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Supplementary Information (8 Pages)

The Steric Influence of *E*- or *Z*-Monosubstituted and Terminally Disubstituted Vinyl Groups in Matrix-Isolated Cyclopentane-1,3-diyl Triplet Diradicals as Probed by the Zero-Field EPR *D* Parameter

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Experimental Section

General Aspects. Elemental analyses were carried out by Mr. C.-P. Gneis of the Microanalytical Division of the Institute of Inorganic Chemistry, University of Würzburg. TLC analysis was conducted on precoated silica-gel foils Polygram SIL G/UV₂₅₄ (40 x 80 mm) from Machery & Nagel. Spots were identified with a UV lamp or by exposure to iodine vapor. Silica gel (63-200 μ m; Woelm) was used for column chromatography, the adsorbant/substrate ratio was ca. 100:1.

EPR Spectroscopy. A sample (5 μ mol) of the corresponding azoalkane was dissolved in 0.3 mL of 2-MTHF, placed into an EPR sample tube (\varnothing ca. 2 mm) and degassed by purging several minutes with argon gas. The samples were sealed and the 77-K matrices

were prepared by freezing with liquid nitrogen. The triplet diradicals were generated by irradiation with the 364-nm line of an INNOVA-100 CW argon-ion laser (widened beam, 2.5 W, 2 min) at 77 K. The EPR spectra were recorded on a Bruker EPR-300 spectrometer (9.43 GHz, spectra accumulation with the Bruker 1620 data system). The D values were determined by analysis of the Z signals.

Computations. Full geometry optimization of the monoradicals **A** was carried out on the highest molecular symmetry to achieve a planar arrangement of the substituents at the radical site by using the semiempirical PM3 method with annihilated UHF wavefunctions. These were available through the VAMP 6.1 program package and run on a IRIS INDIGO R4000 Silicon Graphics workstation.⁹ Alternatively, these values were calculated by the B3LYP/6-31G* method, which is provided in the GAUSSIAN98 program. Both methods result in spin expectation $\langle S^2 \rangle$ values between 0.75 and 0.76.¹⁰ We have conducted a thorough conformational search, by calculating the heat of formation for every 10 degrees of rotation around the CC bond between the π bond and the radical center to locate the minimum energy conformations.

Synthesis.

General Procedure A: The azoalkane **1**⁴ (150.0 mg, 0.56 mmol) was dissolved in 50 mL abs. toluene, and the phosphosponium salt (1.40 mmol) and KO^tBu (1.00 mmol) were added under an argon-gas atmosphere. The suspension was stirred for 18 h at room temperature (ca. 20 °C), the solvent was evaporated (40 °C / 5 torr) and the precipitate was dissolved in 40 mL of methyl *tert*-butyl ether. After extraction with water (3 x 25 mL), brief stirring over anhydrous magnesium sulfate (5.0 g) for drying, filtration and evaporation of the solvent (20 °C, 10 torr), the crude product was purified by silica-gel chromatography [methylene chloride : ethyl acetate (10 : 1)].

General Procedure B: The azoalkane **1** (150.0 mg, 0.56 mmol) was dissolved in 50 mL abs. toluene, and the phosphorane (1.00 mmol) was added under an argon-gas atmosphere. The suspension was stirred for 18 h at room temperature (ca. 20 °C), the solvent was evaporated (40 °C / 5 torr) and the precipitate was dissolved in 40 mL of methyl *tert*-butyl ether. After extraction with water (3 x 25 mL), brief stirring over anhydrous magnesium

sulfate (5.0 g) for drying, filtration and evaporation of the solvent (20 °C, 10 torr), the crude product was purified by silica-gel chromatography [methylene chloride : ethyl acetate (10 : 1)].

