Supporting Information for

**Experimental and Theoretical Investigation of the Coarctate
Cyclization of (2-Ethynylphenyl)phenyldiazenes**

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<u>Table of Contents</u>	<u>Page</u>
Experimental procedures	S2
Cartesian coordinates of calculated structures	S17
X-ray data for 11	S27
X-ray data for 12	S40
NMR spectra	S55

Experimental Section

General. ^1H and ^{13}C NMR spectra were recorded on a 300 MHz spectrometer (^1H , 299.95 MHz; ^{13}C , 75.43 MHz). Chemical shifts (δ) are expressed in ppm downfield from SiMe_4 using the residual nondeuterated solvent as internal standard (CDCl_3 : ^1H , 7.26 ppm; ^{13}C , 77.0 ppm; $\text{C}_4\text{D}_8\text{O}$: ^{13}C , 67.57 ppm). Coupling constants are expressed in hertz. IR spectra were recorded using a FTIR 550 spectrometer. Mass spectra were recorded using an LC mass spectrometer with either APCI or ESI sources. Melting points are uncorrected. CH_2Cl_2 and Et_3N were distilled from CaH_2 under a nitrogen atmosphere prior to use. THF and Et_2O were distilled from Na/benzophenone ketal under a N_2 atmosphere prior to use. All other chemicals were of reagent grade quality and used as obtained from manufacturers. Reactions were carried out in an inert atmosphere (dry N_2 or Ar) when necessary. Column chromatography was performed on reagent grade silica gel (230-400 mesh). Preparative and analytical thin-layer chromatography was performed on precoated silica gel plates.

(2-Iodophenyl)phenyldiazene (9). 2-Iodoaniline (2.0 g, 9.13 mmol) and nitrosobenzene (1.76 g, 16.4 mmol) were reacted according to general procedure B. The crude red oil was chromatographed over silica gel (hexanes) to yield **9** (1.88 g, 67%) as a red solid: mp 58.1-59.2 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 8.05-7.99 (m, 3H), 7.64 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.58-7.50 (m, 3H), 7.44 (dt, $J = 8.4, 1.2$ Hz, 1H), 7.17 (dt, $J = 8.4, 1.2$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 152.3, 151.2, 139.8, 132.2, 131.6, 129.2, 128.9, 123.5, 117.3, 102.5; IR (KBr) 3461, 3361, 2962, 2905, 1585 cm^{-1} ; MS (ESI) m/z (%) 309.0 (100, $\text{M}^+ + \text{H}$), 182.2 (20, $\text{M}^+ - \text{I}^{127}$).

Phenyl[2-(trimethylsilylethynyl)phenyl]diazene (6). Iododiazene **9** (0.68 g, 2.20 mmol), TMSA (0.44 mL, 3.08 mmol), $\text{PdCl}_2(\text{dppf})$ (0.09 g, 0.11 mmol), CuI (41 mg, 0.22 mmol), and THF/DIPA (1:1) were reacted according to general procedure D. Chromatography on silica gel (hexanes) afforded **6** (0.305 g, 50%) as a viscous red oil: ^1H NMR (CDCl_3) δ 8.01-7.98 (m, 2H), 7.72-7.63 (m, 2H), 7.56-7.47 (m, 3H), 7.44-7.37 (m, 2H) 0.23 (s, 9H); ^{13}C NMR (CDCl_3) δ 152.8, 152.5, 133.6, 131.3, 130.5, 129.1, 129.0, 122.8, 123.3, 115.8, 101.9, 101.2, -0.1; IR (KBr) 3057, 2959, 2159, 1473 cm^{-1} ; MS (ESI) m/z (%) 279.1 (100, $\text{M}^+ + \text{H}$).

Phenyl[2-(triisopropylsilylethynyl)phenyl]diazene (7). Iododiazene **9** (1.77 g, 5.74 mmol), TIPSA (1.80 mL, 8.03 mmol), PdCl₂(PPh₃)₂ (0.08 g, 0.114 mmol), CuI (0.109 g, 0.574 mmol), and Et₃N were reacted according to general procedure D. Chromatography on silica gel (0-10% CH₂Cl₂ in hexanes gradient) afforded **7** (2.02 g, 97%) as a viscous red oil: ¹H NMR (CDCl₃) δ 8.02-7.98 (m, 2H), 7.71-7.66 (m, 2H), 7.53-7.45 (m, 3H), 7.24-7.38 (m, 2H), 1.16 (s, 21H); ¹³C NMR (CDCl₃) δ 153.5, 152.8, 134.1, 131.2, 130.3, 129.1, 128.9, 124.0, 123.4, 115.7, 103.8, 97.5, 18.7, 11.4; IR (neat) 2942, 2864, 2155, 1480 cm⁻¹; MS (ESI) *m/z* (%) 363.2 (80, M⁺+H), 385.2 (100, M⁺+Na), 206.3 (20, M⁺-C₉H₂₁Si).

Phenyl[2-(triethylsilylethynyl)phenyl]diazene (8). Iododiazene **9** (1.45 g, 4.71 mmol), TESA (1.18 mL, 6.60 mmol), PdCl₂(PPh₃)₂ (0.07 g, 0.094 mmol), CuI (0.09 g, 0.471 mmol), and Et₃N were reacted according to general procedure D. Chromatography on silica gel (packed with hexanes and eluted with 5% CH₂Cl₂ in hexanes) furnished **8** (1.31 g, 87%) as a viscous red oil: ¹H NMR (CDCl₃) δ 8.02-7.98 (m, 2H), 7.71-7.65 (m, 2H), 7.50-7.47 (m, 3H), 7.41-7.38 (m, 2H), 1.07 (t, *J* = 7.8 Hz, 9H), 0.72 (q, *J* = 7.8, 6H); ¹³C NMR (CDCl₃) δ 153.5, 152.8, 133.9, 131.3, 130.4, 130.0, 128.9, 123.7, 123.4, 115.7, 103.0, 98.7, 7.5, 4.4; IR (neat) 2954, 2910, 2874, 2156, 1474 cm⁻¹; MS (ESI) *m/z* (%) 321.2 (100, M⁺+H), 293.2 (30, M⁺-C₂H₃), 279.2 (40, M⁺-C₃H₅).

2-Phenyl-2*H*-indazole-3-carbaldehyde (10). Diazene **7** (404 mg, 1.11 mmol) was desilylated with TBAF (5.55 mL, 5.55 mmol) according to the first step of general procedure E. After concentrating in vacuo with no heat, the red oil was immediately taken up in DCE (150 mL) and cooled to -5 °C. CuCl (219 mg, 2.22 mmol) was added and the reaction allowed to stir until all s.m. was consumed (by TLC). The reaction mixture was filtered over a short pad of silica and concentrated in vacuo. Chromatography on silica gel (10% CH₂Cl₂, 10% EtOAc, 80% hexanes) afforded **10** (25 mg, 10%) as a tan solid: mp 73.1-74.6 °C; ¹H NMR (CDCl₃) δ 10.12 (s, 1H), 8.32 (dt, *J* = 9.0, 0.9 Hz, 1H), 7.91 (dt, *J* = 9.0, 0.9 Hz, 1H), 7.70-7.60 (m, 5H), 7.52-7.42 (m, 2H); ¹³C NMR (CDCl₃) δ 180.0, 148.5, 130.1, 129.6, 129.5, 129.2, 127.8, 127.1, 126.4, 121.1, 120.9, 118.9; IR (KBr) 3055, 2858, 1667, 1596 cm⁻¹; MS (APCI) *m/z* (%) 223.2 (100, M⁺+H). Isolation of the second band and subsequent recrystallization from CHCl₃ furnished **11**

(25 mg, 11%) as yellow needles suitable for X-ray diffraction: mp 136.5-138.0 °C; ^1H NMR (CDCl_3) δ 7.93-7.91 (m, 4H), 7.85 (d, J = 9.0 Hz, 2H), 7.71 (d, J = 8.4 Hz, 2H) 7.61-7.48 (m, 6H), 7.41 (td, J = 7.5, 1.2 Hz, 2H), 7.27 (td, J = 7.5, 1.2 Hz, 2H); ^{13}C NMR (CDCl_3) δ 148.8, 129.6, 129.1, 129.0, 127.5, 126.5, 124.5, 123.9, 119.8, 118.6, 117.4, 89.0; IR (KBr) 3061, 2921, 1959, 1886, 1502 cm^{-1} ; MS (ESI) m/z (%) 411.1 (100, $\text{M}^+\text{+H}$), 333.1 (20, $\text{M}^+\text{-C}_6\text{H}_5$), 305.2 (40, $\text{M}^+\text{-C}_6\text{H}_5\text{N}_2$) 217.0 (15, $\text{M}^+\text{-C}_{13}\text{H}_9\text{N}_2$). Isolation of the third band afforded **12** (40 mg, 9%) as a light clear violet oil: ^1H NMR (CDCl_3) δ 7.84-7.75 (m, 4H), 7.56-7.48 (m, 3H) 7.34 (td, J = 9.0, 0.9 Hz, 1H) 7.14 (td, J = 9.0, 0.9 Hz, 1H), 5.07 (s, 2H), 1.30 (s, 21H); ^{13}C NMR (CDCl_3) δ 148.5, 139.8, 129.0, 128.7, 126.6, 125.6, 121.9, 121.8, 119.9, 117.8, 55.0, 18.0, 11.9; IR (KBr) 3057, 2941, 2863, 1605, 1460 cm^{-1} ; MS (ESI) m/z (%) 381.1 (80, $\text{M}^+\text{+H}$), 207.2 ($\text{M}^+\text{-SiC}_9\text{H}_{21}\text{O}$).

Alkyne 11 by Mori's method.²⁰ To diazene **6** (141 mg, 0.50 mmol) in DMSO (7 mL) was added CuCl (90 mg, 2.0 mmol). After 30 min the reaction was diluted with Et_2O and the organic layer washed successively with 30% aq. NH_4OH (2 $\times$), brine (2 $\times$), and H_2O (2 $\times$), dried (MgSO_4), filtered over a short pad of silica (Et_2O) and concentrated. The brown solid was dissolved in a minimal amount of Et_2O and the product was precipitated with hexanes to yield **11** (45 mg, 45%) as a fluffy yellow solid. Spectral data were consistent with those given above.

2-Phenyl-3-triethylsilyloxymethyl-2H-indazole (13). Diazene **8** (336 mg, 1.0 mmol) was desilylated with TBAF (2.0 mL, 2.0 mmol) according to the first step of general procedure E. After concentrating in vacuo, the red oil was dissolved in DCE (20 mL) and immediately degassed by four successive freeze/pump/thaw cycles. TESOH (357 mg, 2.7 mmol) was added and the reaction was stirred in a preheated 75 °C sand bath for 17 h. The brown solution was filtered over a short pad of silica and concentrated in vacuo. Chromatography on silica gel (7% CH_2Cl_2 , 7% EtOAc , 86% hexanes) afforded **13** (262 mg, 77%) as a purplish oil: ^1H NMR (CDCl_3) δ 7.82-7.73 (m, 4H), 7.56-7.48 (m, 3H), 7.36-7.31 (m, 1H) 7.17-7.12 (m, 1H), 4.98 (s, 2H), 0.96 (t, J = 8.1 Hz, 9H), 0.66 (q, J = 8.1 Hz, 6H); ^{13}C NMR (CDCl_3) δ 148.6, 139.8, 133.9, 129.1, 128.7, 126.7, 125.5, 122.0, 121.8, 119.7, 117.8, 54.4, 6.7, 4.3.

(2-Iodo-4-methylphenyl)phenyldiazene (18). *p*-Toluidine (1.50 g, 14.0 mmol), BTEA•ICl₂ (6.55 g, 16.8 mmol), and CaCO₃ (1.96 g, 19.60 mmol) were reacted using general procedure A. The iodoaniline (3.07 g, 95%) was obtained as a dark purple oil and used without further purification. A portion of the crude product (1.50 g, 6.46 mmol) and nitrosobenzene (1.24 g, 11.63 mmol) were reacted according to general procedure B. Chromatography on silica gel (hexanes) afforded **18** (1.46 g, 70%) as a red solid: mp 58.5-60.1 °C; ¹H NMR (CDCl₃) δ 8.02-8.00 (m, 2H), 7.99 (d, *J* = 1.5 Hz, 1H), 7.58-7.48 (m, 4H), 7.23 (dd, *J* = 6.6, 0.6 Hz, 1H), 2.41 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 152.3, 149.2, 143.1, 140.1, 131.3, 129.7, 129.1, 123.4, 116.8, 103.3, 20.9; IR (KBr) 3046, 2907, 1585, 1473 cm⁻¹; MS (ESI) *m/z* (%) 323.0 (25, M⁺+H), 245.0 (30, M⁺-C₆H₅), 217.0 (40, M⁺-C₆H₅N₂), 196.0 (20, M⁺-I¹²⁵), 194.0 (100, M⁺-I¹²⁷).

(4-*tert*-Butyl-2-iodophenyl)phenyldiazene (19). 4-*tert*-Butylaniline (0.796 mL, 5 mmol), BTEA•ICl₂ (2.18 g, 5.6 mmol), and CaCO₃ (0.7 g, 7 mmol) were reacted using general procedure A. The iodoaniline (1.35 g, 98%) was obtained as a dark purple solid and used without further purification. A portion of the crude product (0.60 g, 2.2 mmol) and nitrosobenzene (0.471 g, 4.4 mmol) were reacted according to general procedure B. Chromatography on silica gel (hexanes) gave **19** (0.583 g, 72%) as a viscous, bright red oil: ¹H NMR (CDCl₃) δ 8.03-8.01 (m, 3H), 7.62-7.44 (m, 5H), 1.36 (s, 9H); ¹³C NMR (CDCl₃) δ 156.2, 152.4, 149.1, 136.8, 131.3, 129.1, 126.2, 123.4, 116.7, 103.3, 34.9, 31.1; IR (neat) 3459, 2963, 2906, 1585, 1548 cm⁻¹; MS (ESI) *m/z* (%) 365.1 (100, M⁺+H), 350.0 (80, M⁺-CH₃) 335.0 (17, M⁺-2CH₃).

