Supplementary Material for

**The First Examples of a Meta-Benzannulation from the Reaction of
Fischer Carbene Complexes with Alkynes**

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Spectral Data for All New Compounds and X-ray Data for Compound 10b.

SChapter Preparation of Carbene Complexes 6a – 6d.

Entry	n	Series	% Yield 26	% Yield 6
1	6	a	54	44
2	10	b	47	43
3	13	c	46	48
4	16	d	45	41

Preparation of 1,13-Tetradecadiyne 25b

The title compound was prepared according to literature procedure.ⁱ White solid. R_f (hexanes) 0.27; ^1H NMR (CDCl_3) δ 1.28 (br s, 8H), 1.38 (m, 4H), 1.52 (m, 4H), 1.94 (t, 2H, $J = 2.6$ Hz), 2.18 (td, 4H, $J = 7.1, 2.6$ Hz); ^{13}C NMR (CDCl_3) δ 18.38, 28.46, 28.72, 29.05, 29.40, 68.03, 84.79; IR (thin film) 3288 s, 2112 w cm^{-1} .

Preparation of 1,13-Diiodotridecaneⁱⁱ

To a suspension of LAH (1.490 g, 37.30 mmol) in 50 mL of THF at 0 °C was added dropwise 1,11-undecanedicarboxylic acid (3.239 g, 12.46 mmol) in 50 mL of THF. The addition was complete in 15 min. Another 100 mL of THF was added to dilute the resultant suspension, and the reaction was heated to 78 °C for 4 h, then was cooled to rt. Water (3 mL), NaOH (2 N, 3 mL) and water (10 mL) were added in that order. The mixture was filtered through celite and the residue was washed with ether. The organic layer was washed with brine, dried with MgSO_4 and then concentrated to give 1,13-tridecanediol. The crude product was pure enough to be used in the next step.

P_2O_5 (2.5 g) was dissolved in H_3PO_4 (85%, 5 mL). The freshly prepared 97% H_3PO_4 (109 mmol) was cooled to rt and added to a mixture of KI (12 g, 72 mmol) and the crude dialcohol prepared above. The reaction mixture was heated to 115 °C for 4 h, cooled to rt, and diluted with ether and water. The organic layer was separated, washed with $\text{Na}_2\text{S}_2\text{O}_3$, dried with MgSO_4 and concentrated. Flash column chromatography provided the title product as a white solid (5.04 g, 11.56 mmol, 93% over 2 steps). R_f (hexanes) 0.45; ^1H NMR (CDCl_3) δ 1.26 (br s, 14H), 1.38 (m, 4H), 1.82 (m, 4H), 3.19 (t, 4H, $J = 7.0$ Hz); ^{13}C NMR (CDCl_3) δ 7.39, 28.52, 29.39, 29.49,

29.52, 30.49, 33.54; IR (thin film) 2921 s, 1161 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 436 (M^+ , 11), 309 (43), 197 (23), 183 (29), 169 (21), 155 (26), 141 (4), 57 (100). Anal Calcd for $\text{C}_{13}\text{H}_{26}\text{I}_2$: C 35.80, H 6.01; Found: C 35.81, H 6.07.

Preparation of 1,16-Diiodohexadecane

The title compound was prepared in the same manner, obtained as a white solid in 54% yield from 1,16-hexadecanediol. R_f (hexanes) 0.43; ^1H NMR (CDCl_3) δ 1.26 (m, 20H), 1.38 (m, 4H), 1.82 (m, 4H), 3.19 (t, 4H, $J = 7.0$ Hz); ^{13}C NMR (CDCl_3) δ 7.25, 28.54, 29.42, 29.54, 29.60, 29.62, 30.52, 33.60; IR (thin film) 2915 s, 1162 w cm^{-1} ; MS (EI) m/z (% rel. intensity) 478 (M^+ , 10), 351 (24), 211 (12), 197 (13), 183 (16), 169 (11), 155 (14), 141 (4), 57 (100); Anal Calcd for $\text{C}_{16}\text{H}_{32}\text{I}_2$: C 40.18, H 6.74; Found: C 40.10, H 6.95.

Preparation of 1,16-Heptadecadiyne 25c

$n\text{BuLi}$ (15.5 mmol) was added dropwise to trimethylsilylacetylene (newly distilled, 2.5 mL, 17.7 mmol) in 25 mL of THF at -78°C . The mixture was stirred at -78°C for 30 min and warmed to 0°C for 50 min. The anion was added *via* syringe to 1,13-diiodotridecane (1.628 g, 3.73 mmol) in 25 mL of THF at rt followed by the addition of 12 mL of HMPA. The mixture was stirred at rt overnight and quenched with HCl (2 N). The organic layer was separated, dried with MgSO_4 , and concentrated. The residue was redissolved in 30 mL of THF at rt, and TBAF (THF solution, 20 mmol) was added. The mixture was stirred at rt for 6 h and quenched with brine. The organic layer was separated, dried with MgSO_4 and concentrated. Flash column chromatography provided the title compound as a white solid (736.6 mg, 3.17 mmol, 85%). R_f (hexanes) 0.28; ^1H NMR (CDCl_3) δ 1.27 (br s, 14H), 1.38 (m, 4H), 1.53 (m, 4H), 1.94 (t, 2H, $J = 2.6$ Hz), 2.18 (td, 4H, $J = 7.1, 2.6$ Hz); ^{13}C NMR

(CDCl₃) δ 18.38, 28.48, 28.75, 29.09, 29.48, 29.57, 29.59, 68.00, 84.82; IR (thin film) 3278 s, 2112 w cm⁻¹; MS (EI) m/z (% rel. intensity) 232 (M⁺, 0.01), 205 (4), 147 (4), 121 (16), 107 (23), 95 (52), 81 (100); Anal Calcd for C₁₇H₂₈: C 87.86, H 12.14; Found: C 87.86, H 12.43.

1,19-Eicosadiyne 25d

The title compound was prepared in the same manner, obtained as a white solid in 75% yield from 1,16-diiodohexadecane. R_f (hexanes) 0.25; ¹H NMR (CDCl₃) δ 1.26 (m, 20H), 1.39 (m, 4H), 1.53 (m, 4H), 1.93 (t, 2H, J = 2.5 Hz), 2.18 (td, 4H, J = 7.1, 2.5 Hz); ¹³C NMR (CDCl₃) δ 18.40, 28.52, 28.77, 29.11, 29.50, 29.60, 29.64, 29.66, 68.00, 84.82; IR (thin film) 3287 m, 2114 vw cm⁻¹; MS (CI) m/z (% rel. intensity) 274 (M⁺, 2), 273 (5), 149 (5), 135 (12), 123 (23), 109 (54), 95 (100), 81 (98); Anal Calcd for C₂₀H₃₄: C 87.52, H 12.48; Found: C 87.21, H 12.77.

