Supporting Information for:

**Allylcyanation of Alkynes: Regio- and Stereoselective Access to
Functionalized Di- or Trisubstituted Acrylonitriles**

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General. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under an argon atmosphere or in a dry box under a nitrogen atmosphere. Flush column chromatography was performed using Kanto Chemical silica gel (spherical, 40–50 μm). Analytical thin layer chromatography (TLC) was performed on Merck Kieselgel 60 F₂₅₄ (0.25 mm) plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline KMnO_4 solution followed by heating.

Apparatus. Proton and carbon nuclear magnetic resonance spectra (^1H NMR and ^{13}C NMR) were recorded on a Varian Mercury 400 (^1H NMR, 400 MHz; ^{13}C NMR, 101 MHz) spectrometer with solvent resonance as the internal standard (^1H NMR, CHCl_3 at 7.26 ppm; ^{13}C NMR, CDCl_3 at 77.0 ppm). ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, br = broad, m = multiplet), coupling constants (Hz), and integration. IR spectra recorded on a Shimadzu FT-IR 8400 spectrometer are reported in cm^{-1} . Mass spectra (EI) were obtained with a JEOL JMS-700 spectrometer. GC analysis was performed on a Shimadzu GC 2010 equipped with a DB-5 column (30 m x 0.53 mm, pressure = 31.7 kPa, detector = FID, 290 $^\circ\text{C}$) with helium gas as a carrier. Preparative recycling silica gel chromatography was performed with a JAI LC-908 chromatograph equipped with COSMOSIL 5SL-II (250 mm x 20 mm) using hexane–ethyl acetate as an eluent.

Chemicals. Unless otherwise noted, commercially available reagents were used without purification. Anhydrous CH_3CN was purchased from Nacalai Tesque and bubbled vigorously with an argon gas for 20 min before use. Toluene was distilled from sodium/benzophenone ketyl. Allyl

cyanides, **1d** [using (*E*)-4,4-dimethyl-2-pentenyl methyl carbonate],¹ **1e**,¹ **1f**,² **1g**,³ **1h**,⁴ **1i**,³ **1j** (using phenyl vinyl ketone at 0 °C),³ and **1k**,² and alkyne **2h**⁵ were prepared according to the respective literature procedure.

Nickel-catalyzed allylcyanation of 4-octyne (2a). A general procedure. An allyl cyanide (4.0 mmol) and **2a** (110 mg, 1.0 mmol) were added to a solution of Ni(cod)₂ (28 mg, 0.10 mmol) and P(4-CF₃-C₆H₄)₃ (92 mg, 0.20 mmol) in CH₃CN (2.0 mL for **1a–1c**; 1.0 mL for **1d–1e**) in a dry box. The vial was taken outside the dry box and heated at 80 °C for the time specified in Table 1. The resulting mixture was filtered through a silica gel pad, concentrated in vacuo, and purified by flash silica gel column chromatography to give the corresponding allylcyanation products in yields listed in Table 1.

(Z)-2,3-Dipropyl-2,5-hexadienenitrile (3aa). A colorless oil, R_f 0.13 (hexane–ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 5.73 (ddt, *J* = 16.8, 10.1, 6.8 Hz, 1H), 5.12 (dd, *J* = 16.8, 1.6 Hz, 1H), 5.08 (dd, *J* = 10.1, 1.6 Hz, 1H), 3.12 (d, *J* = 6.8 Hz, 2H), 2.18 (t, *J* = 7.7 Hz, 2H), 2.13 (t, *J* = 8.0 Hz, 2H), 1.57 (sext, *J* = 7.5 Hz, 2H), 1.42 (sext, *J* = 7.5 Hz, 2H), 0.94 (t, *J* = 7.5 Hz, 3H), 0.92 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 157.0, 133.9, 119.0, 117.4, 111.0, 40.4, 33.2, 31.5, 21.7, 21.1, 14.1, 13.4; IR (neat) 2963, 2934, 2874, 2208, 1638, 1622, 1456, 1437, 1379, 993, 918 cm⁻¹; Anal. Calcd for C₁₂H₁₉N; C, 81.30; H, 10.80. Found: C, 81.05; H, 10.65.

