1H NMR δ 1.19 (br s, 3H), 1.42 (s, 3H), 2.34-2.60 (m, 3H), 2.90-3.30 (m, 4H), 3.46 (br s, 1H), 3.73 (s, 3H), 3.76 (s, 3H), 5.99 (s, 1H), 7.20-7.36 (m, 5H); ^{13}C NMR δ 23.1, 29.0, 38.8, 43.6, 48.1, 50.1, 50.3, 53.0, 53.1, 56.1, 125.3, 126.1, 126.4, 128.3, 134.2, 144.1, 146.8, 152.8, 171.3, 171.4, 202.5; HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_5$ 382.1780, found 382.1784.

Dimethyl 2,7-dimethyl-4-phenylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3o). According to GP-B, **3o** (68.1 mg, 91 %) was prepared from **1a** (56.7 mg, 0.24 mmol) and **2f** (28.5 mg, 0.19 mmol). **3o**: ^1H NMR δ 1.89 (s, 3H), 1.96 (s, 3H), 2.06-2.19 (m, 2H), 2.45-2.63 (m, 2H), 3.14 (d, $J = 15.6$ Hz, 1H), 3.19 (d, $J = 1.2$ Hz, 2H), 3.29 (d, $J = 15.3$ Hz, 1H), 3.77 (s, 3H), 3.79 (s, 3H), 4.46 (dd, $J = 13.1, 4.1$ Hz, 1H), 7.19-7.29 (m, 5H); ^{13}C NMR δ 16.3, 21.8, 34.5, 39.0, 41.0, 41.8, 51.5, 52.98, 53.04, 56.0, 126.7, 127.9, 129.1, 131.9, 134.0, 139.6, 140.4, 147.3, 171.5, 171.7, 206.1; HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_5$ 382.1780, found 382.1779.

Dimethyl 2,7-dimethyl-4-octylbicyclo[6.3.0]undeca-1,7-dien-3-one-10,10-dicarboxylate (3o). According to GP-B, **3o** (77.2 mg, 91 %, 37% ee) was prepared from **1a** (56.7 mg, 0.24 mmol) and **2g** (37.1 mg, 0.20 mmol, 48% ee). **3p**: ^1H NMR δ 0.83 (t, $J = 6.6$ Hz, 3H), 1.16-1.30 (m, 14H), 1.63-1.80 (m, 1H), 1.80-1.94 (m, 2H), 1.85 (s, 3H), 1.89 (s, 3H), 2.22-2.36

(m, 1H), 2.99-3.12 (m, 1H), 3.00 (d, $J = 15.3$ Hz, 1H), 3.10 (d, $J = 0.6$ Hz, 2H), 3.20 (d, $J = 15.0$ Hz, 1H), 3.71 (s, 3H), 3.72 (s, 3H); ^{13}C NMR δ 14.1, 16.0, 21.6, 22.6, 28.1, 29.2, 29.4, 29.8, 31.3, 31.8, 34.4, 38.9, 41.1, 41.6, 46.5, 52.9 [two carbons], 55.9, 131.5, 133.3, 140.2, 146.5, 171.6, 171.7, 209.3; HRMS calcd for $\text{C}_{25}\text{H}_{38}\text{O}_5$ 418.2719, found 418.2720.

¹³C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl₃
 Acquisition Temperature: 300.2 K
 GEMINI-300RB "zgpg30"

Relax. delay 1.158 sec
 Pulse program delay 0.000 sec
 Acq. time 0.842 sec
 Width 19000.0 Hz
 4032 repetitions
 OBSERVE C13, 75.4519686 MHz
 DECOUPLE H1, 300.0627335 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
 100% H1 decoupling
 Line broadening 1.0 Hz
 F2 size 32768
 Total time 601 hr, 21 min, 16 sec

STANDARD IN OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Reheat temperature
 GEMINI-300DB "varian2"
 Relax delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 28 repetitions
 OBSERVE XL 300.0673325 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