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-(2,2-dimethylethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2c)

According to the General Procedure B, 706 mg (2.32 mmol) of the isopropylenetriphenylphosphorane were added while stirring to 250.0 mg (0.93 mmol) of the azoalkane 1. Colorless needles, mp 78 - 81 °C (dec.), 182.6 mg (67%); λ_{max} (C₆H₆) nm: 361 (log ϵ 2.64); R_F = 0.51 [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) ν = 3060 cm⁻¹, 2960, 2880, 1740, 1500, 14447, 1371, 1238, 1077; ¹H NMR: δ = 0.26 (s, 3H, 9-H), 0.93 (s, 3H, 10-H), 0.99 (d, ³ J = 6.5 Hz, 3H, 13-H), 1.15 (d, ³ J = 6.5 Hz, 3H, 13-H), 1.26-1.72 (m, 6H, 5-H, 6-H und 7-H), 2.62-2.90 (m, 1H, 4a-H), 3.28-3.39 (m, 1H, 7a-H), 5.70 (dd, ³ J = 6.5 Hz, ³ J = 6.5 Hz, 1H, 11-H), 7.11-7.45 (m, 3H, *o*-, *p*-Ph), 7.68-7.75 (m, 2H, *m*-Ph); ¹³C-NMR: δ = 17.0 (q, C-9), 17.9 (q, C-10), 23.4 (2xq, C-13), 25.4 (t, C-5), 25.6 (t, C-6), 28.4 (t, C-7), 48.7 (d, C-4a), 51.3 (d, C-7a), 65.1 (s, C-8), 96.2 (s, C-1), 98.1 (s, C-4), 119.6 (s, C-12), 127.4 (2 x d, *m*-Ph), 127.5 (d, *p*-Ph), 128.2 (2 x d, *o*-Ph), 136.4 (s, *ipso*-Ph), 144.1 (d, C-11); Anal calcd. for C₂₀H₂₆N₂ (294.4): C, 81.59; H, 8.90; N, 9.51. Found: C, 81.83; H, 8.62; N, 9.25.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-8,8-dimethyl-4-phenyl-(Z-2-trimethylsilyl-ethenyl)-1,4-methano-1H-cyclopenta[d]pyridazine (2d)

According to the General Procedure B, 352 mg (1.02 mmol) of the trimethylsilylmethylenephosphorane were added while stirring to 111.0 mg (0.41 mmol) of the azoalkane 1. Colorless needles, mp 111 - 113 °C (dec.), 40.3 mg (29%); λ_{max} (C₆H₆) nm: 362 (log ϵ 1.89); R_F = 0.66 [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) ν = 3060 cm⁻¹, 3020, 1520, 1460, 1430, 1200, 1120, 1000; ¹H-NMR: δ = 0.26 (s, 3H, 9-H), 0.98 (s, 3H, 10-H), 1.35-1.78 (m, 6H, 5-H, 6-H und 7-H), 1.98 (s, 9H, 13-H), 2.93-3.21 (m,

1H, 4a-H), 3.32-3.51 (m, 1H, 7a-H), 5.63 (d, 1H, $^3J = 8.7$ Hz, 11-H), 6.25 (d, 1H, $^3J = 8.7$ Hz, 12-H), 7.31-7.45 (m, 3H, *o*-, *p*-Ph), 7.68-7.79 (m, 2H, *m*-Ph); ^{13}C -NMR: $\delta = -2.20$ (3 x q, C-13), 16.7 (q, C-9), 18.0 (q, C-10), 24.8 (t, C-5), 25.4 (t, C-6), 28.6 (t, C-7), 48.3 (d, C-4a), 48.7 (d, C-7a), 66.5 (s, C-8), 98.4 (s, C-4), 101.8 (s, C-1), 123.4 (d, C-12), 127.5 (d, *p*-Ph), 128.0 (2 x d, *m*-Ph), 128.4 (2 x d, *o*-Ph), 134.4 (s, *ipso*-Ph), 139.1 (d, C-11); Anal calcd. for $\text{C}_{21}\text{H}_{30}\text{N}_2\text{Si}$ (338.6): C, 74.50; H, 8.93; N, 8.27. Found: C, 74.68; H, 8.63; N, 8.29.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-(Z-2-iodoethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2f)