(2Z)-2,3-Dipropyl-2,5-heptadienenitrile (3ba, 5E : 5Z = 85 : 15). A colorless oil, R_f 0.16 (hexane–ethyl acetate = 50:1). ¹H NMR (400 MHz, CDCl₃) δ 5.55 (dqt, *J* = 15.1, 6.4, 1.3 Hz, 0.85H), 5.65–5.52 (m, 0.15H), 5.36 (dtq, *J* = 15.1, 6.8, 1.5 Hz, 0.85H), 5.40–5.29 (m, 0.15H), 3.16 (d, *J* = 7.3 Hz, 0.30H), 3.05 (d, *J* = 6.8 Hz, 1.70H), 2.21–2.10 (m, 4H), 1.70 (ddt, *J* = 7.0, 1.9, 0.9 Hz, 0.45H), 1.67 (dq, *J* = 6.4, 1.5 Hz, 2.55H), 1.59 (sext, *J* = 7.5 Hz, 2H), 1.41 (sext, *J* = 7.6 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H); ¹³C NMR [for (2Z,5E)-**3ba**, 101 MHz, CDCl₃] δ 158.0, 128.2, 126.5, 119.2, 110.2, 39.3, 33.2, 31.5, 21.7, 21.2, 17.9, 14.1, 13.5; IR (neat) 2963, 2934, 2874, 2208, 1622, 1456, 1379, 1082, 966 cm⁻¹;

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Anal. Calcd for C₁₃H₂₁N; C, 81.61; H, 11.06. Found: C, 81.81; H, 11.24.

(2Z,5E)-7,7-Dimethyl-2,3-dipropyl-2,5-octadienenitrile (3da). A colorless oil, *R_f* 0.33 (hexane–ethyl acetate = 30:1). ¹H NMR (400 MHz, CDCl₃) δ 5.57 (dt, *J* = 15.4, 1.3 Hz, 1H), 5.23 (dt, *J* = 15.6, 7.0 Hz, 1H), 3.06 (d, *J* = 7.0 Hz, 2H), 2.18 (t, *J* = 7.6 Hz, 2H), 2.11 (t, *J* = 8.0 Hz, 2H), 1.58 (sext, *J* = 7.5 Hz, 2H), 1.41 (sext, *J* = 7.5 Hz, 2H), 0.99 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 158.2, 144.8, 120.1, 119.2, 110.1, 39.6, 33.1, 33.0, 31.5, 29.5, 21.7, 21.2, 14.1, 13.4; IR (neat) 2961, 2872, 2208, 1464, 1364, 972, 471 cm⁻¹; Anal. Calcd for C₁₆H₂₇N; C, 82.34; H, 11.66. Found: C, 82.55; H, 11.49.

(2Z,5E)-2,3-Dipropyl-6-phenyl-2,5-hexadienenitrile (3ea). A colorless oil, *R_f* 0.18 (hexane–ethyl acetate = 30:1). ¹H NMR (400 MHz, CDCl₃) δ 7.38–7.27 (m, 4H), 7.23 (tt, *J* = 7.1, 1.5 Hz, 1H), 6.49 (d, *J* = 15.7 Hz, 1H), 6.12 (dt, *J* = 15.7, 7.1 Hz, 1H), 3.30 (d, *J* = 7.1 Hz, 2H), 2.27–2.15 (m, 4H), 1.60 (sext, *J* = 7.5 Hz, 2H), 1.47 (sext, *J* = 7.7 Hz, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 157.1, 137.0, 132.6, 128.5, 127.4, 126.2, 125.6, 119.1, 111.0, 39.6, 33.3, 31.6, 21.7, 21.2, 14.1, 13.5; IR (neat) 3026, 2963, 2932, 2872, 2206, 1622, 1497, 1448, 1381, 1113, 966, 912, 735, 694 cm⁻¹; Anal. Calcd for C₁₈H₂₃N; C, 85.32; H, 9.15. Found: C, 85.58; H, 9.44.

Nickel-catalyzed carbocyanation of alkynes using α-siloxyallyl cyanides. A general procedure. An α-siloxyallyl cyanides (1.5 mmol) and an alkyne (1.0 mmol) were added to a solution of Ni(cod)₂ (28 mg, 0.10 mmol) and P(4-CF₃-C₆H₄)₃ (92 mg, 0.20 mmol) in CH₃CN (1.0 mL) in a dry box. The vial was taken outside the dry box and heated at 80 °C for the time specified in Scheme 1 or Table 2. The resulting mixture was filtered through a silica gel pad and concentrated in vacuo. The residue was dissolved in THF (10 mL) and treated with a 1 M HCl aqueous solution (5 mL) at 0 °C. The mixture was stirred at rt overnight and extracted with Et₂O. Combined organic layers were washed with water (10 mL x 3) and brine (10 mL), dried over anhydrous MgSO₄, and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (**3fb** was purified by Kugelrohr distillation) to give the corresponding allylcyanation products in yields listed in Scheme 1 or Table 2. In some cases, regioisomers were further separated by preparative recycling silica gel chromatography.