Preparation of 2-Bromo-1-decen-9-yne 26a

To B-Br-9-BBN (5 mmol) in 5 mL of CH₂Cl₂ at 0 °C was added dropwise 1,9-decadiyne (from GFS, 800 μ L, 4.86 mmol). The mixture was stirred at 0 °C for 3 h. HOAc (2.8 mL) was added and the mixture was stirred at 0 °C for another 1 h. Aqueous NaOH (2 N, 50 mL) and H₂O₂ (30%, 5.5 mL) were added and the mixture was stirred at rt for 30 min. The organic layer was separated, and the aqueous layer was washed with pentane. The combined organic layer was washed with saturated NaHCO₃ (aq.), dried with MgSO₄ and concentrated. Flash column chromatography provided 2,9-dibromo-1,9-decadiene **27a** (286.3 mg, 0.967 mmol, 20%) as a side product. The title compound was obtained as a colorless oil (566.8 mg, 2.63 mmol,

54%). R_f (hexanes) 0.46; ^1H NMR (CDCl_3) δ 1.33 (m, 2H), 1.42 (m, 2H), 1.55 (m, 4H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.19 (td, 2H, $J = 7.0, 2.6$ Hz), 2.42 (t, 2H, $J = 7.4$ Hz), 5.38 (d, 1H, $J = 1.2$ Hz), 5.55 (d, 1H, $J = 1.1$ Hz); ^{13}C NMR (CDCl_3) δ 18.33, 27.68, 27.80, 28.28, 28.33, 41.28, 68.18, 84.57, 116.37, 134.71; IR (neat) 3306 s, 2118 w, 1629 m cm^{-1} ; Anal Calcd for $\text{C}_{10}\text{H}_{15}\text{Br}$: C 55.83, H 7.03; Found: C 55.60, H 7.15. Vinyl bromides **26b**, **26c** and **26d** were prepared in the same manner.

2-Bromo-1-tetradecen-13-yne 26b

1,13-Tetradecadiyne **25b** provided 2,13-dibromo-1,13-tetradecadiene **27b** as a side product (25%). The title compound was obtained as a colorless oil in 47% yield (63% based on recovered starting material). R_f (hexanes) 0.48; ^1H NMR (CDCl_3) δ 1.2-1.5 (m, 16H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.18 (td, 2H, $J = 7.1, 2.6$ Hz), 2.41 (t, 2H, $J = 7.1$ Hz), 5.38 (d, 1H, $J = 1.5$ Hz), 5.55 (d, 1H, $J = 1.3$ Hz); ^{13}C NMR (CDCl_3) δ 18.37, 27.84, 28.35, 28.46, 28.71, 29.05, 29.25, 29.41, 41.38, 68.03, 84.72, 116.18, 134.90, one aliphatic carbon was not located; IR (neat) 3304 s, 2118 w, 1629 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 272 ($\text{M}^+ + 1$, 0.02), 270 ($\text{M}^+ - 1$, 0.02), 149 (10), 135 (19), 121 (23), 109 (57), 107 (30), 95 (98), 93 (51), 81 (100), 79 (59); Anal Calcd for $\text{C}_{14}\text{H}_{23}\text{Br}$: C 62.00, H 8.55; Found: C 62.02, H 8.81.

2-Bromo-1-heptadecen-16-yne 26c

1,16-Heptadecadiyne **25c** provided 2,16-dibromo-1,16-heptadecadiene **27c** as a side product (19%). The title compound was obtained as a colorless oil in 46% yield (67% based on recovered starting material). R_f (hexanes) 0.52; ^1H NMR (CDCl_3) δ 1.2-1.5 (m, 22H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.18 (td, 2H, $J = 7.1, 2.6$ Hz), 2.41 (t, 2H, $J = 7.3$ Hz), 5.38 (d, 1H, $J = 1.4$ Hz), 5.55 (d, 1H, $J = 1.2$ Hz); ^{13}C NMR (CDCl_3) δ 18.39, 27.88, 28.40, 28.49, 28.76, 29.11, 29.30, 29.50, 29.58, 29.60, 41.41, 68.02, 84.83, 116.20, 134.96, two aliphatic carbons were not located; IR (neat) 3310 s, 2119 w, 1630 m cm^{-1} ; Anal Calcd for $\text{C}_{17}\text{H}_{29}\text{Br}$: C 65.17, H 9.33; Found: C 65.39, H 9.60.

2-Bromo-1-eicosen-19-yne 26d

1,19-Eicosadiyne **25d** provided 2,19-dibromo-1,19-eicosadiene **27d** as a side product (19%). The title compound was obtained as a colorless oil in 45% yield (61% based on recovered starting material). R_f (hexanes) 0.43; ^1H NMR (CDCl_3) δ 1.26 (m, 22H), 1.39 (m, 2H), 1.54 (m, 4H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.18 (td, 2H, $J = 7.1, 2.6$ Hz), 2.41 (t, 2H, $J = 7.3$ Hz), 5.38 (d, 1H, $J = 1.4$ Hz), 5.55 (d, 1H, $J = 1.3$ Hz); ^{13}C NMR (CDCl_3) δ 18.37, 27.89, 28.40, 28.49, 28.74, 29.10, 29.29, 29.50, 29.59, 29.64, 41.41, 67.99, 84.62, 116.06, 134.90, five aliphatic carbons were not located; IR (neat) 3312 s, 2119 w, 1630 m cm^{-1} ; MS (CI) m/z (% rel. intensity) 356 ($\text{M}^+ + 1$, 1.4), 354 ($\text{M}^+ - 1$, 1.2), 275 (40), 137 (25), 123 (41), 109 (69), 95 (100), 81 (83); Anal Calcd for $\text{C}_{20}\text{H}_{35}\text{Br}$: C 67.59, H 9.93; Found: C 67.99, H 10.22.

Preparation of Pentacarbonyl(1-methoxy-2-methylene-9-decynylidene) chromium(0) 6a.