According to the General Procedure B, 566 mg (1.41 mmol) of the iodomethylene-triphenylphosphorane were added while stirring to 150 mg (0.56 mmol) of the azoalkane 1. Colorless needles, mp 127 - 129 °C (dec.), 94.5 mg (43%); λ_{max} (C_6H_6) nm: 361 (log ϵ 1.35); $R_F = 0.69$ [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) $\nu = 2960$ cm^{-1} , 2880, 1603, 1490, 1370, 1300, 1240, 1080, 1030, 950; ^1H -NMR: $\delta = 0.25$ (s, 3H, 9-H), 0.91 (s, 3H, 10-H), 1.31-1.70 (m, 6H, 5-H, 6-H und 7-H), 3.25-3.37 (m, 1H, 4a-H), 3.45-3.57 (m, 1H, 7a-H), 6.70 (d, $^3J = 9.2$ Hz, 1H, 11-H), 7.00 (d, $^3J = 9.2$ Hz, 1H, 12-H), 7.28-7.43 (m, 3H, *o*-, *p*-Ph), 7.50-7.64 (m, 2H, *m*-Ph); ^{13}C -NMR: $\delta = 17.1$ (q, C-9), 18.1 (q, C-10), 24.9 (t, C-5), 25.3 (t, C-6), 28.2 (t, C-7), 47.8 (d, C-4a), 47.8 (d, C-7a), 67.1 (s, C-8), 96.4 (s, C-4), 97.0 (s, C-1), 127.8 (d, C-12), 128.6 (2 x d, *m*-Ph), 128.7 (d, *p*-Ph), 128.9 (2 x d, *o*-Ph), 132.5 (d, C-11), 136.0 (s, *ipso*-Ph); Anal calcd. for $\text{C}_{18}\text{H}_{21}\text{IN}_2$ (392.3): C, 55.11; H, 5.40; N, 7.14. Found: C, 54.83; H, 5.76; N, 7.38.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-(Z-2-carboxyethylethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2i)

According to the General Procedure B, 525 mg (1.51 mmol) of the carboxyethyl-methylenetriphenylphosphorane were added while stirring to 180 mg (0.67 mmol) of the azoalkane 1. Colorless needles, mp 118 - 120 °C (dec.), 92.9 mg (41%); λ_{max} (C_6H_6) nm: 363 (log ϵ 1.44); $R_F = 0.59$ [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) $\nu = 3060$ cm^{-1} , 1720, 1500, 1430, 1410, 1120, 1080, 980; ^1H -NMR: $\delta = 0.29$ (s, 3H, 9-H),

1.00 (s, 3H, 10-H), 1.35 (t, $^3J = 7.20$ Hz, 3H, 15-H), 1.23-1.65 (m, 6H, 5-H, 6-H und 7-H), 2.91-3.08 (m, 1H, 4a-H), 3.37-3.51 (m, 1H, 7a-H), 4.28 (q, $^3J = 7.2$ Hz, 2H, 14-H), 6.64 (d, $^3J = 10.1$ Hz, 1H, 11-H), 7.23 (d, $^3J = 10.1$ Hz, 1H, 12-H), 7.31-7.53 (m, 3H, *o*-, *p*-Ph), 7.64-7.75 (m, 2H, *m*-Ph); ^{13}C -NMR: $\delta = 14.2$ (q, C-15), 19.1 (q, C-9), 19.4 (q, C-10), 26.8 (t, C-5), 29.0 (t, C-6), 30.4 (t, C-7), 53.1 (d, C-4a), 55.2 (d, C-7a), 58.2 (t, C-14), 60.7 (s, C-8), 91.5 (s, C-4), 92.9 (s, C-1), 124.4 (t, C-12), 126.4 (2 x d, *m*-Ph), 127.9 (d, *p*-Ph), 128.2 (2 x d, *o*-Ph), 134.9 (s, *ipso*-Ph), 136.6 (d, C-11), 165.9 (s, C-13); Anal calcd. for $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_2$ (338.4): C, 72.52; H, 7.74; N, 8.28. Found: C, 73.01; H, 7.98; N, 7.84.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-8,8-dimethyl-1-(2,2-diphenylethenyl)-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2p)