(Z)-6-Oxo-2,3-dipropyl-2-hexenenitrile (3fa). A colorless oil, R_f 0.25 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.79 (s, 1H), 2.74–2.58 (m, 4H), 2.17 (t, J = 7.7 Hz, 2H), 2.13 (t, J = 7.9 Hz, 2H), 1.56 (sext, J = 7.5 Hz, 2H), 1.43 (sext, J = 7.5 Hz, 2H), 0.93 (t, J = 7.4 Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.3, 157.5, 118.7, 111.5, 42.2, 33.4, 31.4, 28.2, 21.6, 21.3, 14.0, 13.4; IR (neat) 2963, 2934, 2874, 2206, 1726, 1624, 1456, 1381, 1123, 1088, 1049 cm^{-1} ; Anal. Calcd for $\text{C}_{12}\text{H}_{19}\text{NO}$: C, 74.57; H, 9.91. Found: C, 74.64; H, 9.85.

(2Z,5E)-6-(tert-Butyldimethylsilyloxy)-2,3-dipropyl-2,5-hexadienenitrile [(E)-3ga]. A colorless oil, R_f 0.31 (hexane–ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 6.36 (dt, J = 11.7, 1.2 Hz, 1H), 4.93 (dt, J = 11.7, 7.6 Hz, 1H), 2.94 (d, J = 7.5 Hz, 2H), 2.17 (t, J = 7.6 Hz, 2H), 2.13 (t, J = 7.9 Hz, 2H), 1.62–1.50 (m, 2H), 1.41 (sext, J = 7.5 Hz, 2H), 0.94 (t, J = 7.4 Hz, 3H), 0.92 (t, J = 7.2 Hz, 3H), 0.91 (s, 9H), 0.14 (s, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 158.5, 142.7, 119.1, 109.9, 107.0, 34.1, 32.9, 31.5, 25.6, 21.7, 21.1, 18.3, 14.1, 13.5, –5.3; IR (neat) 2961, 2932, 2858, 2206, 1661, 1464, 1256, 1196, 1169, 928, 839, 783 cm^{-1} ; Anal. Calcd for $\text{C}_{18}\text{H}_{33}\text{NOSi}$: C, 70.30; H, 10.82. Found [as a mixture with (Z)-3ga]: C, 70.56; H, 11.02.

(2Z,5Z)-6-(tert-Butyldimethylsilyloxy)-2,3-dipropyl-2,5-hexadienenitrile [(Z)-3ga]. A colorless oil, R_f 0.31 (hexane–ethyl acetate = 20:1). ^1H NMR (400 MHz, CDCl_3) δ 6.29 (dt, J = 5.7, 1.4 Hz, 1H), 4.39 (td, J = 7.4, 5.7 Hz, 1H), 3.20 (d, J = 7.5 Hz, 2H), 2.17 (t, J = 7.3 Hz, 2H), 2.13 (t, J = 7.9 Hz, 2H), 1.56 (sext, J = 7.5 Hz, 2H), 1.44 (sext, J = 7.5 Hz, 2H), 0.94 (t, J = 7.4 Hz, 3H), 0.93 (s, 9H), 0.91 (t, J = 7.4 Hz, 3H), 0.14 (s, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 159.2, 140.5, 119.4, 109.5, 105.5, 33.0, 31.5, 30.6, 25.6, 21.7, 21.3, 18.2, 14.1, 13.5, –5.4; IR (neat) 2961, 2932, 2860, 2206, 1651, 1258, 1103, 837, 781 cm^{-1} .

(Z)-6-Oxo-2,3-dipropyl-2-heptenenitrile (3ha). A colorless oil, R_f 0.17 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 2.66–2.57 (m, 4H), 2.18 (s, 3H), 2.17 (t, J = 7.6 Hz, 2H), 2.12 (t, J = 7.9 Hz, 2H), 1.56 (sext, J = 7.5 Hz, 2H), 1.43 (sext, J = 7.7 Hz, 2H), 0.94 (t, J = 7.3 Hz, 3H), 0.93 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 206.8, 158.2, 118.8, 110.9, 41.8, 33.4, 31.3, 29.7, 29.6, 21.6, 21.2, 14.0, 13.3; IR (neat) 2934, 2874, 2206, 1715, 1624, 1456, 1362, 1163 cm^{-1} ; Anal. Calcd for $\text{C}_{13}\text{H}_{21}\text{NO}$:

C, 75.32; H, 10.21. Found: C, 75.30; H, 9.96.