(4-Chloro-2-iodophenyl)phenyldiazene (20). 4-Chloroaniline (0.87 g, 6.84 mmol), BTEA•ICl₂ (3.20 g, 8.21 mmol), and CaCO₃ (0.96 g, 9.58 mmol) were reacted using general procedure A. The iodoaniline (1.40 g, 80%) was obtained as a dark purple oil and used without further purification. A portion of the crude product (1.0 g, 3.94 mmol) and nitrosobenzene (0.76 g, 7.09 mmol) were reacted according to general procedure B. Chromatography on silica gel (5% CH₂Cl₂/hexanes) furnished **20** (0.547 g, 41%) as a red solid: mp 108.5-109.6 °C; ¹H NMR (CDCl₃) δ 8.03 (d, *J* = 1.8 Hz, 1H), 8.00-7.97 (m, 2H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.56-7.51 (m,

3H), 7.41 (dd, $J = 8.4, 1.8$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 152.12, 149.8, 149.1, 137.4, 131.8, 129.2, 123.6, 120.4, 117.8, 103.3; IR (KBr) 3056, 2920, 1565, 1449 cm^{-1} ; MS (ESI) m/z (%) 365.0 (25, $\text{M}^+ + \text{Na}$), 343.0 (100, $\text{M}^+ + \text{H}$), 267.0 (15, $\text{M}^+ - \text{C}_6\text{H}_3$), 265.0 (55, $\text{M}^+ - \text{C}_6\text{H}_5$) 237.0 (40, $\text{M}^+ - \text{C}_6\text{H}_5\text{N}_2$).

(2,4-Diiodophenyl)phenyldiazene (21). 4-Iodoaniline (1.50 g, 6.84 mmol), BTEA $\cdot\text{ICl}_2$ (3.20 g, 8.21 mmol), and CaCO_3 (0.96 g, 9.59 mmol) were reacted using general procedure A. The iodoaniline (2.27 g, 96%) was obtained as a dark purple solid and used without further purification. The crude product (2.26 g, 6.58 mmol) and nitrosobenzene (1.27 g, 11.8 mmol) were reacted according to general procedure B. Chromatography on silica gel (hexanes) afforded **21** (0.44 g, 32%) as a red solid: mp 115.8-116.8 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 8.39 (d, $J = 1.8$ Hz, 1H), 8.00-7.97 (m, 2H), 7.50 (dd, $J = 9.0, 1.8$ Hz, 1H), 7.56-7.51 (m, 3H), 7.37 (d, $J = 9.0$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 152.1, 150.8, 147.5, 138.1, 131.9, 129.7, 123.7, 118.4, 103.4, 98.1; IR (KBr) 3060, 2908, 1555, 1445 cm^{-1} ; MS (ESI) m/z (%) 435.0 (40, $\text{M}^+ + \text{H}$), 357.0 (100, $\text{M}^+ - \text{C}_6\text{H}_5$), 307.0 (25, $\text{M}^+ - \text{I}^{127}$), 201.9 (100, $\text{M}^+ - \text{C}_6\text{H}_5\text{N}_2\text{I}$).

[4-Methyl-2-(triethylsilylethynyl)phenyl]phenyldiazene (14). Iododiazene **18** (1.0 g, 3.10 mmol), TESA (0.79 mL, 4.34 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.043 g, 0.062 mmol), CuI (0.06 g, 0.310 mmol), and Et_3N (31 mL) were reacted according to general procedure D. Chromatography on silica gel (hexanes) gave **14** (1.02 g, 98%) as a red viscous oil: ^1H NMR (CDCl_3) δ 8.01-7.98 (m, 2H), 7.65 (d, $J = 8.5$ Hz, 1H), 7.52-7.48 (m, 4H), 7.21 (dd, $J = 8.5, 1.5$ Hz, 1H), 2.14 (s, 3H), 1.08 (q, $J = 7.5$ Hz, 9H), 0.74 (t, $J = 7.5$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 152.9, 151.5, 141.0, 134.2, 131.0, 130.0, 128.9, 123.9, 123.3, 115.5, 103.3, 100.0, 21.2, 7.6, 4.5; IR (neat) 2954, 2911, 2874, 2151, 1598 cm^{-1} ; MS (APCI) m/z (%) 335.2 (100, $\text{M}^+ + \text{H}$).

[4-tert-Butyl-2-(triethylsilylethynyl)phenyl]phenyldiazene (15). Iododiazene **19** (1.43 g, 3.93 mmol), TESA (0.99 mL, 5.50 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.055 g, 0.079 mmol), CuI (0.075 g, 0.393 mmol), and Et_3N (40 mL) were reacted according to general procedure C. Chromatography on silica gel (hexanes) furnished **15** (1.48 g, 78%) as a red viscous oil: ^1H NMR (CDCl_3) δ 8.00-7.98 (m, 2H), 7.66-7.63 (m, 2H), 7.51-7.42 (m, 4H), 1.37 (s, 9H), 1.08 (t, $J = 7.8$

Hz, 9H), 0.75 (q, J = 7.8 Hz, 6H); ^{13}C NMR (CDCl_3) δ 153.9, 152.9, 151.4, 131.0, 130.5, 128.9, 126.5, 123.5, 123.3, 115.3, 103.7, 97.8, 34.9, 31.1, 7.6, 4.5; IR (neat) 2956, 2910, 2874, 2155, 1593 cm^{-1} ; MS (APCI) m/z (%) 377.2 (100, $\text{M}^+\text{+H}$), 319.1 (25, $\text{M}^+\text{-C}_4\text{H}_9$).

[4-Chloro-2-(triethylsilylethynyl)phenyl]phenyldiazene (16). Iododiazene **20** (0.40 g, 1.17 mmol), TESA (0.29 mL, 1.63 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.016 g, 0.023 mmol), CuI (0.022 g, 0.117 mmol), and Et_3N (12 mL) were reacted according to general procedure D. Chromatography on silica gel (9:1 hexanes/ CH_2Cl_2) gave **16** (1.02 g, 97%) as a red viscous oil: ^1H NMR (CDCl_3) δ 8.00-7.97 (m, 2H), 7.67-7.64 (m, 2H), 7.52-7.48 (m, 3H), 7.35 (dd, J =8.7, 2.4 Hz, 1H); ^{13}C NMR (CDCl_3) δ 152.7, 151.8, 136.3, 133.4, 131.5, 129.3, 129.00, 125.3, 123.4, 117.0, 101.6, 100.3, 7.3, 4.4; IR (KBr) 3065, 2955, 2934, 2158, 1583 cm^{-1} ; MS (APCI) m/z (%) 355.1 (100, $\text{M}^+\text{+H}$).

[2,4-Bis(triethylsilylethynyl)phenyl]phenyldiazene (17). Iododiazene **21** (0.35 g, 0.81 mmol), TESA (0.36 mL, 2.01 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.03 g, 0.04 mmol), CuI (0.023 g, 0.121 mmol) and Et_3N (10 mL) were reacted according to general procedure D. Chromatography on silica gel (0-5% CH_2Cl_2 gradient in hexanes) furnished **17** (0.314 g, 85%) as a red viscous oil: ^1H NMR (CDCl_3) δ 8.00-7.97 (m, 2H), 7.76 (d, J = 1.8 Hz, 1H), 7.65 (d, J = 8.4 Hz, 1H), 7.51-7.45 (m, 4H), 1.06 (m, 18H), 0.70 (m, 12H); ^{13}C NMR (CDCl_3) δ 152.8, 152.3, 137.3, 132.5, 131.5, 128.3, 125.7, 123.5, 120.5, 115.7, 105.0, 102.2, 99.4, 95.2, 7.5, 7.5, 4.4, 4.3; IR (neat) 2955, 2911, 2874, 2150, 2064, 1594; MS (APCI) m/z (%) 459.1 (100, $\text{M}^+\text{+H}$).

(2-Iodo-4-methoxyphenyl)phenyldiazene (24). Aniline (1.0 g, 10.74 mmol), conc. HCl (3.57 mL, 21.47 mmol), NaNO_2 (0.89 g, 12.88 mmol), KOH (2.20 g, 21.47 mmol), and 3-iodophenol (2.60 g, 11.81 mmol) were reacted according to general procedure C, part 1. The resultant red solid (3.45 g) was reacted with MeI (1.21 mL, 19.44 mmol) and Cs_2CO_3 (7.25 g, 22.3 mmol) in DMF (22 mL) according to general procedure C, part 2. Recrystallization from EtOH/ H_2O yielded **24** (2.75 g, 77%) as a red solid: mp 44.8-46.6 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.98-7.95 (m, 2H), 7.69 (d, J = 8.8 Hz, 1H), 7.55-7.46 (m, 4H), 6.97 (dd, J = 8.8, 2.3 Hz, 1H), 3.88 (s, 3H); ^{13}C NMR (CDCl_3) δ 162.1, 152.3, 130.9, 129.1, 128.8, 123.8, 123.3, 122.3, 115.4, 105.2,

55.8; IR (KBr) 3065, 2963, 2834, 1587, 1480 cm^{-1} ; MS (ESI) m/z (%) 339.0 (100, $\text{M}^+\text{+H}$), 261.0 (30, $\text{M}^+\text{-C}_6\text{H}_5$), 212.0 (15, $\text{M}^+\text{-I}^{125}$), 210.9 (55, $\text{M}^+\text{-I}^{127}$).

(4-Bromophenyl)(2-iodo-4-methoxyphenyl)diazene (25). 4-Bromoaniline (1.25 g, 7.27 mmol), conc. HCl (1.21 mL, 14.54 mmol), NaNO_2 (0.60 g, 8.72 mmol), KOH (0.82 g, 14.54 mmol), and 3-iodophenol (1.76 g, 8.0 mmol) were reacted according to general procedure C, part 1. The resultant dark red solid (2.48 g, 6.18 mmol), Cs_2CO_3 (4.24 g, 12.98 mmol), and MeI (0.46 mL, 7.42 mmol) in DMF (15 mL) were reacted according to general procedure C, part 2. Recrystallization from EtOH/ H_2O gave **25** (1.36 g, 45%) as fluffy orange-red needles: mp 117.5-118.1 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.82 (dt, $J = 8.7, 1.8$ Hz, 2H), 7.68 (d, $J = 8.7$ Hz, 1H), 7.64 (dt, $J = 8.7, 1.8$ Hz, 2H), 7.54 (d, $J = 1.8$ Hz, 1H), 6.96 (dd, $J = 8.7, 1.8$ Hz, 1H) 3.21 (s, 3H); ^{13}C NMR (CDCl_3) δ 162.4, 151.1, 145.2, 132.3, 125.3, 124.7, 123.9, 117.9, 115.5, 105.6, 55.9; IR (KBr) 3083, 3069, 1588, 1477, 1228 cm^{-1} ; MS (ESI) m/z 416.9 (90, $\text{M}^+\text{+H}$), 260.8 (25, $\text{M}^+\text{-C}_6\text{H}_4\text{Br}$), 183.1 (50, $\text{M}^+\text{-C}_7\text{H}_7\text{IO}$).

(4-Fluorophenyl)(2-iodo-4-methoxyphenyl)diazene (26). 4-Fluoroaniline (2.0 g, 18.0 mmol), conc. HCl (3.0 mL, 36.0 mmol), NaNO_2 (1.50 g, 21.6 mmol), KOH (2.02 g, 36.0 mmol), and 3-iodophenol (4.36 g, 19.8 mmol) were reacted according to general procedure C, part 1. The resultant dark red solid (5.75 g, 16.2 mmol), Cs_2CO_3 (11.8 g, 34.02 mmol), and MeI (1.21 mL, 19.5 mmol) in DMF (36 mL) were reacted according to general procedure C, part 2. Recrystallization from EtOH/ H_2O afforded **26** (4.2 g, 66% yield) as orange needles: mp 94.8-95.7 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.96 (tt, $J = 5.4, 3.3$ Hz, 2H), 7.67 (d, $J = 9.3$ Hz, 1H), 7.53 (d, $J = 2.4$ Hz, 1H), 7.19 (tt, $J = 5.4, 3.3$ Hz, 2H), 6.96 (dd, $J = 9.0, 2.7$ Hz, 1H), 3.87 (s, 3H); ^{13}C NMR (CDCl_3) δ 164.6 (d, $J = 1002.0$ Hz) 162.4, 148.9 (d, $J = 12.0$ Hz), 145.1, 125.2 (d, $J = 32.1$ Hz), 123.8, 117.8, 116.1 (d, $J = 92.1$ Hz), 115.4, 105.2, 55.8; IR (KBr) 3004, 2927, 1584, 1496 cm^{-1} ; MS (ESI) m/z 357.0 (100, $\text{M}^+\text{+H}$), 261.1 (30, $\text{M}^+\text{-C}_6\text{H}_5\text{F}$), 231.0 (20, $\text{M}^+\text{-I}^{125}$), 229.3 (35, $\text{M}^+\text{-I}^{127}$).

(4-Carbomethoxyphenyl)(2-iodo-4-methoxyphenyl)diazene (27). Methyl 4-aminobenzoate (1.25 g, 8.27 mmol), conc. HCl (1.37 mL, 16.54 mmol) NaNO_2 (0.68 g, 9.92

mmol), KOH (0.93 g, 16.54 mmol), and 3-iodophenol (2.0 g, 9.20 mmol) were reacted according to general procedure C, part 1. The resultant dark red solid (2.93 g, 7.67 mmol), Cs₂CO₃ (5.75 g, 17.63 mmol), and MeI (0.57 mL, 9.20 mmol) in DMF (17 mL) were reacted according to general procedure C, part 2. Recrystallization from EtOH/H₂O furnished **27** (2.21 g, 69%) as red needles: mp 164.5-165.4 °C; ¹H NMR (CDCl₃) δ 8.20 (dt, *J* = 8.7, 1.8 Hz, 2H), 8.01 (dt, *J* = 8.7, 1.8 Hz, 2H), 7.74 (d, *J* = 8.7 Hz, 1H), 7.58 (d, *J* = 2.7 Hz, 1H), 7.00 (dd, *J* = 8.7, 2.7 Hz, 1H), 3.98 (s, 3H), 3.91 (s, 3H); ¹³C NMR (CDCl₃) δ 166.6, 162.8, 154.8, 145.4, 131.6, 139.6, 124.0, 123.1, 118.0, 115.5, 106.3, 55.9, 52.3; IR (KBr) 3096, 2939, 1713, 1591 cm⁻¹; MS (APCI) *m/z* (%) 379.0 (80, M⁺+H) 269.2 (10, M⁺-I), 260.8 (35, M⁺-C₈H₈O₂), 135.1 (100, M⁺-C₈H₈IO₂).