2-Bromo-1-decen-9-yne **26a** (167.3 mg, 0.778 mmol) was dissolved in 18 mL of THF and cooled to -78°C . PhLi (0.782 mmol) was added dropwise and the mixture was stirred at -78°C for 1.5 h. $t\text{BuLi}$ (1.57 mmol) was then added. The resultant solution turned yellow immediately and then green. It was stirred at -78°C for 30 min before being transferred to $\text{Cr}(\text{CO})_6$ (172.2 mg, 0.782 mmol) in 35 mL of THF at -78°C . The reaction mixture was warmed up to rt and stirred for 2 h. The solvent was removed and the residue was dried on high vacuum for 40 min, then taken up in $\text{H}_2\text{O}/\text{CH}_2\text{Cl}_2$ (10 mL/10 mL). Me_3OBF_4 (230 mg, 1.55 mmol) was added and the yellow suspension turned orange immediately. The mixture was stirred at rt for 80 min and quenched with saturated NaHCO_3 (aq.). The mixture was extracted with ether, and the organic layer was washed with brine, dried with MgSO_4 , concentrated, and chromatographed (pentane) to give the title compound as a red oil (128.5 mg, 0.347 mmol, 44%). R_f (hexanes) 0.14; ^1H NMR (CDCl_3) δ 1.3-1.5 (m, 8H), 1.94 (t, 1H, $J = 2.5$ Hz), 2.19 (m, 4H), 4.59 (s, 3H), 4.95 (s, 1H), 5.06 (s, 1H); ^{13}C NMR (CDCl_3) δ 18.30, 27.26, 28.25, 28.40, 28.79, 32.58, 66.20, 68.20, 84.50, 111.11 (br), 162.84, 216.21, 223.78, 356.73; IR (neat) 3311 m, 2118 w, 2063 s, 1987 shoulder, 1945 vs cm^{-1} ; MS (EI) m/z (% rel. intensity) 370 (M^+ , 0.6), 314 (4), 286 (2), 258 (5), 230 (11),

228 (30), 176 (10), 161 (18), 149 (34), 129 (100). Alkenyl complexes **6b**, **6c** and **6d** were prepared in the same manner.

Pentacarbonyl(1-methoxy-2-methylene-13-tetradecynylidene)chromium(0) 6b

The title compound was obtained as a red oil in 43% yield from 2-bromo-1-tetradecen-13-yne **26b**. R_f (hexanes) 0.18; ^1H NMR (CDCl_3) δ 1.2-1.6 (m, 16H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.18 (m, 4H), 4.58 (s, 3H), 4.93 (s, 1H), 5.05 (s, 1H); ^{13}C NMR (CDCl_3) δ 18.34, 27.36, 28.42, 28.69, 29.03, 29.35, 32.63, 66.15, 68.02, 84.74, 111.26 (br), 162.92, 216.20, 223.81, 356.76, three aliphatic carbons were not located; IR (neat) 3312 m, 2119 w, 2063 s, 1986 shoulder, 1940 vs cm^{-1} ; MS (EI) m/z (% rel. intensity) 426 (M^+ , 0.6), 370 (26), 342 (6), 314 (18), 286 (66), 285 (100), 234 (39).

Pentacarbonyl(1-methoxy-2-methylene-16-heptadecynylidene)chromium(0) 6c

The title compound was obtained as a red oil in 48% yield from 2-bromo-1-hetpadecen-16-yne **26c**. R_f (hexanes) 0.20; ^1H NMR (CDCl_3) δ 1.2-1.4 (m, 20H), 1.52 (m, 2H), 1.94 (t, 1H, $J = 2.6$ Hz), 2.18 (m, 4H), 4.58 (s, 3H), 4.93 (s, 1H), 5.05 (s, 1H); ^{13}C NMR (CDCl_3) δ 18.38, 27.40, 28.47, 28.75, 29.10, 29.38, 29.50, 29.58, 32.66, 66.18, 68.01, 84.82, 110.82 (br), 162.96, 216.22, 223.84, 356.81, four aliphatic carbons were not located; IR (neat) 3313 m, 2119 w, 2064 s, 1945 vs cm^{-1} .

Pentacarbonyl(1-methoxy-2-methylene-19-eicosynylidene)chromium(0) 6d

The title compound was obtained as a red oil in 41% yield from 2-bromo-1-eicosen-19-yne **26d**. The preparation procedure was same as that of its lower homologs except that the deprotonation of PhLi was carried out at -78 °C for 10 min and 0 °C for 50 min due to the lower solubility of the substrate. R_f (hexanes) 0.17; ^1H NMR (CDCl_3) δ 1.2-1.5 (m, 28H), 1.94 (t, 1H, $J = 2.4$ Hz), 2.18 (m, 4H), 4.58 (s, 3H),

4.93 (s, 1H), 5.05 (s, 1H); ^{13}C NMR (CDCl_3) δ 18.35, 27.40, 28.48, 28.72, 29.07, 29.59, 32.65, 66.07, 67.98, 84.68, 110.77 (br), 162.99, 216.21, 223.78, 356.92, nine aliphatic carbons were not located; IR (neat) 3312 w, 2119 vw, 2063 s, 1986 shoulder, 1938 vs cm^{-1} .

General Procedure for the Thermolysis of Alkenyl Complexes **6**.

Alkenyl carbene complex **6** was dissolved in the designated solvent at the concentration of 0.002 M. The red solution was deoxygenated by freeze-pump-thaw method (4 cycles), and heated in an oil bath to the designated temperature. The solution turned from red to yellow during the reaction. After the completion of the reaction, the solvent was removed, and the residue was taken up in CH_2Cl_2 /ether and stirred in air overnight. The mixture was then loaded on silica gel and chromatographed to give the products indicated in the manuscript.

The yield of dimers **11** was determined by their weight upon isolation. The monomers **8**, **9**, **7** and **10** were very difficult to separate from each other completely. In most cases a mixture was obtained containing all or some of the four. Their ratio

and yields were determined by ^1H NMR and taken from the integrals of the following characteristic peaks (δ):

8b: 6.54 (s, 2H); **9b**: 7.36 (s, 1H); **7b**: 6.57 (s, 1H), 6.60 (s, 1H); **10b**: 6.32 (s, 1H), 6.34 (s, 1H).

8c: 6.52 (s, 2H); **9c**: 7.31 (s, 1H); **7c**: 6.56 (s, 1H), 6.59 (s, 1H); **10c**: 6.28 (s, 1H), 6.30 (s, 1H).

8d: 6.52 (s, 2H); **9d**: 7.28 (s, 1H); **7d**: 6.55 (s, 1H), 6.57 (s, 1H); **10d**: 6.27 (s, 1H), 6.28 (s, 1H).

Analytically pure samples of the major products were further isolated from the mixture and fully characterized. The spectral data of **8b**, **8c** and **7c** have been presented in section 6.3, and the new compounds are listed below:

12-Hydroxy-15-methoxy-[10]-paracyclophane 7b

R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.28; ^1H NMR (CDCl_3) δ 0.7-0.8 (m, 8H), 1.1-1.2 (m, 4H), 1.5-1.6 (m, 2H), 1.68 (m, 2H), 2.19 (m, 1H), 2.35 (m, 1H), 2.95 (m, 1H), 3.04 (m, 1H), 3.79 (s, 3H), 4.25 (s, 1H), 6.57 (s, 1H), 6.60 (s, 1H); ^{13}C NMR (CDCl_3) δ 25.20, 25.30, 26.97, 27.02, 27.14, 28.50, 28.52, 29.59, 29.89, 56.10, 114.08, 118.36, 125.67, 129.57, 146.57, 152.24, one aliphatic carbon was not located; IR (neat) 3398 br, 2854 m, 1199 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 262 (M^+ , 100), 163 (12), 150 (23), 137 (17), 121 (11), 86 (48), 84 (72); m/z calcd for $\text{C}_{17}\text{H}_{26}\text{O}_2$: 262.1933; Found: 262.1936.