(Z)-6-Oxo-2,3-dipropyl-2-octenenitrile (3ia). A colorless oil, R_f 0.37 (hexane–ethyl acetate = 5:1) ^1H NMR (400 MHz, CDCl_3) δ 2.67–2.53 (m, 4H), 2.47 (q, $J = 7.3$ Hz, 2H), 2.17 (t, $J = 7.6$ Hz, 2H), 2.12 (t, $J = 7.9$ Hz, 2H), 1.56 (sext, $J = 7.5$ Hz, 2H), 1.43 (sext, $J = 7.6$ Hz, 2H), 1.07 (t, $J = 7.4$ Hz, 3H), 0.94 (t, $J = 7.4$ Hz, 3H), 0.93 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 209.5, 158.4, 118.8, 110.8, 40.5, 35.6, 33.4, 31.3, 29.8, 21.5, 21.2, 13.9, 13.3, 7.6; IR (neat) 2964, 2936, 2874, 2206, 1717, 1458, 1111, 735 cm^{-1} ; Anal. Calcd for $\text{C}_{14}\text{H}_{23}\text{NO}$: C, 75.97; H, 10.47. Found: C, 75.80; H, 10.24.

(Z)-5-Benzoyl-2,3-dipropyl-2-pentenitrile (3ja). A colorless oil, R_f 0.30 (hexane–ethyl acetate = 5:1) ^1H NMR (400 MHz, CDCl_3) δ 7.98 (d, $J = 7.1$ Hz, 2H), 7.58 (t, $J = 7.5$ Hz, 1H), 7.47 (t, $J = 7.5$ Hz, 2H), 3.15 (t, $J = 7.9$ Hz, 2H), 2.81 (t, $J = 7.9$ Hz, 2H), 2.25–2.14 (m, 4H), 1.58 (sext, $J = 7.5$ Hz, 2H), 1.47 (sext, $J = 7.5$ Hz, 2H), 0.96 (t, $J = 7.3$ Hz, 3H), 0.95 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 198.4, 158.6, 136.4, 133.2, 128.6, 128.0, 118.9, 111.1, 37.2, 33.7, 31.5, 30.3, 21.7, 21.3, 14.1, 13.4; IR (neat) 2963, 2874, 2208, 1686, 1597, 1448, 1205, 735, 691 cm^{-1} ; Anal. Calcd for $\text{C}_{18}\text{H}_{23}\text{NO}$: C, 80.26; H, 8.61. Found: C, 80.53; H, 8.55.

(Z)-6-Oxo-5-methyl-2,3-dipropyl-2-hexenenitrile (3ka). A colorless oil, R_f 0.30 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.66 (s, 1H), 2.78 (dd, $J = 13.2, 5.7$ Hz, 1H), 2.62–2.52 (m, 1H), 2.49 (dd, $J = 13.2, 8.4$ Hz, 1H), 2.27–2.03 (m, 4H), 1.58 (sext, $J = 7.5$ Hz, 2H), 1.52–1.36 (m, 2H), 1.17 (d, $J = 7.0$ Hz, 3H), 0.95 (t, $J = 7.3$ Hz, 3H), 0.94 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 203.1, 156.0, 118.9, 112.7, 45.2, 36.2, 33.2, 31.5, 21.6, 21.3, 14.0, 13.3, 13.2; IR (neat) 2963, 2934, 2874, 2208, 1697, 1634, 1558, 1456, 1379 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{21}\text{NO}$: M^+ , 207.1623. Found: m/z 207.1624.

(Z)-6-Oxo-2,3-dimethyl-2-hexenenitrile (3fb). A colorless oil, bp 100 $^\circ\text{C}$ (1.0 mmHg), R_f 0.14 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 1H), 3.81 (t, $J = 7.2$ Hz, 2H), 2.67–2.60 (m, 2H), 1.88 (s, 3H), 1.82 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.3, 153.7, 119.2, 105.1, 41.6, 30.4, 18.1, 16.2; IR (neat) 3420, 2932, 2210, 1722, 1636, 1447, 1387, 1138, 733 cm^{-1} ; Anal. Calcd for $\text{C}_8\text{H}_{11}\text{NO}$: C, 70.04; H, 8.08. Found: C, 69.77; H, 7.82.

(Z)-6-Oxo-2-ethyl-3-methyl-2-hexenenitrile (3fc). A colorless oil, R_f 0.38 (hexane–ethyl acetate = 2:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (t, J = 1.4 Hz, 1H), 2.76–2.68 (m, 2H), 2.67–2.60 (m, 2H), 2.22 (q, J = 7.6 Hz, 2H), 1.83 (s, 3H), 1.12 (t, J = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.3, 152.6, 118.5, 112.6, 41.8, 30.6, 23.3, 17.9, 12.6; IR (neat) 2974, 2936, 2876, 2829, 2729, 2208, 1724, 1630, 1452, 1410, 1387, 1067, 733 cm^{-1} ; Anal. Calcd for $\text{C}_9\text{H}_{13}\text{NO}$: C, 71.49 H, 8.67. Found (as a mixture with **4fc**): C, 71.55; H, 8.75.