¹³C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl₃
Number of Sweeps: 1024
GEMINI-30DBB "variant2"

Relax. delay 1.158 sec
P1 0.000 sec
Acq. time 0.842 sec
Width 19000.0 Hz

4848 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE X1, 300.0687335 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

RF 1.000 sec

Line broadening 1.0 Hz

FT size 32768

Total time 601 hr, 21 min, 16 sec

STANDARD IN OBSERVE

Pulse Sequence: a2gpl
 Solvent: CDCl3
 Acquisition Temperature: 300.2 K
 GEMINI-300SB "vaxiam2"
 Relax delay 1.500 sec
 Relax delay 1.500 sec
 Relax delay 1.500 sec
 Acq. time 3.200 sec
 Width 5000.0 Hz
 44 repetitions
 OBSERVE H1, 300.067335 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Acquisition Temperature: 300 K
 GEMINI-300DB "variant2"
 Relax delay 1.156 sec
 Relax delay 45.000 sec
 Acq. time 0.842 sec
 Width 19000.0 Hz
 1680 Repetitions
 OBSERVE C13, 75.4519686 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 36 dB
 continuously on
 WALTZ16 modulated
 Phase Modulation
 Line broadening 1.0 Hz
 F2 size 32768
 Total time 601 hr, 21 min, 16 sec

13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "variant2"
 Relax delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 19000.0 Hz
 3696 repetitions
 OBSERVE C13, 75.4515698 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 36 dB
 Continuously on
 Water gated
 WALTZ16
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 601 hr, 21 min, 16 sec

STANDARD IN OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300DB "varian2"
Relax delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
76 repetitions
OBSERVE XL 300.067335 MHz
DATA PROCESSING
FT size 32768
Total time 8 min, 46 sec

13C QMHNVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Acquisition Temperature: 300.2 K
 GPCHEM-300RB "varian2"
 Relax delay 1.158 sec
 Relax delay 45.000 sec
 Acq. time 0.842 sec
 Width 19000.0 Hz
 1504 repetitions
 OBSERVE C13, 75.4519698 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
 DARRIN-2000
 Kinase broadening 1.0 Hz
 F1 size 32768
 Total time 601 hr, 21 min, 16 sec

STANDARD IN OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "varian2"
 Relax delay 1.500 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 28 repetitions
 OBSERVE NL 300.0672325 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

STANDARD IN OBSERVE

Pulse Sequence: s2pnl
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300SB "variant2"
 Relax delay 1.502 sec
 Pulse 27.7 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 40 repetitions
 OBSERVE H1, 300.0672325 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

¹³C OBSERVE

Pulse Sequence: a2pul

Solvent: Benzene
 200 MHz
 GEMINI-300B2 "Varian2"

Relax. delay 1.158 sec
 1792 Repetitions
 Width 19000.0 Hz

OBSERVE C13, 75.4519451 MHz

DECOUPLE H1, 300.0687605 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

FT size 32768

Total time 601 hr, 21 min, 16 sec

STANDARD 1H OBSERVE

Pulse Sequence: a2gpl

Solvent: CDCl3

Ambient Temperature

GBMKT-5000S "varian2"

Relax. delay 1.502 sec

Pulse 3.0000000000000000

Acq. time 3.200 sec

Width 5000.0 Hz

40 repetitions

OBSERVE H1, 300.0672125 MHz

DATA PROCESSING

FT size 32768

Total time 8 min, 46 sec

STANDARD 1H OBSERVE

Pulse Sequence: a2pul
Solvent: CDCl3
Acquisition Temperature: 300.2 K
Pulse Program: zgpg30

Relax. delay: 1.500 sec
Pulse: 45.0 degrees
Acq. time: 3.200 sec
Width: 5000.0 Hz
40 repetitions