15-Hydroxy-18-methoxy-[13]-paracyclophane 7c

Pale yellow oil. R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.37; ^1H NMR (CDCl_3) δ 0.9-1.6 (m, 22H), 2.16 (m, 1H), 2.33 (m, 1H), 2.90 (m, 1H), 3.00 (m, 1H), 3.77 (s, 3H), 4.27 (s, 1H), 6.56 (s, 1H), 6.59 (s, 1H); ^{13}C NMR (CDCl_3) δ 26.38, 26.49, 27.02, 27.41, 27.74, 28.12, 28.39, 28.43, 29.43, 29.85, 56.30, 113.79, 117.80, 125.73, 129.80, 146.97, 151.78, three aliphatic carbons were not located; IR (neat) 3439 m br, 2855 s, 1415 s, 1199 s, 845 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 304 (M^+ , 100), 191 (8), 177 (15), 163 (42), 151 (62), 137 (75), 121 (33), 107 (22), 91 (38), 77 (19); m/e Calcd for $\text{C}_{20}\text{H}_{32}\text{O}_2$: 304.2402; Found: 304.2396.

18-Hydroxy-21-methoxy-[16]-paracyclophane 7d

Colorless oil. R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.38; ^1H NMR (CDCl_3) δ 1.1-1.2 (m, 24H), 1.60 (m, 4H), 2.58 (t, 2H, $J = 6.3$ Hz), 2.62 (t, 2H, $J = 6.4$ Hz), 3.76 (s, 3H), 4.21 (s, 1H), 6.55 (s, 1H), 6.57 (s, 1H); ^{13}C NMR (CDCl_3) δ 27.10, 27.16, 27.30, 27.32, 27.50, 28.00, 28.19, 28.46, 29.00, 29.03, 29.47, 56.25, 113.48, 117.47, 125.44, 129.41, 146.90, 151.65, five aliphatic carbons were not located; IR (neat) 3406 br, 2855 s, 1198 s cm^{-1} ; MS (EI) m/z (% rel. intensity) 346 (M^+ , 100), 163 (4), 151 (6), 137 (5); m/z Calcd for $\text{C}_{23}\text{H}_{38}\text{O}_2$: 346.2872; Found: 346.2876; Anal Calcd for $\text{C}_{23}\text{H}_{38}\text{O}_2$: C 79.71, H 11.05; Found: C 79.68, H 11.14.

16-Hydroxy-13-methoxy-[10]-metacyclophane 8b

Pale yellow oil. R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.42; ^1H NMR (CDCl_3) δ 0.69 (m, 2H), 0.93 (m, 4H), 1.19 (m, 2H), 1.31 (m, 4H), 1.45 (m, 2H), 2.01 (m, 2H), 2.47 (m, 2H), 2.90 (m, 2H) 3.76 (s, 3H), 4.47 (s, 1H), 6.54 (s, 2H); ^{13}C NMR (CDCl_3) δ 26.45, 26.79, 27.12, 27.44, 30.46, 55.63, 113.66, 129.44, 146.27, 153.44; IR (neat) 3494 br, 2858 s, 1461 s, 1184 s, 757 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 262 (M^+ , 100), 219 (1), 151 (13), 137 (13); Anal Calcd for $\text{C}_{17}\text{H}_{26}\text{O}_2$: C 77.82, H 9.99; Found: C 77.55, H 10.00.

19-Hydroxy-16-methoxy-[13]-metacyclophane 8c

Pale yellow oil. R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.45; R_f (CHCl_3 /hexanes 1/1) 0.43; ^1H NMR (CDCl_3) δ 1.0-1.3 (m, 18H), 1.66 (m, 4H), 2.64 (t, 4H, $J = 6.2$ Hz), 3.75 (s, 3H), 4.29 (s, 1H), 6.52 (s, 2H); ^{13}C NMR (CDCl_3) δ 26.17, 26.71, 26.82, 27.15, 27.27, 28.87, 29.65, 55.62, 113.62, 128.69, 145.91, 152.91; IR (neat) 3506 br, 2855 s, 1478 s, 1187 s, 763 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 304 (M^+ , 100), 163 (29), 151 (76), 137 (80), 121 (35), 91 (44), 77 (24); Anal Calcd for $\text{C}_{20}\text{H}_{32}\text{O}_2$: C 78.90, H 10.59; Found: C 79.07, H 10.46.

14-Methoxy-tricyclo[10.2.1.1^{1,14}]hexadec-12-en-15-one 9b

R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.42; ^1H NMR (CDCl_3) δ 1.1-1.4 (m, 14H), 1.36 (d, 1H, $J = 4.2$ Hz, H-16), 1.51 (m, 1H, H-2), 1.58 (d, 1H, $J = 4.1$ Hz, H-16'), 1.59 (m, 1H), 1.73 (m, 1H), 1.96 (m, 1H, H-11), 2.25 (m, 1H, H-11'), 2.44 (m, 1H, H-2'), 3.51

(s, 3H, H-methoxy), 7.36 (s, 1H, H-13); ^{13}C NMR (CDCl_3) δ 23.81 (C-11), 25.47, 25.49, 25.51, 25.78, 26.24, 26.40, 26.77, 26.95, 27.79, 41.14 (C-1), 43.93 (C-16), 57.06 (C-methoxy), 72.44 (C-14), 141.27 (C-12), 152.26 (C-13), 204.66 (C-15); IR (neat) 2859 s, 1701 s cm^{-1} ; MS (EI) m/z (% rel. intensity) 262 (M^+ , 100), 151 (20), 137 (41) 123 (10), 91 (13); m/z Calcd for $\text{C}_{17}\text{H}_{26}\text{O}_2$: 262.1933; Found: 262.1929.

The structure of **9b** was further confirmed by X-ray crystallographic analysis. The NMR assignment was based on 2D experiments, namely carbon-proton correlation, carbon-proton long range correlation and COSY. 1D NOE experiment was also performed.

Figure 1 NOE Effect of 9b

17-Methoxy-tricyclo[13.2.1.1^{1,17}]nonadec-15-en-18-one **9c**

The following spectral data for **9c** were extracted from the spectra of a mixture of **9c** and **8c** with the aid of a sample of pure **8c**. R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.42; ^1H NMR (CDCl_3) δ 1.1-1.4 (m, 18H), 1.37 (d, 1H, $J = 4.2$ Hz), 1.38 - 1.54 (m, 5H), 1.57 (d, 1H, $J = 4.2$ Hz), 1.90 - 1.92 (m, 1H), 2.22 - 2.33 (m, 2H), 3.48 (s, 3H), 7.29 (s, 1H); ^{13}C NMR (CDCl_3) δ 23.64, 25.84, 25.97, 26.19, 26.33, 26.64, 26.48, 27.04, 27.13, 27.33, 27.39, 27.62, 27.85, 39.94, 44.00, 57.06, 73.03, 139.94, 151.54, 204.75.