(Z)-6-Oxo-3-ethyl-2-methyl-2-hexenenitrile (4fc). A colorless oil, R_f 0.33 (hexane–ethyl acetate = 2:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (t, J = 1.3 Hz, 1H), 2.75–2.68 (m, 2H), 2.66–2.59 (m, 2H), 2.18 (q, J = 7.6 Hz, 2H), 1.89 (s, 3H), 1.03 (t, J = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.3, 159.2, 119.4, 105.1, 42.0, 27.8, 24.7, 15.8, 11.8; IR (neat) 2974, 2937, 2878, 2829, 2729, 2210, 1724, 1632, 1450, 1410, 1389, 733 cm^{-1} .

(Z)-6-Oxo-3-methyl-2-phenyl-2-hexenenitrile (3fd). A pale yellow oil, R_f 0.26 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.89 (s, 1H), 7.44–7.25 (m, 5H), 2.91 (t, J = 7.4 Hz, 2H), 2.78 (t, J = 7.4 Hz, 2H), 1.91 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 199.9, 156.1, 133.5, 128.9, 128.5, 128.4, 118.1, 112.0, 41.5, 30.6, 19.5; IR (neat) 3447, 2940, 2210, 1722, 1616, 1493, 1447, 1377, 1136, 766, 700 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}$: M^+ , 199.0997. Found: m/z 199.0991.

(E)-6-Oxo-2-methyl-3-phenyl-2-hexenenitrile (4fd). A pale yellow oil, R_f 0.29 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.68 (t, J = 1.1 Hz, 1H), 7.44–7.33 (m, 3H), 7.12–7.06 (m, 2H), 3.07 (t, J = 7.6 Hz, 2H), 2.47 (td, J = 7.6, 1.1 Hz, 2H), 1.82 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.0, 156.4, 136.9, 128.8, 128.7, 127.5, 119.1, 107.1, 41.5, 30.7, 17.7; IR (neat) 2961, 2924, 2856, 2212, 1722, 1493, 1441, 702 cm^{-1} ; HRMS(EI) Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}$: M^+ , 199.0997. Found: m/z 199.0997.

(Z)-6-Oxo-2-hexyl-2-hexenenitrile (3fe). A colorless oil, R_f 0.28 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.79 (s, 1H), 6.15 (t, J = 7.4 Hz, 1H), 2.72–2.59 (m, 4H), 2.18 (t, J = 7.6 Hz, 2H),

1.56–1.22 (m, 8H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.5, 145.0, 117.5, 116.6, 42.7, 34.4, 31.6, 28.5, 28.1, 24.2, 22.7, 14.2; IR (neat) 3420, 2930, 2858, 2216, 1717, 1456 cm^{-1} ; Anal. Calcd for $\text{C}_{12}\text{H}_{19}\text{NO}$: C, 74.57; H, 9.91. Found (as a mixture with **4fe**): C, 74.31; H, 9.73.

(Z)-6-Oxo-3-hexyl-2-hexenenitrile (4fe). A colorless oil, R_f 0.28 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.81 (s, 1H), 5.16 (t, $J = 1.4$ Hz, 1H), 2.75–2.63 (m, 4H), 2.17 (td, $J = 7.6, 1.4$ Hz, 2H), 1.49–1.20 (m, 8H), 0.89 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 199.8, 167.4, 116.7, 96.0, 41.7, 36.3, 31.5, 28.7, 27.03, 27.02, 22.5, 14.0; IR (neat) 2930, 2858, 2216, 1726, 1626, 1454 cm^{-1} .

(Z)-6-Oxo-2-tert-butyl-2-hexenenitrile (3ff). A colorless oil, R_f 0.22 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.79 (s, 1H), 6.16 (t, $J = 7.2$ Hz, 1H), 2.72–2.60 (m, 4H), 1.16 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3) δ 200.2, 140.4, 127.0, 116.6, 42.5, 34.8, 28.7, 24.0; IR (neat) 2966, 2872, 2214, 1726, 1369, 1140, 1042, 662 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{10}\text{H}_{15}\text{NO}$: M^+ , 165.1154. Found: m/z 165.1161.

(Z)-6-Oxo-2-(3-chloroprop-1-yl)-2-hexenenitrile (3fg). A yellow oil, R_f 0.17 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 1H), 6.26 (tt, $J = 1.3, 7.5$ Hz, 1H), 3.55 (t, $J = 6.2$ Hz, 2H), 2.70–2.62 (m, 4H), 2.41 (t, $J = 7.3$ Hz, 2H), 2.07–1.96 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 199.9, 146.5, 116.8, 114.3, 43.2, 42.2, 31.1, 30.2, 24.0; IR (neat) 2959, 2845, 2216, 1724, 1445, 914, 733, 650 cm^{-1} ; Anal. Calcd for $\text{C}_9\text{H}_{12}\text{ClNO}$: C, 58.23; H, 6.52. Found: C, 57.97; H, 6.50.