OBSERVE: NL, 300.0672119 MHz

DATA PROCESSING

FT size 32768
Total time 8 min, 46 sec

STANDARD IN OBSERVE

Pulse Sequence: s2pol
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "vorian2"
 Relax delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 32 repetitions
 OBSERVE HL 300.0672322 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Acquisition Date: 20080801
 GEMINI-300B8 "zgpg30"
 Relax delay 1.158 sec
 Relax delay 45.000 sec
 Acq time 0.842 sec
 Width 19000.0 Hz
 10672 repetitions
 OBSERVE C13, 75.4519675 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
 DPMZ-16 modulated
 Phase 0.000000
 Line broadening 1.0 Hz
 FT size 32768
 Total time 601 hr, 21 min, 16 sec

13C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
GEMINI-300EB "varian2"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz

2544 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 36 dB
SOLVENT CDCl3
WALTZ-16
DATA PROCESSING
Line broadening 1.0 Hz
F2 size 32768
Total time 601 hr. 21 min. 16 sec

STANDARD IN OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient Temperature
 GEMINI-300B8 "varianb"
 Relax. delay 1.502 sec
 Pulse 27.7 degrees
 Acq. time 1.200 sec
 Width 5000.0 Hz
 28 repetitions
 OBSERVE H1, 300.0672322 MHz
 DATA PROCESSING
 FT size 32768
 Total time 6 min, 46 sec

E:\[unclear]\ashida-PhMe-CO2Me-MeH-1-COSY2.als

COMET Sat Jun 04 01:49:27
 DATIM COSY
 EXMOD 1H
 OBNUC 399.65 MHz
 OBFRQ 134136.81 Hz
 OFIN 1024
 POINT 512
 SAMPO 3037.67 Hz
 FREQU 1024
 CLPNT 256
 TODAT 3037.67 Hz
 CLFRQ 88
 SCANS 0.1686 sec
 ACQTM 1.0000 sec
 PD 10.20 usec
 PW1 10.20 usec
 PW2 13.00 usec
 PW3 400.0000 msec
 PT1 1.0000 msec
 PT2 20.0000 msec
 PT3

IRNUC 1H 399.65 MHz
 IRFRQ 0.00 KHz
 IRSET 0
 IRRNS 21.8 c
 CTENP CDCL3
 SLVNT 7.24 ppm
 EXREF 7.24
 CLEXR 18
 RGAIN

measurement no
 by FUJITA

C:\WINALPHA\Fujita\ashida-PhMe-CO2Me-MeH-1-NOESY2ft.als

COMET Sun Jun 05 01:50:41
 DATIM NOESY
 EXMOD 1H
 OENUC 1H 399.65 MHz
 OFFRQ 134136.81 Hz
 OFFIN 1024
 POINT 512
 SAMPO 3037.67 Hz
 FREQU 1024
 CLPNT 256
 TODAT 160
 CLFRQ 3037.67 Hz
 SCANS 0.1686 sec
 ACOTM 1.5000 sec
 PD 10.20 usec
 PW2 26.00 usec
 PW3 13.00 usec
 PI1 400.0000 msec
 PI2 1.0000 msec
 PI3 20.0000 msec
 LRNUC 1H 399.65 MHz
 LRFRQ 0.00 KHz
 LRSET 0
 LRNS 21.7 c
 CTENT CDCL3
 EXREF 7.24 ppm
 CLEXR 7.24
 RGAIN 18
 measurement no
 by FUJITA

Fri Jun 03 15:29:28 2001	
CONVIT	SINGL
EXMOD	SINGL
EXPON	SINGL
EXPON	13C
OFF	100.40 MHz
ON	135.500 MHz
POINT	327.68
FREQU	25974.03 Hz
ACQTM	1.2616 sec
PD	1.2384 sec
PD	2.348 sec
DUMM	8
PM1	5.60 usec
PM2	26.00 usec
PM3	13.00 usec
PI1	80.000 msec
PI2	20.000 msec
IRNUD	BOC1
IRNUC	1H
IRFRQ	399.65 MHz
IRSET	0.00 KHz
IRSW	5.11
IRSWT	22.2 c
SVLMT	QDCL3
EXREF	77.00 ppm
RGAIN	28
RESOL	0.79 Hz
TH5	22.2 c
PROBE	
TH5	
no	
measure	
by	
FLUITA