12-Hydroxy-16-methoxy-[10]paracyclophane **10b**

R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.34; ^1H NMR (CDCl_3) δ 0.6-0.7 (m, 4H), 0.81 (m, 4H), 1.09 (m, 2H), 1.15 (m, 2H), 1.58 (m, 4H), 2.56 (m, 3H, H-10, H-1 and H-1'), 2.88

(m, 1H, H-10'), 3.82 (s, 3H, H-methoxy), 4.61 (s, 1H, OH), 6.32 (s, 1H, H-13), 6.34 (s, 1H, H-15); ^{13}C NMR (CDCl_3) δ 21.76 (C-10), 25.21, 25.32, 25.77, 27.06, 27.20, 28.68, 28.74, 29.03, 35.99 (C-1), 55.62 (C-methoxy), 105.19 (C-15), 109.74 (C-13), 113.58 (C-11), 141.62 (C-14), 154.50 (C-12), 158.87 (C-16); IR (neat) 3438 br, 2854 m, 1424 m, 1101 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 262 (M^+ , 100), 177 (10), 163 (29), 150 (47), 137 (15), 120 (14), 91 (18), 77 (13), 55 (12); m/z Calcd for $\text{C}_{17}\text{H}_{26}\text{O}_2$: 262.1933; Found: 262.1940.

The structure of **10b** was further confirmed by X-ray crystallographic analysis. The NMR assignment was based on 2D experiments, namely carbon-proton correlation and carbon-proton long range correlation. NOE study of the title compound was also performed. Because of the proximity of the two aromatic signals, complete selectivity could not be achieved in the irradiation (for instance, irradiation of the singlet at δ 6.32 led to 38% saturation of the singlet at δ 6.34). However, the data shown below did provide support for the assigned structure.

Table 1 NOE Effect of 10b

Irradiation δ / ppm (saturation%)	Enhancement δ / ppm (percentage)
4.61 (100)	6.32 (8.9); 2.56 (6.2)
3.82 (100)	6.34 (5.9)
6.32 (100); 6.34 (38)	4.61 (6.6); 3.82 (5.9)
6.32 (37); 6.34 (100)	4.61 (3.0); 3.82 (12)

15-Hydroxy-19-methoxy-[13]paracyclophane 10c

R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.34; ^1H NMR (CDCl_3) δ 0.80 - 1.36 (m, 18H), 1.56 (m, 4H), 2.50 (m, 1H), 2.80 (m, 1H), 3.77 (s, 3H), 4.60 (s, 1H), 6.27 (s, 1H), 6.28 (s, 1H); ^{13}C NMR (CDCl_3) δ 22.37, 26.18, 26.76, 26.99, 27.33, 27.49, 27.97, 28.08,

28.27, 29.63, 35.69, 55.62, 104.22, 108.78, 113.84, 141.59, 154.07, 158.53, 2 aliphatic C's not located; ^{13}C NMR (C_6D_6) δ 23.32, 27.18, 27.82, 28.01, 28.35, 28.47, 28.57, 28.96, 29.18, 29.30, 30.54, 36.57, 55.70, 104.60, 109.66, 115.06, 141.82, 155.45, 159.66, 1 aliphatic carbon not located; mass spectrum m/z (% rel intensity) 304 M^+ (100), 274 (4), 191 (4), 177 (8), 163 (22), 151 (41), 137 (5), 91 (5), 55 (3); m/z calcd for $\text{C}_{20}\text{H}_{32}\text{O}_2$: 304.2394, measured 304.2402.

8,20-Dihydroxy-11,23-dimethoxy-[6.6]-paracyclophane 11a

R_f (ether/ CH_2Cl_2 /hexanes 1/1/4) 0.25; ^1H NMR (CDCl_3) δ 1.07 (m, 4H), 1.25 (m, 4H), 1.48 (m, 4H), 1.67 (m, 4H), 2.47 (t, 4H, $J = 6.0$ Hz), 2.60 (t, 4H, $J = 6.0$ Hz), 3.71 (s, 6H), 5.18 (s, 2H), 6.52 (s, 2H), 6.54 (s, 2H); ^{13}C NMR (CDCl_3) δ 27.74, 27.87, 28.08, 28.40, 28.41, 56.34, 112.70, 117.61, 125.77, 129.09, 146.38, 151.38, one aliphatic carbon was not located; IR (neat) 3398 s br, 2856 s, 1414 s, 1200 s cm^{-1} ; MS (EI) m/z (% rel. intensity) 412 (M^+ , 50), 151 (8), 88 (11), 86 (62), 84 (100); m/z Calcd for $\text{C}_{26}\text{H}_{36}\text{O}_4$: 412.2614; Found: 412.2618.

12,28-Dihydroxy-15,31-dimethoxy-[10.10]-paracyclophane 11b

White solid. R_f (ether/ CH_2Cl_2 /hexanes 1/1/4) 0.43; ^1H NMR (CDCl_3) δ 1.2-1.3 (m, 24H), 1.55 (m, 4H), 1.62 (m, 4H), 2.51 (t, 4H, $J = 7.4$ Hz), 2.57 (t, 4H, $J = 7.3$ Hz), 3.74 (s, 6H), 4.86 (s, 2H), 6.57 (s, 2H), 6.60 (s, 2H); ^{13}C NMR (CDCl_3) δ 27.92, 27.99, 28.07, 28.37, 28.51, 28.84, 29.07, 29.12, 29.69, 29.75, 56.36, 113.26, 116.91, 126.10, 129.81, 147.06, 151.36; IR (thin film) 3382 br, 2853 m, 1198 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 524 (M^+ , 100), 191 (7), 177 (6), 163 (12), 151 (22), 137 (18), 121 (13); m/z Calcd for $\text{C}_{34}\text{H}_{52}\text{O}_4$: 524.3866; Found: 524.3869; Anal Calcd for $\text{C}_{34}\text{H}_{52}\text{O}_4$: C 77.82, H 9.99; Found: C 77.17, H 10.21.

15,34-Dihydroxy-18,37-dimethoxy-[13.13]-paracyclophane 11c

R_f (ether/ CH_2Cl_2 /hexanes 1/1/10) 0.09; ^1H NMR (CDCl_3) δ 1.2-1.4 (m, 36H), 1.5-1.6 (m, 8H), 2.51 (t, 4H, $J = 7.6$ Hz), 2.55 (t, 4H, $J = 7.6$ Hz), 3.76 (s, 6H), 4.32 (s, 2H), 6.56 (s, 2H), 6.59 (s, 2H); ^{13}C NMR (CDCl_3) δ 28.93, 28.96, 29.04, 29.06, 29.10,

29.52, 29.69, 29.82, 29.98, 56.23, 112.93, 117.01, 126.04, 129.99, 146.85, 151.55, four aliphatic carbons were not located; IR (thin film) 3604 br cm^{-1} ; MS (FAB) m/z (% rel. intensity) 608 (M^+ , 100), 151 (59); m/z Calcd for $\text{C}_{40}\text{H}_{64}\text{O}_4$: 608.4805; Found: 608.4806.