(4-Cyanophenyl)(2-iodo-4-methoxyphenyl)diazene (28). 4-Aminobenzonitrile (2.0 g, 16.9 mmol), conc. HCl (2.82 mL, 33.8 mmol), NaNO₂ (1.40 g, 20.28 mmol), KOH (1.9 g, 33.8 mmol), and 3-iodophenol (4.10 g, 18.60 mmol) were reacted according to general procedure C, part 1. The resultant dark red solid (5.87 g, 16.78 mmol), Cs₂CO₃ (13.4 g, 41.4 mmol), and MeI (1.15 mL, 18.46 mmol) in DMF (34 mL) were reacted according to general procedure C, part 2. Recrystallization from EtOH/H₂O gave **28** (3.23 g, 53%) as orange needles: ¹H NMR (CDCl₃) δ 8.01 (dt, *J* = 6.9, 1.8 Hz, 2H), 7.81 (dt, *J* = 6.9, 1.8 Hz, 2H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.57 (*J* = 2.7 Hz, 1H), 6.98 (*J* = 9.0, 2.7 Hz, 1H), 3.90 (s, 3H); ¹³C NMR (CDCl₃) δ 163.4, 154.5, 145.4, 133.5, 124.3, 124.0, 118.9, 118.3, 115.9, 113.9, 107.2, 56.2; IR (KBr) 3017, 2976, 2938, 2225, 1479, 1432 cm⁻¹; MS (APCI) *m/z* (%) 364.0 (40, M⁺+H) 235.9 (30, M⁺-I¹²⁷), 129.9 (100, M⁺-C₇H₇IO).

(2-Iodo-4-methoxyphenyl)(4-nitrophenyl)diazene (29). 4-Nitroaniline (1.5 g, 10.86 mmol), conc. HCl (1.80 mL, 21.7 mmol), NaNO₂ (0.90 g, 13.03 mmol), KOH (1.22 g, 21.7 mmol), and 3-iodophenol (2.63 g, 11.95 mmol) were reacted according to general procedure C, part 1. The resultant dark red solid (3.5 g, 9.48 mmol), Cs₂CO₃ (7.56 g, 23.23 mmol), and MeI (0.65 mL, 10.43 mmol) in DMF (21 mL) were reacted according to general procedure C, part 2. Recrystallization from EtOH/H₂O gave **29** (1.91 g, 53%) as a dark red powder: mp 127.5-128.6 °C; ¹H NMR (CDCl₃) δ 8.38 (dt, *J* = 5.4, 1.8 Hz, 2H), 8.06 (dt, *J* = 5.4, 1.8 Hz, 2H), 7.75 (d, *J* =

9.0 Hz, 1H), 7.58 (d, $J = 2.7$ Hz, 1H) 7.00 (dd, $J = 9.0, 2.7$ Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (CDCl_3) δ 163.3, 155.38, 148.5, 145.2, 124.8, 124.1, 123.8, 118.1, 115.6, 107.3, 56.0; IR (KBr) 3094, 2910, 1587, 1481 cm^{-1} ; MS (APCI) m/z (%) 383.9 (100, $\text{M}^+ + \text{H}$), 338.0 (80, $\text{M}^+ - \text{NO}_2$), 150.1 (20, $\text{M}^+ - \text{C}_7\text{H}_6\text{IO}$).

[4-Methoxy-2-(triethylsilylethynyl)phenyl]phenyldiazene (30). Iododiazene **24** (0.75 g, 2.21 mmol), TESA (0.56 mL, 3.10 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (0.03 g, 0.044 mmol), and CuI (0.042 g, 0.221 mmol) were reacted according to general procedure D. Chromatography on silica gel (10-20% CH_2Cl_2 gradient in hexanes) yielded **30** (0.520 g, 67%) as a viscous red oil: ^1H NMR (CDCl_3) 7.97-7.94 (m, 2H), 7.76 (d, $J = 9.0$ Hz, 1H), 7.51-7.43 (m, 3H), 7.12 (d, $J = 2.7$ Hz, 1H), 6.94 (dd, $J = 9.0$ Hz, 2.7 Hz, 1H), 3.89 (s, 3H), 1.07 (t, $J = 8.1$ Hz, 9H), 0.72 (q, $J = 8.1$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 161.3, 147.78, 130.6, 128.9, 126.0, 123.1, 122.3, 117.1, 116.9, 116.4, 103.1, 98.7, 55.7, 7.6, 4.4; IR (neat) 2955, 2910, 2154, 1596, 1461 cm^{-1} ; MS (ESI) m/z (%) 351.3 (100, $\text{M}^+ + \text{H}$) 323.2 (30, $\text{M}^+ - \text{C}_2\text{H}_3$), 309.2 (40, $\text{M}^+ - \text{C}_3\text{H}_5$).

(4-Bromophenyl)[4-methoxy-2-(triethylsilylethynyl)phenyl]diazene (31). Iododiazene **25** (1.50 g, 3.60 mmol), TESA (0.87 mL, 4.86 mmol), $\text{PdCl}_2(\text{dppf})$ (0.15 g, 0.18 mmol), CuI (0.07 g, 0.36 mmol), and THF/DIPA (1:1) were reacted according to general procedure D. Chromatography on silica gel (9:1 hexanes/ Et_2O) afforded **31** (1.43 g, 92%) as a red viscous oil: ^1H NMR (CDCl_3) δ 7.84 (dt, $J = 8.4, 2.1$ Hz, 2H), 7.76 (d, $J = 8.4$ Hz, 1H), 7.62 ($J = 8.4, 2.1$ Hz, 2H), 7.14 (d, $J = 2.1$ Hz, 1H), 6.95 (dd, $J = 8.4, 2.1$ Hz, 1H), 3.91 (s, 3H), 1.10 (t, $J = 7.8$ Hz, 9H), 0.74 (q, $J = 7.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 161.7, 151.7, 147.6, 132.7, 132.1, 126.4, 124.5, 117.1, 116.9, 116.4, 102.9, 99.0, 55.7, 7.6, 4.4; IR (neat) 2964, 2907, 2155, 1596, 1487 cm^{-1} ; MS (APCI) m/z (%) 429.0 (60, $\text{M}^+ + \text{H}$).

(4-Fluorophenyl)[4-methoxy-2-(triethylsilylethynyl)phenyl]diazene (32). Iododiazene **26** (1.50 g, 4.21 mmol), TESA (1.01 mL, 5.68 mmol), $\text{PdCl}_2(\text{dppf})$ (0.16 g, 0.19 mmol), CuI (0.07 g, 0.38 mmol) and THF/DIPA (1:1) were reacted according to general procedure D. Chromatography on silica gel (7:1 hexanes: Et_2O) gave **32** (1.21 g, 78%) as a viscous bright red oil: ^1H NMR (CDCl_3) δ 8.02-7.95 (m, 2H), 7.75 (d, $J = 9.0$ Hz, 1H), 7.18 (m, 3H), 6.95 (dd, $J =$

9.0, 3.0 Hz, 1H), 3.90, (s, 3H), 1.11 (t, $J = 7.8$ Hz, 9H), 0.74 (q, $J = 7.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 164.2 (d, $J = 999.0$ Hz) 161.4, 149.5 (d, $J = 15.9$ Hz) 147.6, 126.0, 125.0 (d, $J = 31.8$ Hz), 117.1, 116.9, 116.4, 115.7 (d, $J = 88.2$), 103.0, 98.8, 55.7, 7.55, 4.4; IR (KBr) 3004, 2830, 1548, 1496 cm^{-1} ; MS (ESI) m/z 369.1 (100, $\text{M}^+ + \text{H}$).

(4-Carbomethoxyphenyl)[4-methoxy-2-(triethylsilylethynyl)phenyl]diazene (33).

Iododiazene **27** (1.50 g, 3.79 mmol), TESA (0.91 mL, 5.12 mmol), $\text{PdCl}_2(\text{dppf})$ (0.15 g, 0.190 mmol), and CuI (0.07 g, 0.38 mmol) in THF/DIPA (1:1) were reacted according to general procedure D. Recrystallization from EtOH/ H_2O furnished **33** (1.34 g, 86%) as a dark red solid: mp 54.4-55.2 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 8.17 (dt, $J = 8.7$, 1.8 Hz, 2H), 8.00 (dt, $J = 8.7$, 1.8 Hz, 2H), 7.79 (d, $J = 8.7$ Hz, 1H), 7.15 (d, $J = 2.2$ Hz, 1H), 6.96 (dd, $J = 8.7$, 2.2 Hz, 1H), 3.98 (s, 3H), 3.91 (s, 3H), 1.10 (t, $J = 7.8$ Hz, 9H), 0.76 (q, $J = 7.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 166.6, 162.0, 155.4, 147.7, 131.3, 130.4, 126.9, 122.9, 117.1, 117.0, 116.4, 102.8, 99.3, 55.7, 52.3, 7.3, 4.4; IR (KBr) 2942, 2870, 2151, 2067, 1723, 1590 cm^{-1} ; MS (APCI) m/z (%) 409.2 (100, $\text{M}^+ + \text{H}$), 394.1 (15, $\text{M}^+ - \text{CH}_3$), 351.1 (20, $\text{M}^+ - \text{CO}_2\text{Me}$);

(4-Cyanophenyl)[4-methoxy-2-(triethylsilylethynyl)phenyl]diazene (34). Iododiazene **28** (1.0 g, 2.75 mmol), TESA (0.69 mL, 3.86 mmol), $\text{PdCl}_2(\text{dppf})$ (0.05 g, 0.07 mmol), and CuI (0.03 g, 0.14 mmol) in THF/DIPA (1:1) were reacted according to general procedure D. Chromatography on silica gel (4:1 hexanes/ Et_2O) gave **34** (0.721 g, 66%) as a bright orange-red solid: mp 80.9-81.5 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 8.01 (dt, $J = 8.4$, 1.8 Hz, 2H), 7.81-7.77 (m, 3H), 7.15 (d, $J = 3.0$ Hz, 1H), 6.96 (dd, $J = 9.0$, 3.0 Hz, 1H), 3.92 (s, 3H), 1.10 (t, $J = 7.8$ Hz, 9H), 0.74 (q, $J = 7.8$ Hz); ^{13}C NMR (CDCl_3) δ 162.4, 154.9, 147.4, 133.0, 127.4, 123.5, 118.6, 117.2, 117.01, 116.5, 113.4, 102.6, 99.7, 55.8, 7.6, 4.4; IR (KBr) 2951, 2872, 2218, 2150, 1593 cm^{-1} ; MS (ESI) m/z (%) 376.1 (100, $\text{M}^+ + \text{H}$), 348.1 (30, $\text{M}^+ - \text{C}_2\text{H}_3$), 334.1 (40, $\text{M}^+ - \text{C}_3\text{H}_5$).

[4-Methoxy-2-(triethylsilylethynyl)phenyl](4-nitrophenyl)diazene (35). Iododiazene **29** (0.75 g, 1.96 mmol), TESA (0.49 mL, 2.74 mmol), $\text{PdCl}_2(\text{dppf})$ (0.08 g, 0.1 mmol), and CuI (0.04 g, 0.196 mmol) in THF/DIPA (1:1) were reacted according to general procedure D. Chromatography on silica gel (4:1 hexanes/ Et_2O) afforded **35** (0.503 g, 65%) as a bright orange-

red solid: mp 81.8-83.6 °C; ^1H NMR (CDCl_3) δ 8.34 (dt, $J = 9.0, 2.4$ Hz, 2H), 8.04 (dt, $J = 9.0, 2.4$ Hz, 2H), 7.80 (d, $J = 9.3$ Hz, 1H), 7.15 (d, $J = 2.7$ Hz, 1H), 6.95 (dd, $J = 9.3, 2.7$ Hz, 1H), 3.92 (s, 3H), 1.08 (t, $J = 7.8$ Hz, 9H), 0.74 (q, $J = 7.8$ Hz, 6H); ^{13}C NMR (CDCl_3) δ 162.7, 156.1, 148.4, 147.5, 127.7, 124.6, 123.5, 117.2, 117.1, 116.6, 102.6, 100.0, 55.8, 7.6, 4.4; IR (KBr) 2955, 2912, 2155, 2064, 1591 cm^{-1} ; (ESI) m/z (%) 396.1 (100, $\text{M}^+ + \text{H}$).

3-Hydroxymethyl-2-phenyl-2*H*-indazole (36). Diazene **8** (0.166 g, 0.52 mmol) was deprotected with TBAF (1.04 mL, 1.04 mmol), cyclized with TESOH (0.20 mL, 1.3 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo afforded isoindazole **36** (67 mg, 75%) as a light tan solid: mp 150.5-151.8 °C; ^1H NMR (CDCl_3) δ 7.76-7.67 (m, 4H), 7.54-7.46 (m, 3H), 7.34 (td, $J = 8.1, 0.9$ Hz, 1H), 7.12 (td, $J = 8.1, 0.9$ Hz, 1H), 4.96 (s, 2H), 2.56 (br s, 1H); ^{13}C NMR (CDCl_3) δ 148.5, 139.4, 133.8, 129.2, 128.9, 126.9, 125.4, 122.2, 121.8, 119.4, 117.8, 54.1; IR (KBr) 3183, 3060, 1595, 1503 cm^{-1} ; MS (ESI) m/z (%) 225.1 (100, $\text{M}^+ + \text{H}$), 207.2 (35, $\text{M}^+ - \text{OH}$), 149.9 (100, $\text{M}^+ - \text{N}_2$).

3-Hydroxymethyl-5-methyl-2-phenyl-2*H*-indazole (37). Diazene **14** (0.233 g, 0.70 mmol) was deprotected with TBAF (1.4 mL, 1.4 mmol), cyclized with TESOH (0.32 mL, 2.09 mmol) and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo afforded isoindazole **37** (0.135 g, 81%) as a light yellow solid: mp 164.4-165.5 °C; ^1H NMR (CDCl_3) δ 7.69-7.65 (m, 3H), 7.52-7.42 (m, 4H), 7.17 (dd, $J = 9.0, 1.5$ Hz, 1H), 4.92 (s, 2H), 2.56 (br s, 1H), 2.42 (s, 3H); ^{13}C NMR (CDCl_3) δ 147.5, 139.5, 132.9, 131.7, 129.8, 129.2, 128.7, 125.4, 122.1, 117.4, 117.4, 54.0, 21.8; IR (KBr) 3215, 3054, 2916, 1535, 1504 cm^{-1} ; MS (ESI) m/z (%) 239.1 (65, $\text{M}^+ + \text{H}$), 221.1 (35, $\text{M}^+ - \text{OH}$).

5-*tert*-Butyl-3-hydroxymethyl-2-phenyl-2*H*-indazole (38). Diazene **15** (0.143 g, 0.378 mmol) was deprotected with TBAF (0.756 mL, 0.756 mmol), cyclized with TESOH (0.144 mL, 0.945 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo furnished isoindazole **38** (63

mg, 80%) as a tan solid: mp 178.4-179.7 °C; ^1H NMR (CDCl_3) δ 7.75-7.70 (m, 3H), 7.62 (d, J = 0.9 Hz, 1H), 7.56-7.46 (m, 4H), 5.00 (s, 2H), 2.10 (br s, 1 H), 1.39 (s, 9H); ^{13}C NMR (CDCl_3) δ 147.4, 145.2, 139.6, 133.5, 129.3, 128.8, 126.8, 125.5, 121.7, 117.5, 113.3, 54.1, 35.0, 31.2; IR (KBr) 3216, 2957, 2858, 1596, 1503 cm^{-1} ; MS (ESI) m/z (%) 281.3 (100, $\text{M}^+\text{+H}$), 263.1 (15, $\text{M}^+\text{--OH}$), 235.1 (20, $\text{M}^+\text{--C}_3\text{H}_9$).