According to the General Procedure A, 1.05 g (2.05 mmol) of the diphenylmethyltriphenylphosphonium bromide were added to a solution of 250.0 mg (0.93 mmol) of the azoalkane **1** in abs. toluene. 235 mg (2.10 mmol) KO^tBu and 500 mg lithium chloride. Colorless needles, mp 143 - 145 °C (dec.), 130.4 mg (38%); λ_{max} (C_6H_6) nm: 368 (log ϵ 2.85); $R_F = 0.51$ [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) $\nu = 3060$ cm^{-1} , 2960, 2880, 1680, 1500, 1480, 1390, 1240, 1060 cm^{-1} ; ^1H -NMR: $\delta = 0.30$ (s, 3H, 9-H), 0.93 (s, 3H, 10-H), 1.23-1.65 (m, 6H, 5-H, 6-H und 7-H), 2.78-2.90 (m, 1H, 4a-H), 3.20-3.33 (m, 1H, 7a-H), 6.67 (s, 1H, 11-H), 7.13-7.20 (m, 9H, *m*-, *p*-, *m'*-, *p'*-Ph), 7.26-7.33 (m, 4H, *o'*-Ph), 7.36-7.39 (m, 2H, *o*-Ph); ^{13}C -NMR: $\delta = 17.8$ (q, C-9), 18.2 (q, C-10), 25.4 (t, C-5), 25.6 (t, C-6), 28.4 (t, C-7), 48.7 (d, C-4a), 50.6 (d, C-7a), 63.9 (s, C-8), 94.6 (s, C-4), 97.9 (s, C-1), 126.3 (s, C-11), 127.3 (2 x d, *m*-Ph), 127.8 (4 x d, *m'*-Ph), 128.3 (d, *p*-Ph), 128.4 (2 x d, *p'*-Ph), 129.8 (2 x d, *o*-Ph), 131.7 (4 x d, *o'*-Ph), 134.8 (s, *ipso*-Ph), 141.5 (s, *ipso'*-Ph), 143.2 (s, *ipso'*-Ph), 143.5 (d, C-12); Anal calcd. for $\text{C}_{30}\text{H}_{30}\text{N}_2$ (418.6): C, 86.08; H, 7.22; N, 6.69. Found: C, 86.39; H, 6.83; N, 7.01.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-(Z-2-methoxyethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2a)

According to the General Procedure A, 480.0 mg (1.40 mmol) of the methoxymethyltriphenylphosphonium chloride were added to a solution of 150.0 mg (0.56 mmol) of the azoalkane **1** in abs. toluene. At $-50\text{ }^{\circ}\text{C}$ 112.2 mg (1.00 mmol) of KO^tBu and 500 mg lithium chloride were added and the suspension was stirred for 8 d at $-50\text{ }^{\circ}\text{C}$. Colorless needles, mp $143 - 145\text{ }^{\circ}\text{C}$ (dec.), 59.8 mg (36%); λ_{max} (C_6H_6) nm: 362 (log ϵ 1.62); $R_F = 0.61$ [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) $\nu = 3060\text{ cm}^{-1}$, 2980, 1580, 1490, 1440, 1200, 1130, 1010; $^1\text{H-NMR}$: $\delta = 0.27$ (s, 3H, 9-H), 0.89 (s, 3H, 10-H), 1.25-1.75 (m, 6H, 5-H, 6-H und 7-H), 2.80-2.93 (m, 1H, 4a-H), 3.25-3.48 (m, 1H, 7a-H), 3.66 (s, 3H, 13-H), 4.95 (d, $^3J = 7.10\text{ Hz}$, 1H, 12-H), 6.28 (d, $^3J = 7.10\text{ Hz}$, 1H, 11-H), 7.28-7.49 (m, 3H, *o*-, *p*-Ph), 7.68-7.75 (m, 2H, *o*-Ph); $^{13}\text{C-NMR}$: $\delta = 17.7$ (q, C-9), 18.1 (q, C-10), 25.4 (t, C-5), 25.5 (t, C-6), 28.4 (t, C-7), 48.6 (d, C-4a), 48.9 (d, C-7a), 58.0 (s, C-13), 64.5 (s, C-8), 96.0 (s, C-4), 97.9 (s, C-1), 100.2 (d, C-12), 127.4 (2 x d, *m*-Ph), 128.2 (d, *p*-Ph), 128.8 (2 x d, *o*-Ph), 136.4 (s, *ipso*-Ph), 151.2 (d, C-11); Anal calcd. for $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}$ (296.4): C, 76.99; H, 8.16; N, 9.45. Found: C, 77.28; H, 8.41; N, 9.32.