18,40-Dihydroxy-21,43-dimethoxy-[16.16]-paracyclophane 11d

R_f (ether/ CH_2Cl_2 /hexanes 1/1/4) 0.57; ^1H NMR (CDCl_3) δ 1.2-1.3 (m, 48H), 1.54 (m, 4H), 1.60 (m, 4H), 2.52 (t, 4H, $J = 7.6$ Hz), 2.56 (t, 4H, $J = 7.6$ Hz), 3.76 (s, 6H), 4.31 (s, 2H), 6.57 (s, 2H), 6.59 (s, 2H); ^{13}C NMR (CDCl_3) δ 29.15, 29.17, 29.19, 29.23, 29.28, 29.34, 29.54, 29.65, 29.81, 30.03, 56.16, 112.76, 116.93, 125.95, 129.92, 146.79, 151.50, six aliphatic carbons were not located; IR (thin film) 3295 br, 1198 w cm^{-1} ; MS (FAB) m/z (% rel. intensity) 692 (M^+ , 100), 151 (88); m/z Calcd for $\text{C}_{46}\text{H}_{76}\text{O}_4$: 692.5744; Found: 692.5744.

8,20,32-Trihydroxy-11,23,35-trimethoxy-[6.6.6]-paracyclophane 28a

White solid. R_f (ether/ CH_2Cl_2 /hexanes 1/1/2) 0.21; ^1H NMR (CDCl_3) δ 1.2-1.6 (m, 24H), 2.49 (t, 6H, $J = 7.2$ Hz), 2.53 (t, 6H, $J = 7.4$ Hz), 3.72 (s, 9H), 4.60 (br s, 3H), 6.50 (s, 3H), 6.55 (s, 3H); ^{13}C NMR (CDCl_3) δ 28.35, 28.42, 29.07, 29.16, 29.45, 29.69, 29.82, 56.27, 113.09, 117.15, 126.14, 129.70, 146.82, 151.42, the compound was contaminated with impurities and one extra aliphatic carbon was detected; IR (thin film) 3392 br, 2850 s, 1201 m cm^{-1} ; MS (EI) m/z (% rel. intensity) 618 (M^+ , 100), 412 (14), 205 (14), 191 (11), 177 (16), 163 (37), 151 (74), 137 (55), 121 (32); m/z Calcd for $\text{C}_{39}\text{H}_{54}\text{O}_6$: 618.3920; Found: 618.3930.

Structural Determination of *para*-cyclophane 10b.

Figure 1. ORTEP Diagram of **10b**.

Table 1. Crystal Data and Structure Refinement of **10b**.

Empirical formula	$C_{17}H_{26}O_2$
Formula weight	262.38
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	$Pna2_1$
Unit cell dimensions	$a = 15.3076(9)$ Å $\alpha = 90^\circ$ $b = 14.6049(9)$ Å $\beta = 90^\circ$ $c = 27.4736(18)$ Å $\gamma = 90^\circ$
Volume, Z	$6142.2(7)$ Å ³ , 16
Density (calculated)	1.135 g/cm ³
Absorption coefficient	0.072 mm ⁻¹
F(000)	2304
Crystal size	0.20 x 0.15 x 0.10 mm
θ range for data collection	1.48 to 28.35°
Limiting indices	$-19 \leq h \leq 18$, $-19 \leq k \leq 19$, $-25 \leq l \leq 36$
Reflections collected	28917
Independent reflections	11701 ($R_{int} = 0.0565$)
Completeness to $\theta = 28.35^\circ$	94.3 %
Absorption correction	Empirical from SADABS
Max. and min. transmission	0.9928 and 0.9857
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11701 / 1 / 305
Goodness-of-fit on F^2	2.055
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.1449$, $wR2 = 0.3510$
R indices (all data)	$R1 = 0.2128$, $wR2 = 0.3742$
Absolute structure parameter	2(3)
Largest diff. peak and hole	1.343 and -0.432 eÅ ⁻³

Table 2. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for **10b**.

U (eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1B)	2672(5)	11198(4)	1150(3)	40(2)
C(2B)	1998(5)	11746(4)	1311(3)	41(2)
C(3B)	1951(5)	12602(5)	1097(3)	45(2)
C(4B)	2470(6)	12866(5)	694(3)	58(2)
C(5B)	3089(6)	12238(5)	527(3)	60(2)
C(6B)	3201(6)	11377(5)	748(3)	56(2)
C(7B)	1329(6)	11433(6)	1665(4)	64(2)
C(8B)	508(12)	11046(11)	1434(6)	140(5)
C(9B)	404(14)	10497(14)	961(9)	167(7)
O(1B)	2746(3)	10349(4)	1396(2)	55(1)
O(2B)	1320(3)	13221(3)	1283(2)	51(1)
C(1)	2288(5)	4937(4)	-268(3)	42(2)
C(3)	1642(5)	5140(4)	507(3)	40(2)
C(4)	2358(5)	5682(4)	641(3)	39(2)
C(5)	2987(5)	5878(5)	320(3)	49(2)
C(6)	2988(5)	5478(5)	-139(3)	44(2)
C(7)	768(6)	4263(5)	-131(3)	61(2)
C(8)	-21(11)	5029(12)	-271(7)	137(5)
C(9)	278(10)	5758(10)	-565(6)	127(5)
O(1)	2213(4)	4508(4)	-728(2)	62(2)
O(2)	975(3)	4928(3)	828(2)	51(1)
C(1C)	-587(4)	7516(4)	1913(3)	37(2)
C(2C)	140(4)	7068(4)	1692(2)	29(1)
C(3C)	415(4)	6261(4)	1924(2)	32(1)
C(4C)	42(5)	5959(4)	2364(3)	43(2)
C(5C)	-610(5)	6456(4)	2569(3)	43(2)
C(6C)	-959(5)	7222(4)	2330(3)	42(2)
C(7C)	665(5)	7483(5)	1295(3)	44(2)
C(8C)	1383(10)	8113(9)	1521(6)	112(4)
C(9C)	1141(17)	8719(18)	1911(10)	210(9)
O(1C)	-847(3)	8280(3)	1667(2)	45(1)
O(2C)	1091(3)	5796(3)	1718(2)	40(1)
C(1A)	3659(5)	4336(5)	2948(3)	44(2)
C(2A)	2853(5)	4562(5)	2734(3)	41(2)
C(3A)	2605(5)	4044(4)	2333(3)	40(2)
C(4A)	3110(5)	3337(5)	2147(3)	49(2)
C(5A)	3893(5)	3122(5)	2387(3)	48(2)
C(6A)	4173(5)	3630(5)	2778(3)	49(2)
C(7A)	2254(5)	5280(5)	2956(3)	57(2)
C(8A)	1763(8)	4937(7)	3411(4)	90(3)
C(9A)	1312(7)	3977(7)	3328(4)	82(3)
O(1A)	3860(4)	4867(3)	3338(2)	58(1)
O(2A)	1804(3)	4250(3)	2099(2)	45(1)
C(2)	1582(5)	4772(5)	35(3)	42(2)
C(10B)	240(20)	10820(20)	525(16)	274(14)
C(11B)	440(20)	10540(19)	25(12)	232(11)
C(12B)	1485(16)	10768(16)	-192(10)	199(9)
C(13B)	1980(30)	11680(30)	-504(18)	340(20)