5-Chloro-3-hydroxymethyl-2-phenyl-2H-indazole (39). Diazene **27** (0.50 g, 1.40 mmol), was deprotected with TBAF (2.8 mL, 2.8 mmol), cyclized with TESOH (0.68 mL, 4.2 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo afforded isoindazole **39** (0.296 g, 82%) as a dark brown solid: mp 172.4-173.7 °C; ^1H NMR (CDCl_3) δ 7.75-7.68 (m, 4H) 7.58-7.50 (m, 3H), 7.27 (dd, J = 9.0, 2.1 Hz, 1H) 4.97 (s, 2H), 2.07 (br s, 1H); ^{13}C NMR (CDCl_3) δ 158.5, 152.1, 139.3, 136.1, 129.4, 129.2, 128.3, 128.0, 125.4, 119.5, 118.3, 54.3; IR (KBr) 3218, 3063, 2920, 1596, 1501 cm^{-1} ; MS (ESI) m/z (%) 259.0 (85, $\text{M}^+\text{+H}$), 241.1 (45, $\text{M}^+\text{--OH}$),

5-Ethynyl-3-hydroxymethyl-2-phenyl-2H-indazole (40). Diazene **30** (0.34 g, 0.74 mmol) was deprotected with TBAF (2.96 mL, 2.96 mmol), cyclized with TESOH (0.36 mL, 2.22 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo afforded isoindazole **40** (0.147 g, 80%) as a yellow solid: mp 136.2-137.4 °C; ^1H NMR (CDCl_3) δ 7.88 (s, 1H), 7.67-7.64 (m, 3H), 7.53-7.42 (m, 3H), 7.37 (d, J = 9.0 Hz, 1H), 4.91 (s, 2H), 3.07 (s, 1H), 2.64 (br s, 1H); ^{13}C NMR (CDCl_3) δ 147.7, 139.1, 134.4, 130.0, 129.3, 129.2, 125.3, 124.6, 121.4, 117.9, 115.7, 84.4, 76.5, 54.1; IR (KBr) 3298, 3235, 2924, 2101, 1598, 1504 cm^{-1} ; MS (ESI) m/z (%) 249.1 ($\text{M}^+\text{+H}$), 231.1 (30, $\text{M}^+\text{--OH}$), 219.1 ($\text{M}^+\text{--CH}_2\text{O}$).

3-Hydroxymethyl-5-methoxy-2-phenyl-2H-indazole (41). Diazene **30** (0.41 g, 1.17 mmol) was deprotected with TBAF (2.34 mL, 2.34 mmol), cyclized with TESOH (0.54 mL, 3.51 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (EtOAc) and concentration in vacuo afforded isoindazole **41** (0.231 g, 79%) as a light brown solid: mp 184.4-185.4 °C; ^1H NMR (CDCl_3) δ 7.73-7.66 (m, 3H), 7.56-7.48 (m,

3H), 7.07 (dd, $J = 9.0, 2.1$ Hz, 1H), 6.95 (d, $J = 2.1$ Hz, 1H), 4.96 (s, 2H), 3.87 (s, 3H), 1.72 (br s, 1H); ^{13}C NMR (CDCl_3) δ 155.5, 145.4, 139.6, 132.6, 129.2, 128.7, 125.3, 122.0, 121.9, 119.3, 95.3, 55.4, 54.2; IR (KBr) 3218, 3051, 2826, 1537, 1506 cm^{-1} ; MS (APCI) m/z (%) 255.1 (100, $\text{M}^+ + \text{H}$), 237.2 (30, $\text{M}^+ - \text{OH}$).

2-(4-Bromophenyl)-3-hydroxymethyl-5-methoxy-2H-indazole (42). Diazene **31** (0.43 g, 1.0 mmol) was desilylated with TBAF (2.0 mL, 2.0 mmol), cyclized with TESOH (0.487 mL, 3.0 mmol) and deprotected with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (Et_2O), and concentration in vacuo gave **42** (0.268 g, 63%) as a light brown solid. mp 162.7-164.4 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.63-7.62 (m, 5H), 7.04 (dd, $J = 9.0, 2.1$ Hz, 1H), 6.83 (d, $J = 2.1$ Hz, 1H), 4.92 (s, 2H), 3.84 (s, 3H), 2.38 (br s, 1H); ^{13}C NMR (CDCl_3) δ 153.9, 145.7, 133.25, 132.9, 132.6, 126.8, 125.1, 122.8, 122.7, 119.4, 95.5, 55.7, 54.1; IR (KBr) 3203, 2923, 1637, 1516 cm^{-1} ; MS (APCI) m/z (%) 333.1 (100, $\text{M}^+ + \text{H}$), 317.0 (30, $\text{M}^+ - \text{CH}_3$), 319.4 (25, $\text{M}^+ - \text{OH}$).

2-(4-Fluorophenyl)-3-hydroxymethyl-5-methoxy-2H-indazole (43). Diazene **32** (0.46 g, 1.24 mmol) was deprotected with TBAF (2.48 mL, 2.48 mmol), cyclized with TESOH (0.57 mL, 3.72 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (Et_2O) and concentration in vacuo furnished **43** (0.264 g, 78%) as a light brown solid: mp 191.6-192.2 $^\circ\text{C}$; ^1H NMR (CDCl_3) δ 7.75-7.70 (m, 2H), 7.64 (d, $J = 9.6$ Hz, 1H), 7.24-7.18 (m, 2H), 7.05 (dd, $J = 9.6, 2.4$ Hz, 1H), 6.89 (d, $J = 2.4$ Hz, 1H), 4.95 (s, 2H), 3.86 (s, 3H), 2.20 (br s, 1H); ^{13}C NMR (CDCl_3) δ 162.5 (d, $J = 990$ Hz), 155.7, 145.4, 132.5 (d, $J = 30.6$ Hz), 127.1 (d, $J = 32.1$ Hz), 126.6, 122.2, 121.9, 119.3, 116.2 (d, $J = 92.1$ Hz), 95.2, 55.4, 54.0; IR (KBr) 3203, 2923, 1516 cm^{-1} ; MS (APCI) m/z (%) 273.2 (100, $\text{M}^+ + \text{H}$), 255.1 (25, $\text{M}^+ - \text{OH}$), 243.1 (45, $\text{M}^+ - \text{CHO}$).

4-(3-Hydroxymethyl-5-methoxy-indazol-2-yl)-benzoic acid methyl ester (44). Diazene **33** (0.45 g, 1.10 mmol) was desilylated with TBAF (2.20 mL, 2.20 mmol), cyclized with and TESOH (0.50 mL, 3.3 mmol), and deprotected with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (Et_2O), and concentration in vacuo **44** (0.247 g,

72%) was obtained as a light brown solid. mp 195.4-197.3 °C (dec); ¹H NMR (CDCl₃, 300MHz) δ 8.20 (dt, *J* = 8.7, 2.1 Hz, 2H) 7.90 (dt, *J* = 8.7, 2.1 Hz, 2H), 7.65 (d, *J* = 9.3 Hz, 1H), 7.06 (dd, *J* = 9.3, 2.1 Hz, 1H), 6.89 (d, *J* = 2.1 Hz, 1H), 5.00 (s, 2H), 3.97 (s, 3H), 3.88 (s, 3H), 2.08 (bs, 1H); ¹³C NMR (CDCl₃, 75 MHz) δ 166.3, 155.8, 145.9, 143.3, 132.4, 130.7, 129.9, 124.8, 122.7, 119.5, 95.0, 77.2, 55.4, 54.0, 52.4. IR (KBr) 3422, 2945, 1723, 1703, 1606 cm⁻¹; MS (APCI) *m/z* (%) 313.1 (100, M⁺ +H), 295.0 (35, M⁺ -OH), 236.1 (40, M⁺ -C₆H₄).

2-(4-Cyanophenyl)-3-hydroxymethyl-5-methoxy-2*H*-indazole (45). Diazene **34** (0.395 g, 1.0 mmol) was deprotected with TBAF (2.0 mL, 2.0 mmol), cyclized with TESOH (0.46 mL, 3.0 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (Et₂O) and concentration in vacuo afforded **45** (0.21 g, 73%) as a light brown solid: mp 217.0-218.8 °C (dec); ¹H NMR (CDCl₃) δ 8.05 (dt, *J* = 8.7, 2.1 Hz, 2H), 7.85 (dt, *J* = 8.7, 2.1 Hz, 2H), 7.65 (d, *J* = 9.0, 1H), 7.06 (dd, *J* = 9.0, 2.1 Hz, 1H), 6.87 (d, *J* = 2.1 Hz, 1H), 5.00 (s, 2H), 3.89 (s, 3H), 2.01 (br s, 1H); ¹³C NMR (C₄D₈O) δ 156.7, 147.2, 144.2, 135.0, 134.1, 125.9, 123.9, 123.5, 120.2, 118.9, 112.7, 96.5 55.7, 53.6; IR (KBr) 3205, 2825, 2234, 1536, 1218 cm⁻¹; MS (APCI) *m/z* (%) 280.2 (100, M⁺+H), 262.2 (25, M⁺-OH), 250.1 (60, M⁺-CHO).

3-Hydroxymethyl-5-methoxy-2-(4-nitrophenyl)-2*H*-indazole (46). Diazene **35** (0.31 g, 0.80 mmol) was deprotected with TBAF (1.6 mL, 1.6 mmol), cyclized with TESOH (0.37 mL, 2.39 mmol), and desilylated with xs TBAF according to general procedure E. After aqueous work-up, filtration over silica (Et₂O) and concentration in vacuo gave **46** (0.177 g, 54%) as a light brown solid: mp 204.6-205.4 °C; ¹H NMR (CDCl₃) δ 8.36 (dt, *J* = 9.0, 2.1 Hz, 2H), 8.05 (dt, *J* = 9.0, 2.1 Hz, 2H), 7.63 (d, *J* = 9.3 Hz, 1H), 7.05 (dd, *J* = 9.3, 2.1 Hz, 1H), 6.80 (d, *J* = 2.1 Hz, 1H), 4.98 (d, *J* = 5.1 Hz, 2H), 3.84 (s, 3H), 2.51 (t, *J* = 5.1 Hz, 1H); ¹³C NMR (CDCl₃) δ 156.1, 146.9, 146.2, 144.5, 132.6, 125.1, 124.8, 123.4, 122., 119.4, 94.8, 55.4, 53.6; IR (KBr) 3534, 3080, 2928, 1596 cm⁻¹; MS (ESI) *m/z* (%) 300.2 (100, M⁺+H), 282.0 (15, M⁺-OH), 254.0 (60, M⁺+H-NO₂).

5-Chloro-3-(2,2,3,3-tetramethyl-cyclopropyl)-2-phenyl-2*H*-indazole (47). Diazene **16** (0.34 g, 0.96 mmol) was desilylated with TBAF (1.92 mL, 1.92 mmol) according to the first step of general procedure E. Upon concentration the deprotected alkyne was dissolved in 2,3-dimethyl-2-butene (20 mL) and purged with Ar (20 min). The solution was placed in a preheated 75 °C sand bath and stirred under Ar for 8 h. After cooling, the solvent was removed and the black oil was dissolved in 5:1 hexanes/CH₂Cl₂ and filtered over silica to yield the cyclopropane **47** (0.234 g, 75%) as a dark brown solid: mp 157.3-158 °C (dec); ¹H NMR (CDCl₃) δ 7.68-7.63 (m, 3H), 7.56-7.45 (m, 4H), 7.22 (dd, *J* = 9.3, 1.8 Hz, 1H), 1.72 (s, 1H), 1.20 (s, 6H), 0.56 (s, 6H); ¹³C NMR (CDCl₃) δ 147.3, 141.0, 134.4, 128.8, 128.3, 127.8, 126.2, 125.2, 123.7, 120.2, 119.3, 100.0, 30.0, 24.7, 23.0, 19.0; IR (KBr) 2979, 2921, 1596, 1498 cm⁻¹; MS (APCI) *m/z* (%) 325.0 (100, M⁺+H), 309.0 (25, M⁺-CH₃), 241.1 (M⁺-C₆H₁₁).