(1 α ,4 α ,4a α ,7a α)-4,4a,5,6,7,7a-Hexahydro-1-(Z-2-bromoethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2g)

According to the General Procedure A, 730 mg (1.68 mmol) of the bromomethyltriphenylphosphonium bromide were added to a solution of 180 mg (0.67 mmol) of the azoalkane **1** in abs. toluene and 140 mg (1.25 mmol) KO^tBu , the suspension was stirred for 4 d. Colorless needles, mp $143 - 145\text{ }^{\circ}\text{C}$ (dec.), 108.6 mg (47%); λ_{max} (C_6H_6) nm: 364 (log ϵ 1.83); $R_F = 0.67$ [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) $\nu = 3060\text{ cm}^{-1}$, 2960, 2880, 1740, 1500, 14447, 1371, 1238, 1077; $^1\text{H-NMR}$: $\delta = 0.28$ (s, 3H, 9-H), 0.95 (s, 3H, 10-H), 1.39-1.61 (m, 6H, 5-H, 6-H und 7-H), 3.30-3.35 (m, 1H, 4a-H), 3.51-3.58 (m, 1H, 7a-H), 6.32 (d, $^3J = 8.4\text{ Hz}$, 1H, 11-H), 6.43 (d, $^3J = 8.4\text{ Hz}$, 1H, 12-H), 7.30-7.43 (m, 3H, *o*-, *p*-Ph), 7.59-7.64 (m, 2H, *m*-Ph); $^{13}\text{C-NMR}$: $\delta = 16.8$ (q, C-9), 17.9 (q, C-10), 24.9 (t, C-5), 25.4 (t, C-6), 28.2 (t, C-7), 47.9 (d, C-4a), 48.3 (d, C-7a), 67.1 (s, C-8), 96.4 (s, C-4), 97.0 (s, C-1), 121.7 (t, C-11), 127.8, (2 x d, *m*-Ph), 128.0 (d, *p*-Ph), 128.6 (2 x d, *o*-Ph), 133.5 (d, C-12), 136.0 (s, *ipso*-Ph); Anal calcd. for $\text{C}_{18}\text{H}_{21}\text{BrN}_2$ (345.3): C, 62.61; H, 6.13; N, 8.11. Found: C, 62.29; H, 6.49; N, 7.81.

(1 α ,4 α ,4 α ,7 α)-4,4a,5,6,7,7a-Hexahydro-1-(Z-2-chloroethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2h)