C:\WINALPHA\Fujita\ashida-PhMe-CO2Me-MeH-1-FGHMQCft.als

COMPT Thu Jun 02 23:21:29 ;
 DATIM
 EXMOD HMQC_FG
 OBRUC 1H
 OBRFQ 399.65 MHz
 OBSIN 134133.11 Hz
 POINT 1824
 SAMP0 512
 FREQU 3146.63 Hz
 CLENT 1824
 TOTAT 21276.60 Hz
 CLFRQ 256
 SCANS 48
 ACQTM 0.1627 sec
 PD 1.2000 sec
 FW1 6.40 usec
 FW2 26.00 usec
 FW3 20.20 usec
 F11 80.0000 msec
 F12 1.0000 msec
 F13 20.0000 msec
 IRNUC 13C
 IRFRQ 100.40 MHz
 IRSET 0.00 KHz
 IRRS 0
 CTMP 22.2 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 CLEXR 77.00
 RGAIN 35
 measurement no
 by FUJITA

C:\WINALPHA\Fujita\ashida-PhMe-CO2Me-MeH-1-FGHMBCft.als

COMPT Fri Jun 03 11:36:47 ;
 DATIM HMEC_FG
 EXNO 1H
 OBSRQ 399.65 MHz
 OBSIN 134153.11 Hz
 POINT 1024
 SAMP 512
 SAMP 3146.63 Hz
 FREQ 1024
 CLNT 236
 TOTAT 21276.60 Hz
 CLFRQ 120
 SCANS 0.1627 sec
 ACQIM 1.2000 sec
 PD 6.40 usec
 PW1 26.00 usec
 PW2 20.20 usec
 F11 60.0000 msec
 F12 1.0000 msec
 F13 20.0000 msec
 IRNUC 13C
 IRFRQ 100.40 MHz
 IRSET 0.00 KHz
 IRNS 0
 CTMP 21.6 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 CLEXR 77.00
 RGAIN 35
 measurement no
 by FUJITA

STANDARD IN OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Acquisition temperature

300.13 K

NAME: 3n

Relax. delay: 1.502 sec

Pulse delay: 0.000 sec

Acq. time: 0.000 sec

Width: 5000.0 Hz

28 repetitions

OBSERVE: H1, 300.0672325 MHz

DATA PROCESSING

FT size 32768

Total time 8 min, 46 sec

E:\[unclear]\ashida-PhMe-CO2Me-MeH-2-COSY.ALS

COMET Wed Jun 08 12:15:11:
 DATIM COSY
 EXMOD 1H
 OENUC 399.65 MHz
 OFRQ 134173.41 Hz
 OFIN 1024
 POINT 512
 SAMPO 3100.78 Hz
 FREQ 1024
 CLPNT 256
 TODAT 3100.78 Hz
 CLFRQ 16
 SCANS 0.1651 sec
 ACQTM 0.9147 sec
 PD 6.40 usec
 PW1 6.40 usec
 PW2 13.00 usec
 PW3 80.0000 msec
 PI1 1.0000 msec
 PI2 20.0000 msec
 PI3 399.65 MHz
 IRNUC 1H
 IRFRQ 0.00 KHz
 IRSET 21.6 c
 IRRNS 0
 CTENP 7.24 ppm
 SLVNT CDCL3 7.24
 EXREF CLEXR 17
 RGAIN
 measurement no
 by FUJITA