Table 2. Continued.

	x	y	z	U(eq)
C(14B)	2760(30)	11000(40)	-400(20)	360(20)
C(15B)	2960(20)	12290(20)	-400(12)	242(12)
C(16B)	3596(9)	12438(9)	37(6)	113(4)
C(17B)	3467(6)	9789(6)	1281(4)	63(2)
C(10)	535(11)	6614(11)	-364(6)	133(5)
C(11)	1125(13)	7270(13)	-605(8)	158(7)
C(12)	1398(9)	8072(10)	-384(5)	113(4)
C(13)	1906(8)	7948(8)	121(5)	99(4)
C(14)	2901(7)	7839(7)	29(4)	81(3)
C(15)	3379(6)	7539(6)	489(4)	67(2)
C(16)	3722(6)	6561(5)	442(3)	58(2)
C(17)	2997(7)	4557(7)	-1037(4)	75(3)
C(10C)	1893(7)	8797(6)	2291(4)	76(3)
C(11C)	1165(14)	8781(14)	2657(8)	178(7)
C(12C)	976(7)	8009(6)	2954(4)	75(3)
C(13C)	270(14)	8164(14)	3300(8)	173(7)
C(14C)	21(7)	7222(7)	3641(4)	81(3)
C(15C)	-948(7)	6961(7)	3446(4)	85(3)
C(16C)	-947(6)	6147(6)	3079(4)	65(2)
C(17C)	-1391(5)	8920(5)	1918(3)	53(2)
C(10A)	1714(8)	3304(8)	3638(5)	100(4)
C(11A)	1370(9)	2336(8)	3561(5)	98(4)
C(12A)	1728(9)	1885(8)	3098(5)	104(4)
C(13A)	2733(8)	1752(8)	3096(5)	93(3)
C(14A)	3059(8)	1289(8)	2637(5)	97(3)
C(15A)	4036(8)	1404(8)	2572(5)	101(4)
C(16A)	4366(6)	2240(6)	2238(4)	66(2)
C(17A)	4655(6)	4626(6)	3603(4)	72(3)

Table 3. Bond Lengths (Å) and Angles (°) for **10b**.

C(1B)-C(2B)	1.379(10)	C(1B)-C(6B)	1.395(11)
C(1B)-O(1B)	1.415(9)	C(2B)-C(3B)	1.383(10)
C(2B)-C(7B)	1.484(12)	C(3B)-C(4B)	1.416(12)
C(3B)-O(2B)	1.418(9)	C(4B)-C(5B)	1.397(12)
C(5B)-C(6B)	1.407(11)	C(5B)-C(16B)	1.582(17)
C(7B)-C(8B)	1.519(19)	C(8B)-C(9B)	1.53(3)
C(9B)-C(10B)	1.31(4)	O(1B)-C(17B)	1.409(10)
C(1)-C(2)	1.387(10)	C(1)-C(6)	1.377(10)
C(1)-O(1)	1.414(10)	C(3)-O(2)	1.385(9)
C(3)-C(4)	1.401(9)	C(3)-C(2)	1.406(10)
C(4)-C(5)	1.338(10)	C(5)-C(6)	1.390(11)
C(5)-C(16)	1.541(11)	C(7)-C(2)	1.522(11)
C(7)-C(8)	1.691(19)	C(8)-C(9)	1.41(2)
C(9)-C(10)	1.42(2)	O(1)-C(17)	1.472(12)
C(1C)-C(6C)	1.350(10)	C(1C)-O(1C)	1.363(8)
C(1C)-C(2C)	1.427(9)	C(2C)-C(3C)	1.404(8)
C(2C)-C(7C)	1.485(10)	C(3C)-O(2C)	1.362(8)
C(3C)-C(4C)	1.407(10)	C(4C)-C(5C)	1.357(10)
C(5C)-C(6C)	1.402(10)	C(5C)-C(16C)	1.561(12)
C(7C)-C(8C)	1.562(16)	C(8C)-C(9C)	1.44(3)
C(9C)-C(10C)	1.56(3)	C(9C)-C(11C)	2.05(3)
O(1C)-C(17C)	1.430(9)	C(1A)-O(1A)	1.358(9)
C(1A)-C(6A)	1.378(10)	C(1A)-C(2A)	1.406(10)
C(2A)-C(3A)	1.388(10)	C(2A)-C(7A)	1.521(11)
C(3A)-C(4A)	1.388(10)	C(3A)-O(2A)	1.416(9)
C(4A)-C(5A)	1.405(11)	C(5A)-C(6A)	1.373(11)
C(5A)-C(16A)	1.533(11)	C(7A)-C(8A)	1.543(14)
C(8A)-C(9A)	1.578(15)	C(9A)-C(10A)	1.438(16)
O(1A)-C(17A)	1.461(11)	C(10B)-C(11B)	1.47(4)
C(11B)-C(12B)	1.74(4)	C(12B)-C(13B)	1.76(5)
C(12B)-C(14B)	2.07(5)	C(13B)-C(14B)	1.57(6)
C(13B)-C(15B)	1.76(5)	C(14B)-C(15B)	1.90(5)
C(15B)-C(16B)	1.56(3)	C(10)-C(11)	1.47(2)
C(11)-C(12)	1.38(2)	C(12)-C(13)	1.598(19)
C(13)-C(14)	1.552(16)	C(14)-C(15)	1.523(15)
C(15)-C(16)	1.527(12)	C(10C)-C(11C)	1.50(2)
C(11C)-C(12C)	1.42(2)	C(12C)-C(13C)	1.46(2)
C(13C)-C(14C)	1.71(2)	C(14C)-C(15C)	1.623(15)
C(15C)-C(16C)	1.558(14)	C(10A)-C(11A)	1.524(17)
C(11A)-C(12A)	1.532(18)	C(12A)-C(13A)	1.551(17)
C(13A)-C(14A)	1.514(17)	C(14A)-C(15A)	1.515(18)
C(15A)-C(16A)	1.609(15)		
C(2B)-C(1B)-C(6B)	125.4(7)	C(2B)-C(1B)-O(1B)	114.5(6)
C(6B)-C(1B)-O(1B)	119.8(6)	C(1B)-C(2B)-C(3B)	115.3(7)
C(1B)-C(2B)-C(7B)	123.2(6)	C(3B)-C(2B)-C(7B)	121.4(7)
C(2B)-C(3B)-C(4B)	123.3(7)	C(2B)-C(3B)-O(2B)	117.3(6)
C(4B)-C(3B)-O(2B)	119.3(6)	C(5B)-C(4B)-C(3B)	117.4(7)
C(4B)-C(5B)-C(6B)	121.8(8)	C(4B)-C(5B)-C(16B)	119.5(8)
C(6B)-C(5B)-C(16B)	118.3(8)	C(1B)-C(6B)-C(5B)	116.1(7)
C(2B)-C(7B)-C(8B)	114.2(9)	C(7B)-C(8B)-C(9B)	129.4(15)
C(10B)-C(9B)-C(8B)	127(3)	C(17B)-O(1B)-C(1B)	117.6(6)
C(2)-C(1)-C(6)	123.4(7)	C(2)-C(1)-O(1)	113.4(6)
C(6)-C(1)-O(1)	123.2(7)	O(2)-C(3)-C(4)	122.4(7)
O(2)-C(3)-C(2)	116.9(6)	C(4)-C(3)-C(2)	120.6(7)