Cartesian Coordinates for all structures in Figure 1

4 – Conformation 1

N 0.101450 0.006610 -0.177560
C -0.346360 0.079330 -1.516970
C -1.055380 0.222740 -4.222960
C 0.665400 0.110040 -2.508200
C -1.697760 0.120900 -1.895440
C -2.050090 0.191970 -3.236430
C 0.286570 0.182220 -3.862470
C 2.052540 0.069870 -2.170130
H -2.448340 0.095860 -1.113560
H -3.098560 0.223720 -3.520000
H 1.062720 0.205660 -4.620600
H -1.329960 0.278350 -5.272670
N -0.815070 -0.019190 0.688220
C 3.244550 0.040130 -1.964650
H 4.290240 0.012240 -1.758440
C -0.357830 -0.092040 2.027680
C -1.361200 -0.121900 3.005530
C -1.022250 -0.193230 4.355640
C 0.321180 -0.234940 4.732460
C 1.325590 -0.205160 3.755720
C 0.995550 -0.134110 2.407800
H -2.396860 -0.088080 2.681030
H -1.802860 -0.216200 5.110920
H 0.589600 -0.290560 5.784070
H 2.370230 -0.237830 4.054040
H 1.756040 -0.109970 1.635120

4 Conformation 2

N -0.726380 0.084920 0.380200
C -0.150100 0.188990 1.665700
C 0.654340 0.464360 4.342540
C 1.210940 -0.040270 2.013970
C -1.072750 0.521060 2.669420
C -0.682530 0.672960 3.994680
C 1.578920 0.105300 3.365940
C 2.230190 -0.454700 1.103440
H -2.104590 0.665470 2.364950
H -1.413480 0.943980 4.750920
H 2.614610 -0.076500 3.635400
H 0.976740 0.569500 5.374640
N 0.084380 0.108270 -0.583980
C 3.192940 -0.834750 0.474690
H 4.004830 -1.159910 -0.134920
C -0.511520 -0.024000 -1.863950
C 0.375310 0.059710 -2.945830
C -0.099350 -0.056110 -4.251190
C -1.461690 -0.258140 -4.479480
C -2.349290 -0.344280 -3.398800
C -1.883020 -0.229140 -2.094640
H 1.428450 0.216100 -2.733860
H 0.590630 0.010330 -5.087780
H -1.835290 -0.350100 -5.495840
H -3.408920 -0.503420 -3.581050
H -2.553400 -0.293670 -1.244870

Pseudocoarctate TS conformation 1

N -1.585071 -0.174047 -0.946318
C -1.006323 -0.071821 0.312544
C 0.359266 0.129549 2.698032
C 0.404754 -0.027732 0.298954
C -1.746823 -0.016678 1.502075
C -1.051087 0.082918 2.697315
C 1.091073 0.074972 1.517169
C 0.969942 -0.077229 -1.017779
H -2.831172 -0.053974 1.465342
H -1.590450 0.127127 3.638763
H 2.175544 0.115420 1.512598
H 0.884915 0.212142 3.645687
N -0.659456 -0.261241 -1.812505
C 2.130706 0.039067 -1.582448
H 2.321261 0.007080 -2.650676
C -0.973343 -0.292957 -3.184157
C -0.105035 -0.984088 -4.041447
C -0.372841 -1.011981 -5.408865
C -1.492840 -0.353820 -5.920878
C -2.355166 0.333527 -5.059736
C -2.100529 0.370622 -3.692941
H 0.743374 -1.513549 -3.624347
H 0.292064 -1.555158 -6.074232
H -1.696034 -0.376502 -6.987797
H -3.226556 0.846178 -5.457502
H -2.751062 0.906125 -3.010031

Pseudocoarctate TS conformation 2

H -4.871870 -1.917200 -0.152300
C -4.046880 -1.213430 -0.096230
C -2.736820 -1.670430 -0.149960
C -4.310360 0.163650 0.030620
C -3.279900 1.096790 0.103860
H -5.340890 0.506030 0.072300
H -2.496450 -2.724570 -0.248670
C -1.950350 0.653570 0.054570
C -1.704550 -0.728340 -0.073530
H -3.492590 2.157250 0.201820
N -0.356910 -1.073410 -0.122120
C -0.778410 1.476280 0.122510
N 0.344670 -0.021500 -0.061530
C -0.218840 2.625200 0.243620
H -0.759530 3.556630 0.385160
C 1.751310 -0.129430 -0.032220
C 2.507250 1.021080 -0.304370
C 3.898340 0.939090 -0.282700
C 4.532960 -0.270770 0.004190
C 3.771390 -1.412470 0.280210
C 2.383350 -1.349200 0.267510
H 1.988000 1.951540 -0.500390
H 4.488280 1.826680 -0.493240
H 5.618060 -0.327050 0.017210
H 4.264070 -2.353230 0.510020
H 1.777580 -2.221320 0.485240

Isoindazole carbene conformation 1

H -0.708250 -0.002210 5.219550
C -0.270360 0.030210 4.225980
C -1.084970 -0.065310 3.114950
C 1.138810 0.171980 4.099840
C 1.760830 0.223500 2.863700
H 1.738110 0.240060 5.003850
H -2.162300 -0.171430 3.192740
C 0.949800 0.123090 1.723400
C -0.453480 -0.015550 1.854320
H 2.834820 0.335550 2.755690
N -1.048320 -0.109760 0.627140
C 1.233850 0.155420 0.317040
N -0.080420 -0.009030 -0.255610
C 2.460690 0.395220 -0.281100
H 2.339490 0.512840 -1.373060
C -0.403570 -0.081310 -1.641120
C 0.392160 -0.838170 -2.507430
C 0.062780 -0.890950 -3.861960
C -1.053380 -0.206100 -4.344010
C -1.851010 0.532990 -3.465900
C -1.529650 0.601020 -2.112980
H 1.231230 -1.402720 -2.117960
H 0.675840 -1.480960 -4.536880
H -1.306250 -0.253200 -5.399370
H -2.723460 1.063600 -3.836020
H -2.133000 1.173740 -1.417510

Isoindazole carbene conformation 2

H -0.848440 0.147770 5.182600
C -0.371990 0.129670 4.206720
C -1.147110 0.048010 3.064870
C 1.043360 0.190800 4.130400
C 1.711530 0.172730 2.915410
H 1.614530 0.251650 5.052800
H -2.230860 0.001550 3.102020
C 0.942690 0.090570 1.745200
C -0.467590 0.030450 1.831260
H 2.795870 0.220190 2.867270
N -1.025250 -0.059860 0.585080
C 1.264470 0.054320 0.341320
N -0.032500 -0.042610 -0.269440
C 2.423910 0.143180 -0.407830
H 3.267700 0.195130 0.306980
C -0.350450 -0.084810 -1.664940
C 0.512330 -0.688050 -2.587340
C 0.147160 -0.718020 -3.932720
C -1.063180 -0.168070 -4.356840
C -1.919210 0.424910 -3.425500
C -1.567630 0.470870 -2.079810
H 1.459130 -1.086310 -2.250910
H 0.816320 -1.183030 -4.650900
H -1.339160 -0.200530 -5.407210
H -2.861490 0.859710 -3.746420
H -2.218920 0.931880 -1.346870

TS (carbene conformation 2 to 5)

H 4.267260 9.319660 0.481710
C 3.340780 9.885350 0.523740
C 2.131410 9.214680 0.547730
C -5.261100 10.603330 0.474340
C -4.557280 11.815190 0.462770
H -6.344760 10.603050 0.381310
H 2.073240 8.131100 0.533780
C -3.168210 11.809370 0.457840
C 0.956450 9.991300 0.601820
H -5.103800 12.753830 0.395970
N -0.330590 9.541980 0.579330
C -2.493670 10.589560 0.603830
N -1.081720 10.628360 0.704700
C -0.997420 13.036300 0.980560
H -2.265940 12.811770 0.849590
C -0.353820 11.835680 0.750290
C 1.014820 11.414970 0.638030
C 2.251530 12.074240 0.626570
C 3.401620 11.300910 0.555780
C -4.571200 9.390420 0.585660
C -3.182470 9.370700 0.675520
H -0.485850 13.923390 0.589410
H 2.310500 13.158440 0.660230
H 4.372160 11.787620 0.521510
H -5.121740 8.454560 0.622990
H -2.631950 8.447950 0.825950

Tetracycline 5

H 4.399465 9.709472 0.611983
C 3.401096 10.138950 0.612379
C 2.304784 9.306952 0.612743
C -5.130942 10.917280 0.612329
C -4.199986 11.966430 0.612496
H -6.193029 11.144850 0.611887
H 2.404984 8.225908 0.612662
C -2.846343 11.661400 0.612992
C 1.016991 9.897958 0.613214
H -4.537645 12.999670 0.612168
N -0.179900 9.261176 0.613517
C -2.451491 10.313050 0.613414
N -1.044773 10.286020 0.614084
C -1.610345 12.556920 0.613509
H -1.579605 13.211750 1.494771
C -0.503793 11.537630 0.614228
C 0.878721 11.339950 0.613319
C 2.031324 12.165410 0.613097
C 3.267272 11.559920 0.612553
C -4.709913 9.583951 0.612727
C -3.349471 9.256428 0.613319
H -1.578378 13.211520 -0.267860
H 1.940155 13.248310 0.613284
H 4.165889 12.170860 0.612322
H -5.448521 8.787421 0.612598
H -2.999911 8.229923 0.613639

(Pseudo)pericyclic TS to cinnolinium zwitterion

N -0.300130 -0.625000 0.187880
C -1.678420 -0.393430 0.144620
C -4.467300 -0.302490 -0.047750
C -2.317510 0.838990 -0.174180
C -2.448600 -1.555770 0.366790
C -3.829460 -1.510170 0.286600
C -3.721700 0.844850 -0.283370
C -1.529520 2.022240 -0.392780
H -1.919990 -2.474700 0.603810
H -4.417170 -2.405050 0.469890
H -4.204230 1.782630 -0.542470
H -5.550910 -0.268450 -0.124180
N 0.500940 0.356570 0.152870
C -0.271760 2.117370 -0.291280
H 0.610620 2.727210 -0.307210
C 1.880520 0.023540 0.078520
C 2.794940 0.963840 0.571960
C 4.162200 0.704400 0.499220
C 4.620280 -0.482650 -0.076490
C 3.705200 -1.416010 -0.574950
C 2.337830 -1.169720 -0.500440
H 2.425950 1.873600 1.035630
H 4.869500 1.428740 0.893060
H 5.686750 -0.679740 -0.140370
H 4.061700 -2.337180 -1.027920
H 1.612000 -1.878070 -0.884190

Cinnolinium Zwitterion

N -0.628210 0.047660 -0.482180
C -0.138360 0.024890 -1.739360
C 0.772920 0.263190 -4.374390
C 1.274530 -0.047290 -2.006160
C -1.079720 0.168130 -2.802920
C -0.623330 0.278570 -4.091220
C 1.689480 0.110080 -3.361700
H 2.104830 -0.681850 1.185250
H -2.138300 0.193920 -2.561870
H -1.330910 0.387320 -4.908960
H 2.757540 0.074290 -3.554840
H 1.104840 0.367270 -5.404010
N 0.251610 -0.135730 0.485050
C 1.591570 -0.384800 0.273250
C 2.219480 -0.282200 -0.964210
C -0.292390 -0.061890 1.819170
C -1.557500 -0.598410 2.073690
C -2.068120 -0.552180 3.368400
C -1.325230 0.024930 4.401930
C -0.067220 0.565620 4.134230
C 0.455420 0.528280 2.841230
H -2.119860 -1.040600 1.259910
H -3.048800 -0.973010 3.570910
H -1.728560 0.057470 5.409930
H 0.508880 1.030740 4.928790
H 1.417420 0.979400 2.623120

Crystal structural analysis

Crystals were used as supplied. A needle of dimensions $0.044 \times 0.08 \times 0.36$ mm was mounted in a fiber loop under oil and transferred to a Bruker SMART APEX diffractometer fitted with a CCD area detector. A total of 2400 30-second frames were collected at 100(2) K with a scan width of 0.3° on ω or ϕ (software: SMART¹). The cell dimensions were refined from the coordinates of 4231 reflections in the range $5.1^\circ \leq \theta \leq 65.9^\circ$ (software: SAINT²). The following table contains a summary of crystal data and the final residuals. A more extensive table including particulars of data collection and structure refinement is in the supplementary material. The systematic absences and 2/m Laue symmetry indicated the space-group $P2_1/c$. Absorption corrections were not required. A SIR92 E-map³ showed all non-hydrogen atoms. The final difference synthesis was featureless. The TEXSAN program suite⁴ was used in all calculations.

References

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2. Bruker AXS Inc., Madison, Wisconsin, USA. SAINT software for cell determination and data reduction, 2001.
3. Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, N. *J. Appl. Crystallography*, 1994, 27, 435.
4. Molecular Structures Corporation, 3200A Research Forest Drive, The Woodlands, TX 77381, USA. *Texsan Software for Single-Crystal Structure Analysis*, version 1.7, 1997.

Crystallographic data

Composition	$C_{28}H_{18}N_4$	d_{calc}	1.418 g cm^{-3}
Formula wt	410.48	T	-173 °C
Space-group	$P2_1/c$	λ	$0.71073 \text{ \AA}$
a	$3.7861(2) \text{ \AA}$	μ	0.86 cm^{-1}
b	$11.5027(6) \text{ \AA}$	Rel trans coefft	0.985-1.000
c	$22.1711(11) \text{ \AA}$	No. obs. rflns	1909 [$I \geq \sigma(I)$]
β	$93.890(2)^\circ$	Total indep. rflns	2181
V	$960.99(8) \text{ \AA}^3$	R(F), wR(F) (obs.)	0.067, 0.072
Z	2	R(F ²), wR(F ²) (all)	0.085, 0.135

$$R(F) = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$$

$$wR(F^2) = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w|F_o|^4]^{1/2}$$

Atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Atom	x	y	z	B_{eq}
N(1)	0.8762(4)	0.05678(13)	0.32830(6)	1.27(3)
N(2)	0.7533(4)	0.08497(13)	0.38256(6)	1.17(3)
C(1)	0.8935(5)	-0.05895(16)	0.32952(8)	1.18(4)
C(2)	1.0136(5)	-0.13351(17)	0.28446(8)	1.37(4)
C(3)	1.0086(5)	-0.24921(17)	0.29453(8)	1.47(4)
C(4)	0.8877(5)	-0.29627(16)	0.34883(8)	1.41(4)
C(5)	0.7729(5)	-0.22638(16)	0.39334(8)	1.25(4)
C(6)	0.7779(4)	-0.10618(15)	0.38404(8)	1.09(3)
C(7)	0.6890(4)	-0.00907(15)	0.41752(8)	1.10(3)
C(8)	0.5550(5)	-0.00122(14)	0.47527(8)	1.10(3)
C(9)	0.7017(4)	0.20376(15)	0.39665(8)	1.16(4)
C(10)	0.8201(5)	0.24518(16)	0.45294(8)	1.30(4)
C(11)	0.7704(5)	0.36065(16)	0.46622(9)	1.57(4)
C(12)	0.6086(5)	0.43419(17)	0.42316(9)	1.58(4)
C(13)	0.4951(5)	0.39147(16)	0.36708(9)	1.55(4)
C(14)	0.5399(5)	0.27599(16)	0.35332(8)	1.35(4)
H(1)	1.095(5)	-0.1011(16)	0.2476(9)	1.3(4)
H(2)	1.088(5)	-0.3024(16)	0.2650(9)	0.9(4)
H(3)	0.887(5)	-0.3773(17)	0.3541(8)	1.0(4)
H(4)	0.676(6)	-0.2636(19)	0.4283(10)	2.3(5)
H(5)	0.930(5)	0.1941(18)	0.4828(10)	2.0(4)
H(6)	0.866(6)	0.3932(17)	0.5047(10)	2.0(4)
H(7)	0.586(5)	0.5143(15)	0.4310(8)	0.5(3)
H(8)	0.376(5)	0.4448(19)	0.3360(9)	1.8(4)
H(9)	0.454(5)	0.2451(16)	0.3149(9)	1.5(4)

Bond lengths (Å)

N(1)-N(2)	1.356(2)	C(6)-C(7)	1.394(2)
N(1)-C(1)	1.333(2)	C(7)-C(8)	1.409(2)
N(2)-C(7)	1.361(2)	C(8)-C(8 ⁱ)	1.197(3)
N(2)-C(9)	1.418(2)	C(9)-C(10)	1.379(3)
C(1)-C(2)	1.413(2)	C(9)-C(14)	1.380(3)
C(1)-C(6)	1.419(2)	C(10)-C(11)	1.376(3)
C(2)-C(3)	1.350(3)	C(11)-C(12)	1.385(3)
C(3)-C(4)	1.421(3)	C(12)-C(13)	1.375(3)
C(4)-C(5)	1.365(2)	C(13)-C(14)	1.376(3)
C(5)-C(6)	1.398(3)		