E:\00000001\ashida-PhMe-CO2Me-MeH-2-NOESY.ALS

COMET Wed Jun 08 16:39:41
 DATIM
 EXMOD NOESY
 OBNUC 1H
 OBFRQ 399.65 MHz
 OBFIN 134173.41 Hz
 POINT 1024
 SAMPO 512
 FREQU 3100.78 Hz
 CLENT 1024
 TODAT 256
 CLFRQ 3100.78 Hz
 SCANS 32
 ACQTM 0.1651 sec
 PD 1.4000 sec
 FW1 6.40 usec
 FW2 6.40 usec
 FW3 13.00 usec
 PI1 300.0000 msec
 PI2 1.0000 msec
 PI3 20.0000 msec
 IRRUC 1H
 IRFRQ 399.65 MHz
 IRSET 0.00 KHz
 IRRNS 0
 CTMP 22.0 c
 SLVNT CDCl3
 EXREF 7.24 ppm
 CLEXR 7.24
 RGAIN 17
 measurement no
 by FUJITA

C:\N1ALPHA\Fujita\Yashida-Phile-00216-Met-2-BCMT.als

C:\WINALPHA\Fujitalpha\ashida-PhMe-CO2Me-MeH-2-FGHMOCft.als

COMNT Tue Jun 07 18:36:31 ;
 DATIM HMQC_FG
 EXMOD 1H
 OBNUC 399.65 MHz
 OBPRQ 134163.61 Hz
 OBFIN 1024
 POINT 512
 SAMPO 3120.12 Hz
 FREQU 1024
 CLENT 256
 TODAT 19841.27 Hz
 CLFRQ 60
 SCANS 0.1641 sec
 ACQTM 1.2000 sec
 PD 6.40 usec
 PW1 26.00 usec
 PW2 20.20 usec
 PW3 80.0000 msec
 P11 1.0000 msec
 P12 20.0000 msec
 P13 100.40 MHz
 IRNUC 13C
 IRFRQ 0.00 KHz
 IRSET 22.3 c
 IRNS 0
 CTMP CDCL3
 SLVNT 7.24 ppm
 EXREF 77.00
 CLEXR 35
 RGAIN
 measurement no
 by FUJITA

E:\01a00d10\ashida-PhMe-CO2Me-MeH-2-FGHMBC.als

COMET Wed Jun 08 10:09:08 ;
 DATIM HMEC_FG
 EXMOD 1H
 OBNUC 399.65 MHz
 OBFRQ 134163.61 Hz
 OBFIN 1024
 POINT 512
 SAMPO 3120.12 Hz
 FREQU 1024
 CLENT 256
 TODAT 19841.27 Hz
 CLFRQ 152
 SCANS 0.1641 sec
 ACQTM 1.2000 sec
 PD 6.40 usec
 PW1 26.00 usec
 PW2 20.20 usec
 PW3 60.0000 msec
 P11 1.0000 msec
 P12 20.0000 msec
 P13 13C
 IRNUC 100.40 MHz
 IRFRQ 0.00 KHz
 IRSET 21.7 c
 IRNS 0
 CTEMP 7.24 ppm
 SLVNT CDCL3
 EXREF 77.00
 CLEXR 35
 RGAIN
 measurement no
 by FUJITA

¹³C OBSERVE

Pulse Sequence: e2pul
Solvent: CDCl₃
Number of Scans: 1000
SOLVENT-300B8 "varia2"

Relax. delay 1.158 sec
Pulse 16.000 sec
Pulse 16.000 sec
Width 19000.0 Hz
240 repetitions

OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
P1 16.000 sec
P2 16.000 sec
P3 16.000 sec
P4 16.000 sec
P5 16.000 sec
P6 16.000 sec
P7 16.000 sec
P8 16.000 sec
P9 16.000 sec
P10 16.000 sec
P11 16.000 sec
P12 16.000 sec
P13 16.000 sec
P14 16.000 sec
P15 16.000 sec
P16 16.000 sec
P17 16.000 sec
P18 16.000 sec
P19 16.000 sec
P20 16.000 sec
P21 16.000 sec
P22 16.000 sec
P23 16.000 sec
P24 16.000 sec
P25 16.000 sec
P26 16.000 sec
P27 16.000 sec
P28 16.000 sec
P29 16.000 sec
P30 16.000 sec
P31 16.000 sec
P32 16.000 sec
P33 16.000 sec
P34 16.000 sec
P35 16.000 sec
P36 16.000 sec
P37 16.000 sec
P38 16.000 sec
P39 16.000 sec
P40 16.000 sec
P41 16.000 sec
P42 16.000 sec
P43 16.000 sec
P44 16.000 sec
P45 16.000 sec
P46 16.000 sec
P47 16.000 sec
P48 16.000 sec
P49 16.000 sec
P50 16.000 sec
P51 16.000 sec
P52 16.000 sec
P53 16.000 sec
P54 16.000 sec
P55 16.000 sec
P56 16.000 sec
P57 16.000 sec
P58 16.000 sec
P59 16.000 sec
P60 16.000 sec
P61 16.000 sec
P62 16.000 sec
P63 16.000 sec
P64 16.000 sec
P65 16.000 sec
P66 16.000 sec
P67 16.000 sec
P68 16.000 sec
P69 16.000 sec
P70 16.000 sec
P71 16.000 sec
P72 16.000 sec
P73 16.000 sec
P74 16.000 sec
P75 16.000 sec
P76 16.000 sec
P77 16.000 sec
P78 16.000 sec
P79 16.000 sec
P80 16.000 sec
P81 16.000 sec
P82 16.000 sec
P83 16.000 sec
P84 16.000 sec
P85 16.000 sec
P86 16.000 sec
P87 16.000 sec
P88 16.000 sec
P89 16.000 sec
P90 16.000 sec
P91 16.000 sec
P92 16.000 sec
P93 16.000 sec
P94 16.000 sec
P95 16.000 sec
P96 16.000 sec
P97 16.000 sec
P98 16.000 sec
P99 16.000 sec
P100 16.000 sec
Total time 601 hr, 21 min, 16 sec

E:\1988\11\11\ashida-Phi-CO2Me-MeMe-ROEST.als

COMET Mon Jun 13 15:41:02 ;
 DATIM COSY
 EXMOD 1H
 OBNUC 399.65 MHz
 OBFRQ 134187.50 Hz
 OFFIN 1024
 POINT 512
 SAMPO 2784.74 Hz
 FRFQU 1024
 CLENT 256
 TOTAT 2784.74 Hz
 CLFRQ 16
 SCANS 0.1839 sec
 ACQTM 0.9000 sec
 PD 6.40 usec
 PW1 13.00 usec
 PW2 300.0000 msec
 P11 1.0000 msec
 P12 20.0000 msec
 P13 399.65 MHz
 IRNUC 1H
 IRFRQ 0.00 KHz
 IRSET 22.5 c
 IRRNS 0
 CTMP CDCL3
 SLVNT 7.24 ppm
 EXREF 7.24
 CLEXR 17
 RGAIN
 measurement no
 by FUJITA