Table 3. Continued.

C(5)-C(4)-C(3)	120.7(7)	C(4)-C(5)-C(6)	120.6(7)
C(4)-C(5)-C(16)	121.4(7)	C(6)-C(5)-C(16)	118.0(7)
C(5)-C(6)-C(1)	118.3(7)	C(2)-C(7)-C(8)	109.3(8)
C(9)-C(8)-C(7)	113.4(13)	C(8)-C(9)-C(10)	122.0(16)
C(1)-O(1)-C(17)	115.3(6)	C(6C)-C(1C)-O(1C)	124.0(6)
C(6C)-C(1C)-C(2C)	123.0(6)	O(1C)-C(1C)-C(2C)	113.1(6)
C(3C)-C(2C)-C(1C)	115.2(6)	C(3C)-C(2C)-C(7C)	121.0(6)
C(1C)-C(2C)-C(7C)	123.2(5)	O(2C)-C(3C)-C(4C)	120.7(5)
O(2C)-C(3C)-C(2C)	117.1(6)	C(4C)-C(3C)-C(2C)	122.1(6)
C(5C)-C(4C)-C(3C)	119.1(6)	C(4C)-C(5C)-C(6C)	121.0(7)
C(4C)-C(5C)-C(16C)	117.4(6)	C(6C)-C(5C)-C(16C)	121.6(7)
C(1C)-C(6C)-C(5C)	119.3(7)	C(2C)-C(7C)-C(8C)	109.2(8)
C(9C)-C(8C)-C(7C)	118.5(15)	C(8C)-C(9C)-C(10C)	110.7(19)
C(8C)-C(9C)-C(11C)	140.1(19)	C(10C)-C(9C)-C(11C)	46.7(10)
C(1C)-O(1C)-C(17C)	117.7(6)	O(1A)-C(1A)-C(6A)	124.4(7)
O(1A)-C(1A)-C(2A)	113.2(6)	C(6A)-C(1A)-C(2A)	122.4(7)
C(3A)-C(2A)-C(1A)	116.3(6)	C(3A)-C(2A)-C(7A)	121.9(6)
C(1A)-C(2A)-C(7A)	121.5(7)	C(2A)-C(3A)-C(4A)	123.1(7)
C(2A)-C(3A)-O(2A)	118.8(6)	C(4A)-C(3A)-O(2A)	118.2(6)
C(5A)-C(4A)-C(3A)	117.8(7)	C(6A)-C(5A)-C(4A)	120.9(7)
C(6A)-C(5A)-C(16A)	121.0(7)	C(4A)-C(5A)-C(16A)	117.6(7)
C(5A)-C(6A)-C(1A)	119.3(7)	C(8A)-C(7A)-C(2A)	113.3(7)
C(7A)-C(8A)-C(9A)	112.6(9)	C(10A)-C(9A)-C(8A)	109.5(9)
C(1A)-O(1A)-C(17A)	116.4(6)	C(1)-C(2)-C(3)	116.0(6)
C(1)-C(2)-C(7)	122.9(7)	C(3)-C(2)-C(7)	121.1(7)
C(9B)-C(10B)-C(11B)	136(4)	C(10B)-C(11B)-C(12B)	117(3)
C(13B)-C(12B)-C(11B)	135(2)	C(13B)-C(12B)-C(14B)	47.6(19)
C(11B)-C(12B)-C(14B)	176(2)	C(14B)-C(13B)-C(12B)	77(3)
C(14B)-C(13B)-C(15B)	69(3)	C(12B)-C(13B)-C(15B)	132(3)
C(13B)-C(14B)-C(15B)	60(3)	C(13B)-C(14B)-C(12B)	56(3)
C(15B)-C(14B)-C(12B)	108(3)	C(16B)-C(15B)-C(13B)	136(3)
C(16B)-C(15B)-C(14B)	104(3)	C(13B)-C(15B)-C(14B)	50.6(19)
C(5B)-C(16B)-C(15B)	108.7(15)	C(9)-C(10)-C(11)	124.5(17)
C(12)-C(11)-C(10)	122.5(18)	C(11)-C(12)-C(13)	115.6(13)
C(14)-C(13)-C(12)	110.4(10)	C(15)-C(14)-C(13)	111.6(9)
C(16)-C(15)-C(14)	111.4(8)	C(15)-C(16)-C(5)	111.9(7)
C(11C)-C(10C)-C(9C)	84.1(14)	C(12C)-C(11C)-C(10C)	123.1(16)
C(12C)-C(11C)-C(9C)	122.3(16)	C(10C)-C(11C)-C(9C)	49.2(11)
C(11C)-C(12C)-C(13C)	113.7(14)	C(12C)-C(13C)-C(14C)	113.5(14)
C(15C)-C(14C)-C(13C)	102.2(10)	C(16C)-C(15C)-C(14C)	113.0(8)
C(5C)-C(16C)-C(15C)	111.1(7)	C(9A)-C(10A)-C(11A)	113.9(11)
C(12A)-C(11A)-C(10A)	113.0(11)	C(11A)-C(12A)-C(13A)	114.3(11)
C(14A)-C(13A)-C(12A)	112.8(11)	C(13A)-C(14A)-C(15A)	112.0(10)
C(14A)-C(15A)-C(16A)	117.5(10)	C(5A)-C(16A)-C(15A)	109.7(8)

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