(i) 1-x, y, 1-z

Bond angles (°)

N(2)-N(1)-C(1)	103.9(1)	C(5)-C(6)-C(7)	135.1(2)
N(1)-N(2)-C(7)	113.5(1)	N(2)-C(7)-C(6)	106.0(1)
N(1)-N(2)-C(9)	119.1(1)	N(2)-C(7)-C(8)	123.6(2)
C(7)-N(2)-C(9)	127.5(1)	C(6)-C(7)-C(8)	130.4(2)
N(1)-C(1)-C(2)	127.6(2)	C(7)-C(8)-C(8 ¹)	177.5(2)
N(1)-C(1)-C(6)	112.5(2)	N(2)-C(9)-C(10)	119.3(2)
C(2)-C(1)-C(6)	120.0(2)	N(2)-C(9)-C(14)	119.3(2)
C(1)-C(2)-C(3)	118.2(2)	C(10)-C(9)-C(14)	121.4(2)
C(2)-C(3)-C(4)	121.6(2)	C(9)-C(10)-C(11)	119.0(2)
C(3)-C(4)-C(5)	121.5(2)	C(10)-C(11)-C(12)	120.2(2)
C(4)-C(5)-C(6)	117.9(2)	C(11)-C(12)-C(13)	119.9(2)
C(1)-C(6)-C(5)	120.8(2)	C(12)-C(13)-C(14)	120.6(2)
C(1)-C(6)-C(7)	104.1(2)	C(9)-C(14)-C(13)	118.9(2)

(i) 1-x, -y, 1-z

Supplementary material

Crystallographic information

Crystal data

Formula	$C_{28}H_{18}N_4$
Formula weight	410.48
Crystal system, space-group	monoclinic, $P2_1/c$
a, b, c	3.7861(2), 11.5027(6), 22.1711(11) Å
α , β , γ	90, 93.890(2), 90°
V	960.00(8) Å ³
Z	2
d_{calc}	1.418 g cm ⁻³
μ	0.86 cm ⁻¹
F ₀₀₀	428

Data collection

Crystal appearance	colorless needle
Crystal dimensions	0.044 × 0.08 × 0.36 mm
Diffractometer	Bruker D8 SMART APEX
Radiation, wavelength	Mo K α , 0.71073 Å
Monochromator	graphite
Temperature	-173 °C
$2\theta_{max}$	65.9°
Index range h, k, l	-4→4, -14→14, -28→28
Frames: number, time, ω width	2400, 30 s, 0.3°
No. rflns scanned, R_{int}	10556, 0.075

Refinement

Absorption correction	none
Sec. extinction parameter	predicted neg., fixed at zero
No. rflns in refinement (N)	1909 [I $\geq\sigma$ (I)]
	2181 (all indep. data)
No. parameters (V)	181
Refinement on	F ²
Weighting factor w	1/ σ^2 (F)
R(F), wR(F) [I $\geq\sigma$ (I)]	0.067, 0.072
R(F ²), wR(F ²) (all data)	0.085, 0.135
S [I $\geq\sigma$ (I)], S (all data)	1.46, 1.33
Max. Δ/σ , last cycle	0.01
Max., min. in final diff. map	0.41, -0.31 e $\text{\AA}^{-3}$

$$R(F) = \sum ||F_o| - |F_c|| / \sum |F_o|$$

$$wR(F^2) = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^4]^{1/2}$$

$$S = [\sum w(|F_o| - |F_c|)^2 / (N-V)]^{1/2}$$

Supporting data

Anisotropic thermal parameters ($\text{\AA}^2$)

The temperature factor is given by: $T = \exp[-2\pi^2 \sum_i \sum_j U_{ij} h_i h_j a_i^* a_j^*]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	0.0204(8)	0.0206(8)	0.0080(8)	-0.0001(6)	0.0067(6)	-0.0021(6)
N(2)	0.0199(8)	0.0179(8)	0.0073(7)	0.0002(6)	0.0060(6)	0.0001(6)
C(1)	0.0157(9)	0.0199(10)	0.0093(9)	-0.0013(7)	0.0015(7)	0.0015(7)
C(2)	0.0185(9)	0.0256(10)	0.0086(9)	-0.0001(7)	0.0042(7)	-0.0014(7)
C(3)	0.0188(9)	0.0246(10)	0.0127(9)	0.0034(7)	0.0034(7)	-0.0070(8)
C(4)	0.0222(10)	0.0149(10)	0.0166(10)	0.0004(7)	0.0019(7)	-0.0023(7)
C(5)	0.0167(9)	0.0193(10)	0.0118(9)	-0.0010(7)	0.0022(7)	-0.0003(7)
C(6)	0.0123(8)	0.0211(10)	0.0083(8)	-0.0001(6)	0.0021(6)	-0.0018(7)
C(7)	0.0147(8)	0.0188(10)	0.0086(8)	-0.0018(6)	0.0028(6)	0.0002(7)
C(8)	0.0167(8)	0.0139(9)	0.0114(9)	0.0003(6)	0.0025(6)	-0.0002(7)
C(9)	0.0162(9)	0.0159(9)	0.0128(9)	-0.0011(6)	0.0082(7)	-0.0002(7)
C(10)	0.0172(9)	0.0209(10)	0.0116(9)	-0.0014(7)	0.0044(7)	0.0015(8)
C(11)	0.0232(9)	0.0209(10)	0.0165(10)	-0.0041(7)	0.0075(8)	-0.0032(8)
C(12)	0.0222(10)	0.0159(10)	0.0232(10)	0.0001(7)	0.0103(8)	-0.0017(8)
C(13)	0.0213(9)	0.0204(10)	0.0177(10)	0.0016(7)	0.0062(8)	0.0040(8)
C(14)	0.0190(9)	0.0224(10)	0.0105(9)	-0.0010(7)	0.0053(7)	0.0012(7)

Supporting data

Bond lengths (Å) for hydrogen atoms

C(2)	H(1)	0.97(2)	C(11)	H(6)	0.98(2)
C(3)	H(2)	0.96(2)	C(12)	H(7)	0.94(2)
C(4)	H(3)	0.94(2)	C(13)	H(8)	1.00(2)
C(5)	H(4)	0.98(2)	C(14)	H(9)	0.96(2)
C(10)	H(5)	0.96(2)			

Supporting data

Intermolecular distances (Å) for non-hydrogen atoms

N(1)	N(2 ⁱ)	3.469(2)	C(3)	C(5 ⁱ)	3.516(3)
N(1)	C(3 ⁱⁱ)	3.565(2)	C(4)	C(5 ⁱ)	3.522(3)
N(1)	C(14 ⁱ)	3.576(2)	C(7)	C(8 ⁱ)	3.438(3)
C(1)	C(2 ⁱⁱⁱ)	3.518(3)	C(7)	C(8 ^{iv})	3.593(3)
C(1)	C(7 ⁱ)	3.521(2)	C(8)	C(8 ^{iv})	3.472(3)
C(1)	C(6 ⁱ)	3.523(2)	C(9)	C(14 ⁱ)	3.477(2)
C(2)	C(6 ⁱ)	3.530(3)	C(11)	C(12 ⁱ)	3.479(2)
C(3)	C(4 ⁱ)	3.503(3)			

Symmetry code:

(i) 1+x, y, z; (ii) 2-x, 0.5+y, 0.5-z; (iii) -1+x, y, z;

(iv) 2-x, 1-y, -z

Supporting data

Torsion angles (°)

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
N(1)	N(2)	C(7)	C(6)	0.4(2)	C(2)	C(1)	C(6)	C(5)	-1.6(3)
N(1)	N(2)	C(7)	C(8)	-178.9(2)	C(2)	C(1)	C(6)	C(7)	178.6(2)
N(1)	N(2)	C(9)	C(10)	-134.5(2)	C(2)	C(3)	C(4)	C(5)	-0.4(3)
N(1)	N(2)	C(9)	C(14)	44.3(2)	C(3)	C(2)	C(1)	C(6)	1.1(3)
N(1)	C(1)	C(2)	C(3)	-179.8(2)	C(3)	C(4)	C(5)	C(6)	-0.1(3)
N(1)	C(1)	C(6)	C(5)	179.2(1)	C(4)	C(5)	C(6)	C(7)	-179.3(2)
N(1)	C(1)	C(6)	C(7)	-0.6(2)	C(5)	C(6)	C(7)	C(8)	-0.4(3)
N(2)	N(1)	C(1)	C(2)	-178.4(2)	C(6)	C(7)	N(2)	C(9)	179.5(2)
N(2)	N(1)	C(1)	C(6)	0.8(2)	C(6)	C(7)	C(8)	C(8)	17(6)
N(2)	C(7)	C(6)	C(1)	0.1(2)	C(7)	N(2)	C(9)	C(10)	46.4(2)
N(2)	C(7)	C(6)	C(5)	-179.7(2)	C(7)	N(2)	C(9)	C(14)	-134.8(2)
N(2)	C(7)	C(8)	C(8)	-164(6)	C(7)	C(8)	C(8)	C(7)	180
N(2)	C(9)	C(10)	C(11)	179.7(1)	C(8)	C(7)	N(2)	C(9)	0.2(3)
N(2)	C(9)	C(14)	C(13)	-179.1(1)	C(9)	C(10)	C(11)	C(12)	-1.0(2)
C(1)	N(1)	N(2)	C(7)	-0.7(2)	C(9)	C(14)	C(13)	C(12)	-0.2(2)
C(1)	N(1)	N(2)	C(9)	-179.9(1)	C(10)	C(9)	C(14)	C(13)	-0.4(2)
C(1)	C(2)	C(3)	C(4)	-0.2(3)	C(10)	C(11)	C(12)	C(13)	0.4(2)
C(1)	C(6)	C(5)	C(4)	1.0(3)	C(11)	C(10)	C(9)	C(14)	1.0(2)
C(1)	C(6)	C(7)	C(8)	179.4(2)	C(11)	C(12)	C(13)	C(14)	0.2(3)

The sign is positive if a clockwise motion of atom 1 would superimpose it on atom 4 when the direction of view is from atom 2 to atom 3.

Supporting data

Least-squares mean planes (deviations in Å)

<u>Plane 1</u>	Defining atoms	Distance	Other atoms	Distance
	N(1)	-0.014(2)	C(8)	0.009
	N(2)	0.007(2)	C(9)	0.005
	C(1)	-0.007(2)		
	C(2)	0.010(2)		
	C(3)	0.010(2)		
	C(4)	-0.004(2)		
	C(5)	-0.010(2)		
	C(6)	-0.001(2)		
	C(7)	0.010(2)		

Mean deviation from plane: 0.008 Å; χ^2 , 272

<u>Plane 2</u>	Defining atoms	Distance	Other atoms	Distance
	C(9)	-0.00392)	N(2)	0.012
	C(10)	0.006(2)		
	C(11)	-0.004(2)		
	C(12)	-0.001(2)		
	C(13)	0.003(2)		
	C(14)	-0.000(2)		

Mean deviation from plane: 0.003 Å; χ^2 , 23.8

Dihedral angle between planes, 45.8°

Crystal structural analysis

A crystal of dimensions $0.02 \times 0.08 \times 0.50$ mm was mounted on a fiber. The orientation parameters and cell dimensions were obtained from the setting angles of a Nonius CAD-4 serial diffractometer for 25 centered reflections in the range $12.2^\circ \leq \theta \leq 13.5^\circ$. The following table contains a summary of crystal data and the final residuals. A more extensive table including particulars of data collection and structure refinement is in the supplementary material. The monoclinic Laue symmetry and systematic absences together indicated the space-group $P2_1/c$. A SIR92 E-map¹ showed all the non-hydrogen atoms. Absorption and decay corrections were not deemed necessary. Hydrogen atoms were included at positions recalculated after each cycle of refinement [$B(H) = 1.2B_{eq}(C)$; $d(C-H) = 0.95 \text{ \AA}$]. The final difference synthesis was featureless. The TEXSAN program suite² was used in all calculations.

References

1. Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, N. *J. Appl. Crystallography*, 1994, 27, 435.
2. Molecular Structures Corporation, 3200A Research Forest Drive, The Woodlands, TX 77381, USA. *TeXsan Software for Single-Crystal Structure Analysis*, version 1.7, 1997.