Methyl (Z)-9-oxo-5-cyano-5-nonenoate (3fh, containing 5% of 4fh).

A yellow oil, R_f 0.19 (hexane–ethyl acetate = 2:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 0.05H), 9.79 (s, 0.95H), 6.20 (t, $J = 7.5$ Hz, 0.95H), 5.19 (s, 0.05H), 3.68 (s, 3H), 2.73–2.60 (m, 4H), 2.33 (t, $J = 7.2$ Hz, 2H), 2.26 (t, $J = 7.6$ Hz, 2H), 1.87 (quint, $J = 7.5$ Hz, 2H); ^{13}C NMR (for **3o**, 101 MHz, CDCl_3) δ 199.9, 173.1, 145.8, 116.8, 115.1, 51.6, 42.2, 33.2, 32.5, 23.9, 23.0; IR (neat) 2953, 2216, 1732, 1437, 1254, 1200, 1167 cm^{-1} ; Anal. Calcd for $\text{C}_{11}\text{H}_{15}\text{NO}_3$: C, 63.14; H, 7.23. Found: C, 63.06; H, 7.28.

3fi

***N*-(*Z*)-9-Oxo-5-cyano-5-nonenylphthalimide (3fi, containing 10% of 4fi).** A yellow oil, R_f 0.19 (hexane–ethyl acetate = 2:1).

^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 0.10H), 9.78 (s, 0.90H), 7.87–7.79 (m, 2H), 7.75–7.66 (m, 2H), 6.20 (br t, J = 7.5 Hz, 0.90H), 5.17 (s, 0.10H), 3.68 (t, J = 7.0 Hz, 2H), 2.70–2.60 (m, 4H), 2.26 (t, J = 7.3 Hz, 2H), 1.86–1.39 (m, 4H); ^{13}C NMR (for **3fi**, 101 MHz, CDCl_3) δ 200.1, 168.3, 145.5, 134.0, 132.0, 123.2, 117.0, 115.5, 42.4, 37.2, 33.5, 27.3, 25.0, 24.0; IR (neat) 2941, 2216, 1771, 1713, 1437, 1396, 1371, 1038, 916, 721, 530 cm^{-1} ; Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_3$: C, 69.66; H, 5.85. Found: C, 69.86; H, 6.02.

3fj

(*E*)-6-Oxo-2-trimethylsilyl-2-hexenenitrile (3fj). A yellow oil, R_f 0.18 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 1H), 6.57 (t, J = 7.0 Hz, 1H), 2.79 (q, J = 7.0 Hz, 2H), 2.67 (t, J = 7.0 Hz, 2H), 0.22 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3) δ 199.9, 159.0, 117.8, 117.2, 42.0, 26.6, –2.2; IR (neat) 2959, 2899, 2827, 2727, 2199, 1726, 1595, 1254, 847, 760 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_9\text{H}_{15}\text{NOSi}$: M^+ , 181.0923. Found: m/z 181.0915.

5

Reduction of 3aa using DIBAL-H. To a solution of **3aa** (53 mg, 0.30 mmol) in toluene (3.0 mL) was added a 1.5 M solution of DIBAL-H in toluene (0.50 mL, 0.75 mmol) at –78 °C, and the resulting mixture was stirred at the same temperature for 1 h.

The reaction was quenched with MeOH (0.150 mL) at –78 °C and was warmed at rt.

The mixture was diluted with CH_2Cl_2 , filtered through a Celite pad, and concentrated in vacuo. The residue was purified by flash chromatography on silica gel (hexane–ethyl acetate = 25:1 as an eluent) to give (*Z*)-2,3-dipropyl-2,5-hexadienal (**5**, 49 mg, 90%) as a colorless oil, R_f 0.30 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 10.02 (s, 1H), 5.89–5.77 (m, 1H), 5.12–5.00 (m, 2H), 3.29 (d, J = 6.0 Hz, 2H), 2.28–2.18 (m, 4H), 1.49 (sext, J = 7.6 Hz, 2H), 1.33 (sext, J = 7.6 Hz, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.91 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 191.9, 158.8, 137.9, 135.6, 116.7, 36.8, 34.5, 27.4, 22.8, 21.5, 14.4, 14.3; IR (neat) 2961, 2932, 2872, 1670, 1616, 1464, 1080, 991, 916 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{12}\text{H}_{20}\text{O}$: M^+ , 180.1514. Found: m/z 180.1523.