According to the General Procedure A, 720 mg (2.08 mmol) of the chloromethyltriphenylphosphonium chloride were added to a solution of 180 mg (0.67 mmol) of the azoalkane **1** in abs. toluene and 180 mg (1.60 mmol) KO^tBu, the suspension stirred for 4 d. Colorless needles, mp 127 - 129 °C (dec.), 111.0 mg (45%); λ_{max} (C₆H₆) nm: 359 (log ϵ 1.83); R_F = 0.58 [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) ν = 3060 cm⁻¹, 2980, 1520, 1430, 1410, 1190, 1130, 960, 950; ¹H-NMR: δ = 0.35 (s, 3H, 9-H), 1.03 (s, 3H, 10-H), 1.44-1.58 (m, 6H, 5-H, 6-H und 7-H), 3.23-3.41 (m, 1H, 4a-H), 3.53-3.71 (m, 1H, 7a-H), 6.40 (d, ³ J = 8.4 Hz, 1H, 11-H), 6.51 (d, ³ J = 8.4 Hz, 1H, 12-H), 7.34-7.50 (m, 3H, *o*-, *p*-Ph), 7.67-7.80 (m, 2H, *m*-Ph); ¹³C-NMR: δ = 17.2 (q, C-9), 18.3 (q, C-10), 25.2 (t, C-5), 25.7 (t, C-6), 28.4 (t, C-7), 48.1 (d, C-4a), 49.3 (d, C-7a), 67.2 (s, C-8), 96.2 (s, C-4), 96.6 (s, C-1), 121.4 (d, C-11), 126.4, (d, C-12), 127.4 (2 x d, *m*-Ph), 127.6 (d, *p*-Ph), 128.2 (2 x d, *o*-Ph), 135.6 (s, *ipso*-Ph); Anal calcd. for C₁₈H₂₁ClN₂ (300.8): C, 71.87; H, 7.04; N, 9.31. Found: C, 72.05; H, 7.39; N, 9.36.

(1 α ,4 α ,4 α ,7 α)-4,4a,5,6,7,7a-Hexahydro-1-(2,2-dicarboxyethylethenyl)-8,8-dimethyl-4-phenyl-1,4-methano-1H-cyclopenta[d]pyridazine (2i)

To a solution of 180 mg (0.67 mmol) of the azoalkane **1** in 50 mL abs. toluene were added 129 mg (0.80 mmol) of diethyl malonate. The mixture was cooled to -78 °C and 760 μ L (0.80 mmol) butyllithium were added dropwise. After stirring for 2 h, 750 mg of molecular sieves (4 Å) was added, the suspension was allowed to warm to room temperature (ca. 20 °C) and stirred for an additional 18 h. The molecular sieves were removed by filtration, the solvent was evaporated (40 °C / 5 torr), and the crude product was purified by silica-gel chromatography [methylene chloride : ethyl acetate (10 : 1)]. Colorless plates, mp 136 - 138 °C (dec.), 92.9 mg (41%); λ_{max} (C₆H₆) nm: 358 (log ϵ 1.98); R_F = 0.43 [silica gel, methylene chloride : ethyl acetate (10 : 1)]; IR (KBr) ν = 3060 cm⁻¹, 2969, 2880, 1750, 1520, 1420, 1180, 1040, 980; ¹H-NMR: δ = 0.29 (s, 3H, 9-H), 1.00 (s, 3H, 10-H), 1.33 (t, ³ J = 7.20 Hz, 3H, 15-H), 1.36 (t, ³ J = 7.20 Hz, 3H, 15-H), 1.23-1.65 (m, 6H, 5-H, 6-H und 7-H), 2.91-3.08 (m, 1H, 4a-H), 3.37-3.51 (m, 1H, 7a-H), 4.24 (q,

$^3J = 7.20$ Hz, 2H, 14-H), 4.28 (q, $^3J = 7.20$ Hz, 2H, 14-H'), 7.30 (s, 1H, 11-H), 7.15-7.26 (m, 3H, *o*-, *p*-Ph), 7.34-7.52 (m, 2H, *m*-Ph); ^{13}C -NMR: $\delta = 14.2$ (2 x q, C-15), 19.1 (q, C-9), 19.4 (q, C-10), 26.8 (t, C-5), 29.0 (t, C-6), 30.4 (t, C-7), 53.1 (d, C-4a), 55.2 (d, C-7a), 58.2 (2 x t, C-14), 60.7 (s, C-8), 91.5 (s, C-4), 92.9 (s, C-1), 126.4 (2 x d, *m*-Ph), 127.9 (d, *p*-Ph), 128.2 (2 x d, *o*-Ph), 134.9 (s, *ipso*-Ph), 136.6 (d, C-12), 144.2 (d, C-11), 163.4 (s, C-13), 165.9 (s, C-13); Anal calcd. for $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_4$ (410.5): C, 70.22; H, 7.37; N, 6.82. Found: C, 70.37; H, 7.12; N, 7.03.