Crystallographic data

Composition	C ₂₃ H ₃₂ N ₂ O ₂ Si	d _{calc}	1.116 g cm ⁻³
Formula wt	380.6	T	23 °C
Space-group	P2 ₁ /c	λ	0.71073 Å
a	15.954(4) Å	μ	1.18 cm ⁻¹
b	8.689(3) Å	No. obs. rflns	2342 [I ≥ σ(I)]
c	17.933(8) Å	Total indep. rflns	3925
β	114.35(3) °	R(F), wR(F) (obs.)	0.063, 0.057
V	2265(2) Å ³	R(F ²), wR(F ²) (all)	0.104, 0.120
Z	4		

$$R(F) = \sum ||F_o| - |F_c|| / \sum |F_o|$$

$$wR(F^2) = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^4]^{1/2}$$

Atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Atom	x	y	z	B_{eq}
Si	0.31838 (6)	0.76234 (10)	0.16218 (5)	3.726 (19)
O	0.25820 (12)	0.6035 (2)	0.12509 (11)	4.01 (5)
N(1)	0.13294 (16)	0.3160 (3)	0.01636 (14)	3.84 (6)
N(2)	0.08227 (17)	0.1924 (3)	0.02048 (16)	4.51 (7)
C(1)	0.04329 (19)	0.2415 (4)	0.07026 (18)	4.16 (8)
C(2)	-0.0164 (2)	0.1585 (4)	0.0953 (2)	5.24 (9)
C(3)	-0.0483 (2)	0.2302 (5)	0.1453 (2)	5.92 (11)
C(4)	-0.0248 (2)	0.3855 (5)	0.1708 (2)	5.71 (10)
C(5)	0.0327 (2)	0.4664 (4)	0.14705 (19)	4.77 (9)
C(6)	0.06882 (18)	0.3956 (4)	0.09664 (17)	3.75 (7)
C(7)	0.12715 (18)	0.4395 (3)	0.06052 (17)	3.56 (7)
C(8)	0.16916 (19)	0.5927 (3)	0.06029 (17)	3.96 (8)
C(9)	0.1866 (2)	0.3001 (3)	-0.03045 (18)	3.60 (7)
C(10)	0.2764 (2)	0.3489 (4)	0.00163 (18)	4.54 (8)
C(11)	0.3280 (2)	0.3295 (4)	-0.0431 (2)	5.21 (9)
C(12)	0.2907 (2)	0.2600 (4)	-0.1182 (2)	5.34 (9)
C(13)	0.2008 (3)	0.2114 (4)	-0.15000 (18)	5.36 (10)
C(14)	0.1482 (2)	0.2308 (4)	-0.10618 (18)	4.62 (8)
C(15)	0.4077 (2)	0.6924 (4)	0.2623 (2)	5.60 (9)
C(16)	0.4753 (3)	0.8125 (5)	0.3137 (3)	10.91 (15)
C(17)	0.3723 (3)	0.5958 (6)	0.3114 (3)	10.48 (16)
C(18)	0.2403 (2)	0.9192 (4)	0.16709 (19)	4.74 (9)
C(19)	0.2860 (3)	1.0783 (4)	0.1869 (2)	6.46 (11)
C(20)	0.1917 (3)	0.8863 (5)	0.2232 (3)	8.10 (13)
C(21)	0.3745 (2)	0.8305 (4)	0.0950 (2)	5.23 (9)
C(22)	0.4444 (3)	0.7191 (5)	0.0878 (3)	8.52 (14)
C(23)	0.3064 (3)	0.8782 (5)	0.0103 (2)	7.69 (13)

Bond lengths (Å)

Si-O	1.654 (2)	C(6)-C(7)	1.387 (4)
Si-C(15)	1.871 (3)	C(7)-C(8)	1.490 (4)
Si-C(18)	1.870 (3)	C(9)-C(10)	1.370 (4)
Si-C(21)	1.868 (3)	C(9)-C(14)	1.375 (4)
O-C(8)	1.418 (3)	C(10)-C(11)	1.375 (4)
N(1)-N(2)	1.363 (3)	C(11)-C(12)	1.366 (4)
N(1)-C(7)	1.358 (3)	C(12)-C(13)	1.371 (4)
N(1)-C(9)	1.430 (3)	C(13)-C(14)	1.374 (4)
N(2)-C(1)	1.349 (4)	C(15)-C(16)	1.509 (5)
C(1)-C(2)	1.406 (4)	C(15)-C(17)	1.485 (5)
C(1)-C(6)	1.421 (4)	C(18)-C(19)	1.533 (4)
C(2)-C(3)	1.350 (5)	C(18)-C(20)	1.527 (4)
C(3)-C(4)	1.423 (5)	C(21)-C(22)	1.520 (5)
C(4)-C(5)	1.355 (4)	C(21)-C(23)	1.515 (5)
C(5)-C(6)	1.397 (4)		

Bond angles (°)

O-Si-C(15)	101.5 (1)	N(1)-C(7)-C(6)	105.9 (3)
O-Si-C(18)	109.8 (1)	N(1)-C(7)-C(8)	124.4 (3)
O-Si-C(21)	111.0 (1)	C(6)-C(7)-C(8)	129.4 (3)
C(15)-Si-C(18)	116.7 (1)	O-C(8)-C(7)	110.8 (2)
C(15)-Si-C(21)	110.1 (2)	N(1)-C(9)-C(10)	119.8 (3)
C(18)-Si-C(21)	107.7 (2)	N(1)-C(9)-C(14)	119.3 (3)
Si-O-C(8)	127.1 (2)	C(10)-C(9)-C(14)	120.8 (3)
N(2)-N(1)-C(7)	114.0 (2)	C(9)-C(10)-C(11)	119.3 (3)
N(2)-N(1)-C(9)	117.8 (2)	C(10)-C(11)-C(12)	120.4 (3)
C(7)-N(1)-C(9)	128.2 (2)	C(11)-C(12)-C(13)	120.0 (3)
N(1)-N(2)-C(1)	103.4 (2)	C(12)-C(13)-C(14)	120.2 (3)
N(2)-C(1)-C(2)	127.3 (3)	C(9)-C(14)-C(13)	119.3 (3)
N(2)-C(1)-C(6)	111.8 (3)	Si-C(15)-C(16)	115.5 (3)
C(2)-C(1)-C(6)	121.0 (3)	Si-C(15)-C(17)	115.2 (3)
C(1)-C(2)-C(3)	117.7 (3)	C(16)-C(15)-C(17)	112.2 (3)
C(2)-C(3)-C(4)	121.9 (3)	Si-C(18)-C(19)	114.0 (2)
C(3)-C(4)-C(5)	120.9 (3)	Si-C(18)-C(20)	115.0 (2)
C(4)-C(5)-C(6)	119.1 (3)	C(19)-C(18)-C(20)	109.7 (3)
C(1)-C(6)-C(5)	119.5 (3)	Si-C(21)-C(22)	114.5 (3)
C(1)-C(6)-C(7)	105.0 (3)	Si-C(21)-C(23)	113.3 (2)
C(5)-C(6)-C(7)	135.5 (3)	C(22)-C(21)-C(23)	109.7 (3)

Supplementary material

Crystallographic information

Crystal data

Formula	$C_{23}H_{32}N_2OS$
Formula weight	380.6
Crystal system, space-group	monoclinic, $P2_1/c$
a, b, c	15.954(4), 8.689(3), 17.933(8) Å
α , β , γ	90, 114.35(3), 90°
V	2265(2) Å ³
Z	4
d_{calc}	1.116 g cm ⁻³
μ	1.18 cm ⁻¹
F_{000}	824

Data collection

Crystal appearance	colorless prism
Crystal dimensions	0.02 × 0.08 × 0.50 mm
Diffractometer	Enraf-Nonius CAD-4
Radiation, wavelength	Mo K α , 0.71073 Å
Monochromator	graphite
Temperature	23 °C
$2\theta_{max}$	50°
Index range h, k, l	0→18, 0→10, -21→19
Scan mode, speed (on ω)	ω -2 θ , 1.0-4.1° min ⁻¹
Scan width	(1.05 + 0.35tan θ)°

Reference reflections	3, every 3600 s exposure
No. indep. rflns scanned, R_{int}	4292, 0.038

Refinement

Absorption correction	none
Sec. extinction parameter	refined neg., fixed at zero
No. rflns in refinement (N)	2342 [$I \geq \sigma(I)$] 3925 (all indep. data)
No. parameters (V)	244
Refinement on	$ F ^2$
Weighting factor w	$1/\sigma^2(F)$
$R(F)$, $wR(F)$ [$I \geq \sigma(I)$]	0.063, 0.057
$R(F^2)$, $wR(F^2)$ (all data)	0.104, 0.120
S [$I \geq \sigma(I)$], S (all data)	1.64, 1.37
Max. Δ/σ , last cycle	0.01
Max., min. in final diff. map	0.41, -0.44 e $\text{\AA}^{-3}$

$$R(F) = \sum |F_o| - |F_c| / \sum |F_o| \quad wR(F^2) = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^4]^{1/2}$$

$$S = [\sum w(|F_o| - |F_c|)^2 / (N-V)]^{1/2}$$

Supporting data

Anisotropic thermal parameters ($\text{\AA}^2$)

The temperature factor is given by: $T = \exp[-2\pi^2 \sum_i \sum_j U_{ij} h_i h_j a_i^* a_j^*]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si	0.0519(5)	0.0379(5)	0.0498(5)	-0.0036(5)	0.0190(4)	-0.0014(5)
O	0.0459(11)	0.0361(13)	0.0580(13)	-0.0034(10)	0.0090(10)	-0.0034(10)
N(1)	0.0480(15)	0.0401(16)	0.0563(16)	-0.0060(13)	0.0198(13)	-0.0030(13)
N(2)	0.0594(17)	0.0433(17)	0.0656(17)	-0.0103(14)	0.0226(14)	-0.0036(13)
C(1)	0.0451(17)	0.054(2)	0.0565(19)	-0.0007(17)	0.0186(15)	0.0079(19)
C(2)	0.058(2)	0.063(3)	0.074(2)	-0.0089(19)	0.0239(19)	0.010(2)
C(3)	0.060(2)	0.096(3)	0.072(2)	-0.004(2)	0.0304(19)	0.022(3)
C(4)	0.062(2)	0.095(3)	0.063(2)	0.011(2)	0.0296(19)	0.004(2)
C(5)	0.055(2)	0.066(2)	0.060(2)	0.0055(18)	0.0231(18)	0.0003(19)
C(6)	0.0418(17)	0.046(2)	0.0480(18)	0.0028(15)	0.0123(15)	0.0034(16)
C(7)	0.0419(17)	0.039(2)	0.0452(18)	0.0016(15)	0.0084(14)	-0.0012(15)
C(8)	0.0544(19)	0.0353(19)	0.0550(19)	0.0016(15)	0.0165(16)	-0.0005(15)
C(9)	0.0518(18)	0.0322(19)	0.0502(18)	0.0041(14)	0.0185(15)	0.0032(14)
C(10)	0.058(2)	0.059(2)	0.056(2)	-0.0042(17)	0.0229(17)	-0.0098(17)
C(11)	0.062(2)	0.067(3)	0.072(2)	-0.0044(18)	0.0307(19)	-0.007(2)
C(12)	0.082(3)	0.066(2)	0.067(2)	0.006(2)	0.0435(19)	-0.001(2)
C(13)	0.086(3)	0.061(3)	0.053(2)	0.003(2)	0.024(2)	-0.0061(18)
C(14)	0.063(2)	0.050(2)	0.055(2)	-0.0040(18)	0.0180(17)	-0.0014(18)
C(15)	0.072(2)	0.061(3)	0.060(2)	-0.0110(19)	0.0082(18)	0.0030(19)
C(16)	0.134(4)	0.096(4)	0.100(3)	-0.029(3)	-0.037(3)	-0.001(3)
C(17)	0.128(4)	0.166(5)	0.085(3)	-0.015(4)	0.025(3)	0.056(3)
C(18)	0.071(2)	0.041(2)	0.070(2)	0.0026(17)	0.0308(18)	-0.0079(17)
C(19)	0.104(3)	0.047(2)	0.096(3)	0.004(2)	0.043(2)	-0.013(2)
C(20)	0.122(3)	0.082(3)	0.142(4)	-0.001(3)	0.093(3)	-0.016(3)
C(21)	0.069(2)	0.059(2)	0.078(2)	-0.0043(19)	0.038(2)	-0.003(2)
C(22)	0.106(3)	0.102(4)	0.150(4)	0.013(3)	0.088(3)	0.010(3)
C(23)	0.120(3)	0.105(4)	0.085(3)	0.004(3)	0.060(3)	0.025(3)

Supporting data

Calculated coordinates and thermal parameters ($\text{\AA}^2$) for hydrogen atoms

Atom	x	y	z	B
H(1)	-0.0336	0.0557	0.0777	6.3
H(2)	-0.0876	0.1755	0.1639	7.1
H(3)	-0.0497	0.4330	0.2049	6.9
H(4)	0.0483	0.5699	0.1644	5.7
H(5)	0.1311	0.6709	0.0667	4.8
H(6)	0.1739	0.6064	0.0096	4.8
H(7)	0.3026	0.3957	0.0542	5.4
H(8)	0.3899	0.3645	-0.0217	6.3
H(9)	0.3270	0.2453	-0.1484	6.4
H(10)	0.1748	0.1643	-0.2024	6.4
H(11)	0.0861	0.1968	-0.1279	5.5
H(12)	0.4442	0.6236	0.2470	6.7
H(13)	0.5256	0.7634	0.3562	13.1
H(14)	0.4973	0.8698	0.2803	13.1
H(15)	0.4456	0.8799	0.3369	13.1
H(16)	0.3400	0.6588	0.3340	12.6
H(17)	0.3318	0.5197	0.2772	12.6
H(18)	0.4223	0.5471	0.3543	12.6
H(19)	0.1930	0.9272	0.1134	5.7
H(20)	0.3319	1.0788	0.2415	7.8
H(21)	0.3135	1.1005	0.1502	7.8
H(22)	0.2408	1.1540	0.1814	7.8
H(23)	0.1488	0.9660	0.2175	9.7
H(24)	0.1603	0.7907	0.2084	9.7
H(25)	0.2359	0.8818	0.2784	9.7
H(26)	0.4078	0.9208	0.1201	6.3
H(27)	0.4138	0.6287	0.0602	10.2
H(28)	0.4746	0.7663	0.0579	10.2
H(29)	0.4885	0.6929	0.1410	10.2
H(30)	0.2638	0.9497	0.0150	9.2

H(31)	0.3384	0.9246	-0.0183	9.2
H(32)	0.2742	0.7900	-0.0189	9.2

Supporting data

Intermolecular distances (Å) for non-hydrogen atoms

N(1)	C(5 ⁱ)	3.567(4)	C(1)	C(8 ⁱ)	3.530(4)
N(2)	C(19 ⁱⁱ)	3.524(5)	C(2)	C(8 ⁱ)	3.572(4)
N(2)	C(2 ⁱⁱⁱ)	3.590(5)	C(6)	C(7 ⁱ)	3.531(4)

Symmetry code

(i) -x, 1-y, -z; (ii) x, -1+y, z; (iii) -x, -y, -z

Supporting data

Torsion angles (°)