6

Reduction of 5 using LiAlH_4 . To a suspension of LiAlH_4 (51 mg, 1.34 mmol) in THF (1.4 mL) was added a solution of **5** (49 mg, 0.27 mmol) in THF (1.5 mL) at rt, and the resulting mixture was stirred for 10 min. The mixture was diluted with diethyl ether (4.0 mL), quenched with H_2O (75 μL) at 0 °C, dried over anhydrous MgSO_4 , filtered,

and concentrated in vacuo. The residue was purified by flash chromatography on silica gel to give (Z)-2,3-dipropyl-2,5-hexadienol (**6**, 45 mg, 91%) as a colorless oil, R_f 0.25 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 5.87–5.74 (m, 1H), 5.08–4.94 (m, 2H), 4.08 (s, 2H), 2.86 (dt, J = 6.0, 1.6 Hz, 2H), 2.17–2.09 (m, 2H), 2.05–1.98 (m, 2H), 1.50–1.31 (m, 4H), 1.30 (br s, 1H), 0.92 (t, J = 7.3 Hz, 3H), 0.91 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 137.6, 135.9, 134.9, 114.7, 62.0, 36.0, 34.4, 32.7, 22.3, 21.8, 14.34, 14.30; IR (neat) 3319, 2959, 2932, 2870, 1636, 1466, 1377, 1011, 910, 737 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{12}\text{H}_{22}\text{O}$: M^+ , 182.1671. Found: m/z 182.1666.

Reduction of **3fa using NaBH_4 .** To a solution of **3fa** (19.3 mg, 0.100 mmol) in MeOH (0.33 mL) was added NaBH_4 (0.140 g, 0.30 mmol) portionwise at 0 °C, and the resulting mixture was stirred at the same temperature for 10 min. The reaction was quenched with a 1.0 M HCl aqueous solution (5 mL) at 0 °C, and the resulting mixture was extracted with diethyl ether. The combined organic layers were washed with a saturated NaHCO_3 aqueous solution and brine, dried over anhydrous MgSO_4 , concentrated in vacuo, and purified by flash chromatography on silica gel to give (Z)-6-hydroxyl-2,3-dipropyl-2-hexenenitrile (**7**, 15.8 mg, 81%) as a colorless oil, R_f 0.13 (hexane–ethyl acetate = 3:1). ^1H NMR (400 MHz, CDCl_3) δ 3.67 (t, J = 6.4 Hz, 2H), 2.46 (t, J = 7.8 Hz, 2H), 2.22–2.10 (m, 4H), 1.79–1.65 (m, 3H), 1.56 (sext, J = 7.5 Hz, 2H), 1.43 (sext, J = 7.6 Hz, 2H), 0.933 (t, J = 7.5 Hz, 3H), 0.929 (t, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 159.5, 119.4, 110.4, 62.0, 33.4, 32.2, 31.4, 31.2, 21.7, 21.3, 14.1, 13.4; IR (neat) 3439, 2961, 2934, 2874, 2208, 1622, 1458, 1061 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{12}\text{H}_{21}\text{NO}$: M^+ , 195.1623. Found: m/z 195.1615.

Methylenation of the formyl group of **3fa using $\text{CH}_2(\text{ZnI})_2$.**⁶ To a solution of **3fa** (19.3 mg, 0.100 mmol) in THF (0.50 mL) was added a 0.40 M solution of $\text{CH}_2(\text{ZnI})_2$ in THF (1.00 mL, 0.40 mmol) at rt, and the resulting mixture was stirred for 0.5 h. The reaction was quenched with MeOH at 0 °C, and a saturated NH_4Cl aqueous solution was added to the mixture. The resulting bilayer solution was extracted with diethyl ether, and the combined organic layers were washed with a saturated NH_4Cl aqueous solution and brine, dried over anhydrous MgSO_4 , and concentrated in vacuo. The residue was purified by flash chromatography on silica gel to give (Z)-2,3-dipropyl-2,6-heptadienenitrile (**8**, 13.7 mg, 72%) and unreacted **3fa** (3.6 mg, 19%). **8** is a colorless oil, R_f 0.38 (hexane–ethyl acetate = 15:1). ^1H NMR (400 MHz, CDCl_3) δ 5.88–5.74 (m, 1H), 5.09–4.95 (m, 2H), 2.48 (t, J = 7.7 Hz, 2H), 2.28–2.08 (m, 6H), 1.56 (sext, J = 7.4

(6) Matsubara, S.; Mizuno, T.; Otake, T.; Kobata, M.; Utimoto, K.; Takai, K. *Synlett*, **1998**, 1369–1371.

Hz, 2H), 1.43 (sext, $J = 7.6$ Hz, 2H), 0.94 (t, $J = 7.3$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 158.8, 137.0, 119.2, 115.6, 110.6, 35.3, 33.3, 32.5, 31.5, 21.7, 21.3, 14.1, 13.4; IR (neat) 2963, 2934, 2874, 2208, 1641, 1622, 1456, 1381, 995, 912 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{21}\text{N}$: M^+ , 191.1674. Found: m/z 191.1683.