C:\WINALPHA\Fujita\ashida-PhH-CO2Me-MeMe-NOESY.als

C:\N1\ALPHA\Fujita\Yashida-PhH-CO2Me-Melle-BCMF5ft.als

E:\1\12gdlj\4-clj\ashida-PhH-CO2Me-MeMe-FGHMQC.als

COMET Sat Jun 11 01:08:07 ;
 DATIM Sat Jun 11 01:08:07 ;
 EXMOD HMQC FG
 OBNUC 1H
 OBFREQ 399.65 MHz
 OBFIN 134185.31 Hz
 POINT 1024
 SAMPO 512
 FREQ 2768.55 Hz
 CLENT 2048
 TOTAT 21097.04 Hz
 CLFRQ 512
 SCANS 32
 ACQTM 0.1849 sec
 PD 1.2000 sec
 PW1 6.40 usec
 PW2 26.00 usec
 PW3 20.20 usec
 P11 80.0000 msec
 P12 1.0000 msec
 P13 20.0000 msec
 IRNUC 13C
 IRFRQ 100.40 MHz
 IRSET 0.00 KHz
 IRRNS 0
 CTEMP 23.1 C
 SLVNT CDCL3
 EXREF 7.24 ppm
 CLEXR 77.00
 RGAIN 35
 measurement no
 by FUJITA

E:\01\005411\01\01\ashida-PhH-CO2Me-MeMe-FGHMBC.a1s

COMET Sat Jun 11 19:27:54 ;
 DATIM HMEC_FG
 EXMOD 1H
 OBNUC 399.65 MHz
 OBFRQ 134185.31 Hz
 OBFIN 1024
 POINT 512
 SAMPO 2768.55 Hz
 FREQU 2048
 CLENT 21097.04 Hz
 TODAT 512
 CLFRQ 88
 SCANS 0.1849 sec
 ACQTM 1.2000 sec
 PD 6.40 usec
 PW1 26.00 usec
 PW2 20.20 usec
 PW3 60.0000 msec
 P11 1.0000 msec
 P12 20.0000 msec
 P13 13C
 IRNUC 100.40 MHz
 IRFRQ 0.00 KHz
 IRSET 22.8 C
 IRRS 0
 CTMP 7.24 ppm
 SLVNT CDCL3
 EXREF 77.00
 CLEXR 35
 RGAIN
 measurement no
 by FUJITA

13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300MHz "varian2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 19000.0 Hz
 F2 300.135360 MHz
 OBSERVE C13, 75.4519686 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 F2 300.135360 MHz
 PR size 32768
 Total time 601 hr, 21 min, 16 sec

DEPT			H ^a	H ^b	H ^c , H ^d	H ^e	H ^f	H ^g	H ^h	H ⁱ	H ^{arom}
			1.37	1.92	3.72, 3.74	2.17, 3.02	2.62	3.29	3.12	5.97	7.19-7.35
CH ₃ (sp ³)	C ^a	30.1	HMQC				HMBC				
	C ^b	15.9		HMQC							
	C ^c	53.0			HMQC						
	C ^d	53.0									
CH ₂ (sp ³)	C ^e	48.6	HMBC				HMBC				
	C ^f	39.8	HMBC				HMQC				
	C ^g	41.5							HMBC		
	C ^h	43.0									
CH (sp ²)	C ⁱ	131.0					HMBC		HMBC	HMQC	
	C ^{arom}	125.4 126.1 128.3									HMQC HMBC
C (sp ³)	C ^j	52.3	HMBC				HMBC				
	C ^k	55.7									
C (sp ²)	C ^l	133.1		HMBC			HMBC		HMBC		
	C ^m	140.8									
	C ⁿ	146.1									
	C ^{arom}	146.5	HMBC				HMBC				
	C ^o	202.3		HMBC							
	C ^p	171.4									
	C ^q	171.5							HMBC

DEPT			H ^a	H ^b	H ^c , H ^d	H ^e	H ^f	H ^g , H ^h	H ⁱ	H ^{arom}
			1.42	1.20		2.36, 3.47	2.48	2.90-3.30	5.99	7.20-7.36
CH ₃ (sp ³)	C ^a	29.0	HMQC							
	C ^b	23.1		HMQC			HMBC			
	C ^c	53.0			HMQC					
	C ^d	53.1								
CH ₂ (sp ³)	C ^e	50.1	HMBC			HMQC				
	C ^f	48.1	HMBC				HMQC			
	C ^g	43.6						HMQC	HMBC	
	C ^h	38.7						HMQC		
CH (sp ²)	C ⁱ	126.1							HMQC	
	C ^{arom}	125.3 126.3 126.4								HMQC HMBC
C (sp ³)	C ^j	50.3								
	C ^k	56.0						HMBC		
C (sp ²)	C ^l	134.2	HMBC	HMBC						
	C ^m	144.2								
	C ⁿ	152.8								
	C ^{arom}	146.8	HMBC				HMBC			HMBC
	C ^o	202.6								
	C ^p	171.37								
	C ^q	171.43						HMBC