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
Si	O	C(8)	C(7)	-163.5(2)	C(3)	C(2)	C(1)	C(6)	-0.1(5)
O	Si	C(15)	C(16)	-179.3(3)	C(3)	C(4)	C(5)	C(6)	0.2(5)
O	Si	C(15)	C(17)	-45.9(3)	C(4)	C(5)	C(6)	C(7)	179.5(3)
O	Si	C(18)	C(19)	-170.3(2)	C(5)	C(6)	C(7)	C(8)	-4.3(5)
O	Si	C(18)	C(20)	61.9(3)	C(6)	C(7)	N(1)	C(9)	-178.0(3)
O	Si	C(21)	C(22)	-62.7(3)	C(7)	N(1)	C(9)	C(10)	44.5(4)
O	Si	C(21)	C(23)	64.1(3)	C(7)	N(1)	C(9)	C(14)	-137.8(3)
O	C(8)	C(7)	N(1)	-93.2(3)	C(8)	O	Si	C(15)	163.8(2)
O	C(8)	C(7)	C(6)	93.7(3)	C(8)	O	Si	C(18)	39.7(2)
N(1)	N(2)	C(1)	C(2)	-180.0(3)	C(8)	O	Si	C(21)	-79.3(2)
N(1)	N(2)	C(1)	C(6)	0.5(3)	C(8)	C(7)	N(1)	C(9)	7.6(4)
N(1)	C(7)	C(6)	C(1)	0.2(3)	C(9)	C(10)	C(11)	C(12)	-1.0(5)
N(1)	C(7)	C(6)	C(5)	-178.3(3)	C(9)	C(14)	C(13)	C(12)	0.4(5)
N(1)	C(9)	C(10)	C(11)	178.4(3)	C(10)	C(9)	C(14)	C(13)	-0.3(5)
N(1)	C(9)	C(14)	C(13)	-178.1(3)	C(10)	C(11)	C(12)	C(13)	1.1(6)
N(2)	N(1)	C(7)	C(6)	0.0(3)	C(11)	C(10)	C(9)	C(14)	0.7(5)
N(2)	N(1)	C(7)	C(8)	-174.4(2)	C(11)	C(12)	C(13)	C(14)	-0.7(5)
N(2)	N(1)	C(9)	C(10)	-133.5(3)	C(15)	Si	C(18)	C(19)	74.9(3)
N(2)	N(1)	C(9)	C(14)	44.3(4)	C(15)	Si	C(18)	C(20)	-52.9(3)
N(2)	C(1)	C(2)	C(3)	-179.6(3)	C(15)	Si	C(21)	C(22)	48.9(3)
N(2)	C(1)	C(6)	C(5)	178.4(3)	C(15)	Si	C(21)	C(23)	175.7(3)
N(2)	C(1)	C(6)	C(7)	-0.5(3)	C(16)	C(15)	Si	C(18)	-60.0(4)
C(1)	N(2)	N(1)	C(7)	-0.3(3)	C(16)	C(15)	Si	C(21)	63.1(4)

C(1) N(2) N(1) C(9)	177.9(2)	C(17)C(15)Si C(18)	73.5(4)
C(1) C(2) C(3) C(4)	1.4(5)	C(17)C(15)Si C(21)	-163.5(3)
C(1) C(6) C(5) C(4)	1.1(4)	C(18)Si C(21)C(22)	177.0(3)
C(1) C(6) C(7) C(8)	174.3(3)	C(18)Si C(21)C(23)	-56.1(3)
C(2) C(1) C(6) C(5)	-1.2(4)	C(19)C(18)Si C(21)	-49.4(3)
C(2) C(1) C(6) C(7)	180.0(3)	C(20)C(18)Si C(21)	-177.2(3)
C(2) C(3) C(4) C(5)	-1.5(5)		

The sign is positive if a clockwise motion of atom 1 would superimpose it on atom 4 when the direction of view is from atom 2 to atom 3.

Supporting data

Least-squares mean planes (deviations in Å)

<u>Plane 1</u>	Defining atoms	Distance	Other atoms	Distance
	N(1)	-0.002(3)	C(8)	-0.102
	N(2)	-0.010(3)	C(9)	0.033
	C(1)	0.004(3)		
	C(2)	0.004(3)		
	C(3)	0.010(4)		
	C(4)	-0.012(4)		
	C(5)	-0.011(2)		
	C(6)	0.009(3)		
	C(7)	0.009(3)		

Mean deviation from plane: 0.008 Å; χ^2 , 84.2

<u>Plane 2</u>	Defining atoms	Distance	Other atoms	Distance
	C(9)	-0.001(3)	N(1)	0.045
	C(10)	0.003(4)		
	C(11)	-0.005(4)		
	C(12)	0.004(4)		
	C(13)	-0.001(4)		
	C(14)	0.000(3)		

Mean deviation from plane: 0.002 Å; χ^2 , 4.4

Dihedral angle between planes, 45.1°

PULSE SEQUENCE	OBSERVE C13, 75.421881	DATA PROCESSING	13C OBSERVE
Relax delay 1.000 sec	DECOUPLE H1, 289.9478514	Line broadening 1.0 Hz	
Pulse 42.8 degrees	Power 32.06	F1 size 32768	Pulse Sequence: szpu1
Acq. time 0.593 sec	Continuously on	Total time 10 minutes	Solvent: CDCl3
Width 18501.7 Hz	WALTZ-16 modulated		Ambient temperature
387 repetitions			INOVA-300 "sunofmr"

nmrdate/13-C-H-alkyne-dimer

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

INNOVA-300 "sunofmr.uoregon.edu"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 47.4 degrees

Acq. time 0.599 sec

Width 16501.7 Hz

20539 repetitions

OBSERVE C13, 253.130944 MHz

PROBHD 1H, 253.135118 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 11 hr, 10 min, 1 sec

11

13C OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3
 Ambient temperature
 INOVA-300 "Sunomim, nor-egon.edu"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 47.4 degrees

Acq. time 0.593 sec

Waltz-16 0.72 sec

564 repetitions

OBSERVE C13, 75.4180664 MHz

DECOUPLE H1, 299.333198 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 26 min, 48 sec

13C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
INNOVA-300 "sunofmr.uoregon.edu"
PULSE SEQUENCE
Relax delay: 1.000 sec
Pulse: zgpg30
Acq. time: 0.599 sec
Width: 16501.7 Hz
1028 repetitions
OBSERVE: C13, 75.4160944 MHz
DECOUPLE: H1, 299.935198 MHz
Power: 32 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
F2 size: 32768
Total time: 44 hr, 40 min, 5 sec

unmrdata/1-H-CH3-TES-diazene
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Acquisition Temperature
 INOVA-300 500MHz, uoregon.edu
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 37.9 degrees
 Width 1.000 sec
 Width 478.0 Hz
 8 repetitions
 OBSERVE H1, 299.9325252 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 32768
 Total time 0 min, 28 sec

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vnmrdata/I-H-Cl-1TESA-diazene

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient Temperature: 300
INDO=300 "unoffnmr.vorjagun.edu"

PULSE SEQUENCE
Relax1: delay 1.000 sec
Acq1: time 2.5 degrees
Acq1: time 2.501 sec
16 repetitions
OBSERVE: H1, 299.9325267 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 56 sec

```

16

13C Observe

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient Temperature
INNOVA-300 "SunOffmr.uoregon.edu"
PULSE SEQUENCE
Relax: delay 1.000 sec
Pulse: 47.4 degrees
Acq: time 0.559 sec
Width: 16501.7 Hz
1854 repetitions
OBSERVE: C13, 75.418933 MHz
DECODE: H1, 299.935198 MHz
Power: 32 dB
CONTINUOUSLY ON
VOLTAGE: 100.000 V
LINE BROADENING: 1.0 Hz
FI size: 32768
Total time: 1 hr, 7 min, 0 sec

13C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient Temperature
INNOVA-300 "sunofmr.uoregon.edu"
Pulse Sequence
Pulse delay 1.000 sec
Acq. time 0.593 sec
Width 16501.7 Hz
5000 repetitions
OBSERVE C13, 75.4180933 MHz
DECOUPLE H1, 299.935186 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line centering 1.0 Hz
F2 125.32765
Total time 2 hr, 14 min, 0 sec

25

13C OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INNOVA-300 "sunofmr.uoregon.edu"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 47.4 degrees
Acq. time 0.599 sec
Width 16501.7 Hz
308 repetitions
OBSERVE CH, 75.4180742 MHz
DECOUPLE H1, 299.9355198 MHz
Power 35.06 dB on
P1 1.00000000 sec
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 26 min, 48 sec

13C OBSERVE

Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-300 "sunofmr.uoregon.edu"
 PULSE SEQUENCE
 Relax: delay 1.000 sec
 Pulse: 47.4 degrees
 Acq: time 0.598 sec
 Width 16501.7 Hz
 2054 repetitions
 OBSERVE: C13, 75.4160742 MHz
 DECOUPLE: H1, 299.9333198 MHz
 Power: 32 dB on
 Spectroscopy on
 VOLTAGE: 1000 V
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 1 hr, 7 min, 0 sec

13C OBSERVE

Pulse Sequence: szpul
Solvent: CDCl3
Acquire Temperature
INSTRUMENT: spectromet.uniconn.edu
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 47.4 degrees
Acq. time 0.393 sec
Waltz 16
2294 repetitions
OBSERVE C13, 75.418054 MHz
DECOUPLE H1, 299.935198 MHz
Power 32 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FI size 32768
Total time 4 hr, 1 min, 12 sec

nmrdata/1-methoxy-TES-bromo-diazene

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

INNOVA-300 "sunofmr.uoregon.edu"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 37.9 degrees

Acq. time 2.501 sec

Width 4799.0 Hz

Observations 299.9325256 MHz

Offset 0.000 Hz

Line broadening 0.2 Hz

FT size 32768

Total time 0 min, 28 sec

31

13C OBSERVE

Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient temperature
 INOVA-300 "sunofmr.jocregon.edu"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 47.4 degrees
 Acq. time 0.599 sec
 Width 16501.7 Hz
 1000 repetitions
 OBSERVE C13, 4180954 MHz
 PULSE 41, 259.553198 MHz
 POWER 32 dB
 Continuously on
 WAIT-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 26 min, 48 sec

13C OBSERVE

Pulse Sequence: zgpg1
 Solvent: CDCl3
 Ambient Temperature
 INOVA-300 "Sunofmr.uoregon.edu"
 PULSE SEQUENCE
 Relax, delay 1.000 sec
 Pulse 47.4 degrees
 Acq. time 0.589 sec
 Width 18801.7 Hz
 13C frequencies 418954 MHz
 OBSERVE C13 298.935138 MHz
 DECOUPLE H1 298.935138 MHz
 Power 32 dB
 continuously on
 VOLTAGE modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 26 min, 48 sec

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-300 "sunofmr.uoregon.edu"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 47.4 degree
Acq. time 0.599 sec
Width 16501.7 Hz
1259 repetitions
OBSERVE C13, 75.416064 MHz
DECOUPLE H1, 299.9355198 MHz
Power 32 db
Continuously on
VARIABLE MODULATED
DATA ACQUISITION
Line broadening 1.0 Hz
FT size 32768
Total time 8 hr, 56 min, 1 sec

nmrdata/13-C-methyl-OH-isoindazole

Pulse sequence: szpul

Solvent: CDCl3

Ambient temperature

INNOVA-300 "Sunofnmr.uoregon.edu"

PULSE SEQUENCE

Relax. delay 1.000 sec

Acq. time 0.461 sec

Width 16501.7 Hz

1688 repetitions

OBSERVE C13, 75.480964 MHz

DECOUPLE H1, 299.935198 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 3 hr, 7 min, 36 sec

STANDARD 1H OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient Temperature

INSTRUMENT: spectrometer, uoregon.edu

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 37.9 degrees

Acq. time 2.501 sec

NUC1 13C

NUC2 13C

NUC3 13C

NUC4 13C

NUC5 13C

NUC6 13C

NUC7 13C

NUC8 13C

NUC9 13C

NUC10 13C

NUC11 13C

NUC12 13C

NUC13 13C

NUC14 13C

NUC15 13C

NUC16 13C

NUC17 13C

NUC18 13C

NUC19 13C

NUC20 13C

NUC21 13C

NUC22 13C

NUC23 13C

NUC24 13C

NUC25 13C

NUC26 13C

NUC27 13C

NUC28 13C

NUC29 13C

NUC30 13C

NUC31 13C

NUC32 13C

NUC33 13C

NUC34 13C

NUC35 13C

NUC36 13C

NUC37 13C

NUC38 13C

NUC39 13C

NUC40 13C

NUC41 13C

NUC42 13C

NUC43 13C

NUC44 13C

NUC45 13C

NUC46 13C

NUC47 13C

NUC48 13C

NUC49 13C

NUC50 13C

NUC51 13C

NUC52 13C

NUC53 13C

NUC54 13C

NUC55 13C

NUC56 13C

NUC57 13C

DATA PROCESSING

Line broadening 0.2 Hz

FT size 32768

Total time 0 min, 26 sec

39

vnmrdata/13-C-COH-OH-(isotindazole)
 Pulse Sequence: sepul
 Solvent: CDCl3
 Ambient temperature
 INOVA-300 "sunofmr.uoregon.edu"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 47.4 degrees
 Acq. time 0.599 sec
 Width 16501.7 Hz
 32700 repetitions
 OBSERVE C13 4180954 MHz
 DECOUPLE H1 293.935198 MHz
 Power 32 dB
 continuously on
 VALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 4 hr, 28 min, 0 sec

nmrdata/17-03-04-13-C-methoxy-OH-isoindazole

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

INNOVA-300 "sunofmr.uoregon.edu"

PULSE SEQUENCE

Pulse 1: 9.40 sec

Pulse 2: 0.598 sec

Acq. time 0.598 sec

Width 16501.7 Hz

25152 repetitions

OBSERVE C13, 75.416094 MHz

DECOUPLE H1, 299.935198 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

File 170303

Total time 13 hr, 24 min, 1 sec

nmrdata/13-C-methoxy-OH-fluoro-isoindazole
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-300 "sunofmr.uoregon.edu"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 47.4 degrees
 Acq. time 0.599 sec
 Width 16501.7 Hz
 23186 repetitions
 OBSERVE C13, 75.418944 MHz
 DECOUPLE H1, 299.335136 MHz
 Power 32.06
 CONTINUOUSLY ON
 VOLTAGE REGULATED
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 32768
 Total time 12 hr, 3 min, 37 sec

nmrdata/16-03-04-13-C-methoxy-OH-methyl-ester-isoindazole

Pulse Sequence: szpul

Solvent: COCl₃

Ambient temperature

INNOVA-300 "sunofnmr.uoregon.edu"

PULSE SEQUENCE

Relax: delay 1.000 sec

Acq: time 0.589 sec

Width 16501.7 Hz

2136 repetitions

OBSERVE C13, 75.418044 MHz

DECOUPLE H1, 299.9335198 MHz

Power 32 dB

continuously on

VALT2-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 11 hr, 10 min, 1 sec

vnmrdata/13-C-methoxy-OH-nitro-isolindazole
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-300 "sunofmr.uoregon.edu"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 47.4 degrees
 Acq. time 0.599 sec
 Width 16501.7 Hz
 3605 repetitions
 OBSERVE C13, 75.418944 MHz
 DECOUPLE H1, 299.935198 MHz
 Power 32.45 dB
 Continuous on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 3 hr, 7 min, 38 sec

nmrdate/16-03-04-13-C-chloro-cyclopropyl-isotriazole

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

INNOVA-300 "sunofmr.uoregon.edu"

PULSE SEQUENCE

Relax: delay 1.000 sec

Pulse: 47.4 degrees

Acq. time 0.599 sec

Width 16501.7 Hz

2739 repetitions

OBSERVE C13, 75.4180944 MHz

DECOUPLE H1, 299.9335198 MHz

Power 35.06

VOLUNTARILY ON

WATERGATE

DATA PROCESSING

Line broadening 1.0 Hz

FI size 32768

Total time 3 hr, 7 min, 36 sec