α -Methylenation of 3fa.⁷ To a mixture of **3fa** (58 mg, 0.30 mmol), a 37% formaldehyde aqueous solution (37 mg, 0.45 mmol), and 2-propanol (30 μL) were added propionic acid (2.2 mg, 30 μmol) and pyrrolidine (2.1 mg, 30 μmol). The resulting mixture was stirred at 45 $^{\circ}\text{C}$ for 24 h, concentrated in vacuo, and purified

by flash chromatography on silica gel to give (*Z*)-6-oxo-5-methylene-2,3-dipropyl-2-hexenenitrile (**9**, 51 mg, 83%) as a colorless oil, R_f 0.23 (hexane–ethyl acetate = 5:1). ^1H NMR (400 MHz, CDCl_3) δ 9.59 (s, 1H), 6.27 (t, $J = 1.5$ Hz, 1H), 6.14 (t, $J = 1.0$ Hz, 1H), 3.34 (s, 2H), 2.23 (t, $J = 7.6$ Hz, 2H), 2.06 (t, $J = 7.9$ Hz, 2H), 1.60 (sext, $J = 7.5$ Hz, 2H), 1.40 (sext, $J = 7.6$ Hz, 2H), 0.96 (t, $J = 7.4$ Hz, 3H), 0.90 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 193.5, 155.7, 146.2, 135.5, 119.0, 113.1, 33.5, 33.3, 31.5, 21.7, 21.3, 14.1, 13.5; IR (neat) 2963, 2934, 2874, 2208, 1693, 1626, 1466, 1437, 1344, 1242, 1111, 1086, 959, 735 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{13}\text{H}_{19}\text{NO}$: M^+ , 205.1467. Found: m/z 205.1469.

(7) Erkkilä, A.; Pihko, P. M. *J. Org. Chem.* **2006**, *71*, 2538–2541.

5513-70-06-96-clummm

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-96-H

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480270 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 23 sec

3ka

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: 06-96-13C

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

360 repetitions

OBSERVE C13, 100.5670190 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 41 min, 28 sec

5623-70-06-160-column-frc35-40

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-160-frc34-40

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480264 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 31 sec

3fd

5623-70-06-160-frc35-40-13C

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-160-frc35-40-13C

Mercury-400HB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

48 repetitions

OBSERVE C13, 100.5669929 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 57 min, 13 sec

3fd

5623-70-06-160-clumm-frc17-24

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: 06-160-frc17-24

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480266 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 31 sec

4fd

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-160-frc17-24-13C

Mercury-400BE "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

432 repetitions

OBSERVE C13, 100.5670128 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 57 min, 13 sec

4fd

5705-70-06-191-H

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-191-H

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480266 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 31 sec

5705-70-06-191-13C

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 06-191-13C

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

72 repetitions

OBSERVE C13, 100.5670174 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 57 min, 13 sec

5820-70-07-57-H

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: 07-57-H

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480275 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 31 sec

3fj

¹³C OBSERVE

Pulse Sequence: s2pul1

Solvent: CDCl₃

Ambient temperature

File: 06-194-13C

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

120 repetitions

OBSERVE C13, 100.5670159 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 57 min, 13 sec

6406-62-17-0 -frc.9-15

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480270 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 42 sec

5

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

1668 repetitions

OBSERVE C13, 100.5670121 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 909 hr, 17 min, 12 sec

6415-62-17-025-column-2

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480268 MHz

DATA PROCESSING

Line broadening 0.2 Hz

Ft size 65536

Total time 1 min, 42 sec

6415-62-17-025-column-2

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

318 repetitions

OBSERVE C13, 100.5670136 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 62536

Total time 1 hr, 6 min, 54 sec

6314-62-16-19 -column

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480270 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 42 sec

6314-62-16-19 -column

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

Mercury-400EB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

186 repetitions

OBSERVE C13, 100.5670151 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 6 min, 54 sec

5804-62-14-006-column

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480273 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65336

Total time 1 min, 42 sec

5804-62-14-006-column

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

200 repetitions

OBSERVE C13, 100.5670121 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 6 min, 54 sec

8

77.320
77.000
76.680

35.302
33.312
32.465
31.474
21.738
21.280
14.136
13.404

119.247
115.626
110.609

137.012

158.803

ppm

160

140

120

100

80

60

40

20

0

6318-62-16-196-column

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Mercury-400BB "m400"

Relax. delay 1.500 sec

Pulse 45.0 degrees

Acq. time 3.502 sec

Width 5995.2 Hz

16 repetitions

OBSERVE H1, 399.9480268 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min, 42 sec

9

6318-62-16-196-column

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

Mercury-400BB "m400"

Relax. delay 0.801 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

1024 repetitions

OBSERVE C13, 100.5669874 MHz

DECOUPLE H1, 399.9500406 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 6 min, 54 sec

9