DEPT			H ^a	H ^b	H ^c , H ^d	H ^e , H ^f	H ^g	H ^h	H ⁱ	H ^{arom}
CH ₃ (sp ³)	C ^a	16.3	1.89	1.96	3.77, 3.79	2.06-2.21, 2.43-2.63	3.19	3.10-3.32	4.47	7.19-7.29
	C ^b	21.7	HMBC	HMBC		HMBC				
	C ^c	52.97			HMBC					
	C ^d	53.02								
CH ₂ (sp ³)	C ^e	34.5		HMBC		HMBC			HMBC	
	C ^f	40.9								
	C ^g	41.8					HMBC	HMBC		
	C ^h	39.0					HMBC	HMBC		
CH (sp ³)	C ⁱ	51.4				HMBC			HMBC	
CH (sp ²)	C ^{arom}	126.8 128.0 129.1								HMBC HMBC
C (sp ³)	C ^j	55.9					HMBC			
C (sp ²)	C ^k	134.0		HMBC				HMBC		
	C ^l	140.5								
	C ^m	131.9	HMBC				HMBC			
	C ⁿ	147.3								
	C ^{arom}	139.6							HMBC	HMBC
	C ^o	206.2	HMBC				HMBC		HMBC	
	C ^p	171.6					HMBC			
	C ^q	171.8

Figure 1. Thermal ellipsoid diagram (30% probability) of **3b**. Hydrogen atoms are omitted for clarity.

Figure 2. Another View of **3b** (30% probability). Some atoms are omitted.

Table 1. Crystallographic Data for **3b**

crystal color, habit	colorless, cube
crystal size (mm)	0.10 × 0.10 × 0.10
empirical formula	C ₂₉ H ₃₀ O ₅
formula weight	458.55
radiation, λ (Å)	0.71069
T (°C)	296
crystal system	triclinic
space group	<i>P</i> $\bar{1}$ (No. 2)
<i>a</i> (Å)	8.928(2)
<i>b</i> (Å)	11.884(3)
<i>c</i> (Å)	12.955(3)
α (deg)	95.42(1)
β (deg)	109.67(1)
γ (deg)	107.105(6)
<i>V</i> (Å ³)	1208.1(5)
<i>Z</i>	3
<i>D</i> _{calcd} (g cm ⁻³)	1.891
<i>F</i> ₀₀₀	732.00
no. of reflections	total: 10458, unique: 5328
<i>R</i> _{int}	0.059
<i>R</i> , <i>R</i> _w	0.157, 0.191
GOF	3.62

Table 2. Selected Bond Distances (Å) and Angles (deg)

C1–C2	1.51(1)
C2–C3	1.52(1)
C3–C4	1.56(1)
C4–C5	1.52(1)
C5–C6	1.35(1)
C6–C7	1.48(1)
C7–C8	1.35(1)
C8–C1	1.51(1)
C1–O1	1.23(1)
C6–C9	1.52(1)
C9–C10	1.51(1)
C10–C11	1.54(1)
C11–C7	1.55(1)
C1–C2–C3	114.0(9)
C2–C3–C4	103.0(8)
C3–C4–C5	116.2(8)
C4–C5–C6	122.3(9)
C5–C6–C7	127.7(9)
C6–C7–C8	136.1(1)
C7–C8–C1	123.6(1)
C8–C1–C2	121.8(9)