Supporting Information for:

Phosphine-Catalyzed [4+2] Annulation: Synthesis of Cyclohexenes

Yang S. Tran and Ohyun Kwon*

Department of Chemistry and Biochemistry, University of California, Los Angeles, California 90095-1569

Contents

1. General Information

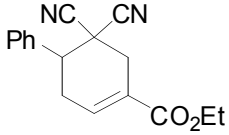
All reactions were performed under an argon atmosphere with dry solvents and anhydrous conditions, unless otherwise noted. Benzene, toluene, and dichloromethane were distilled afresh from CaH_2. All other reagents were used as received from commercial sources. All ethyl 2-substitutedmethylallenoates **1** were synthesized according to procedures reported previously.[1] The arylidenenemalononitriles **2** were obtained from commercially available sources or synthesized through phosphine-catalyzed Knoevenagel condensation of the pertinent aldehyde and malononitrile, according to the procedure reported by Yadav.[2] Reactions were monitored using thin layer chromatography (TLC) performed on 0.25-mm E. Merck silica gel plates (60F-254) and visualized under UV light or through permanganate staining. Flash column chromatography was performed using E. Merck silica gel 60 (230–400 mesh) and compressed air. IR spectra were recorded on a Perkin–Elmer pargon 1600 FT-IR spectrometer. NMR spectra were obtained on Bruker Avance-500, ARX-500, or Bruker Avance-300 instruments (as indicated), calibrated using residual undeuterated chloroform as an internal reference (7.26 and 77.0 ppm for ^{1}H and ^{13}C NMR spectra, respectively). ^{1}H NMR spectral data are reported as follows: chemical shift (δ, ppm), multiplicity, coupling constant (Hz), and integration. ^{13}C spectral data are reported in terms of the chemical shift. The following abbreviations are used to indicate multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; br = broad. High-resolution EI mass spectra were recorded after rapid thermal vaporization of samples deposited on a desorption ionization filament inserted directly into the electron ionization (EI, 70 eV, 200 °C) source of a triple-sector high-resolution instrument (VG/Micromass Autospec) tuned to 8000 static resolution (M/DM, 10% valley) using perfluorinated kerosene (formula

[1] Zhu, X.-F.; Lan, J. ; Kwon, O. *J. Am. Chem. Soc*. **2003**, *125*, 4716.
[2] Yadav, J. S.; Subba Reddy, B. V.; Basak, A. K.; Visali, B.; Narsaiah, A. V.; Nagaiah, K. *Eur. J. Org. Chem.* **2004**, 546.

weight 705, Lancaster Synthesis, Inc., NH) as the internal calibrant. High-resolution electrospray ionization (HRESI) mass spectra were recorded after flow injection of chloroform solutions into an ESI source attached to a 7.5-tesla FTMS (Ion Spec Ultima, Irvine, CA) instrument. Data were analyzed using the instrument-supplied software. X-ray crystallographic data were collected using a Bruker SMART CCD-based diffractometer equipped with a low-temperature apparatus operated at 100 K. Melting points (m.p.) are uncorrected and were recoded on an Electrothermal capillary melting point apparatus.

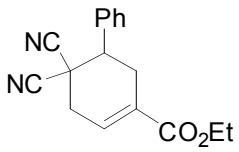
2. General Procedure for the Formation of Cyclohexenes 3/4

Ethyl 2-alkylallenoate **1** (1.2 mmol) in benzene (10 mL) was added slowly over 3 h via syringe under an argon atmosphere to a solution of arylidenemalononitrile **2** (1.0 mmol) and phosphine (0.2 mmol) in benzene (10 mL) at 80 °C. The mixture was heated under reflux and the progress of the reaction monitored using TLC. After 8 h, additional ethyl 2-alkylallenoate (0.2 mmol) in benzene (2 mL) was added over 5 min if it is necessary. After the reaction had reached completion [ca. 14 h, except for the reaction using $(4\text{-}ClC_6H_4)_3P$, which required 120 h], the resulting mixture was concentrated and the regioisomeric ratio determined using ^{1}H NMR spectroscopy. The crude residue was purified through flash column chromatography on silica gel (gradient eluent: 5–20% ethyl acetate in hexanes) to provide the cyclohexene derivatives **3** or **4**.



Ethyl 5,5-Dicyano-4-phenylcyclohex-1-enecarboxylate (3a). 98% yield as a white solid: m.p. 99–101 °C; IR (film) ν_{max} 2982, 2924, 2251, 2229, 1710, 1658, 1277, 1260, 1235, 1103, 700 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 5H), 7.23 (m, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.34 (dm, *J* = 17.7 Hz, 1H), 3.26 (dd, *J* = 10.4, 5.5 Hz, 1H), 3.06 (ddt, *J* = 17.7, 3.7, 2.1 Hz, 1H), 2.96 (dddt, *J* = 20.3, 10.4, 3.5, 2.6 Hz, 1H), 2.78 (dm, *J* = 20.3

Hz, 1H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.7, 138.4, 136.0, 129.3, 129.2, 128.0, 124.9, 114.8, 113.9, 61.3, 45.2, 37.1, 34.8, 29.2, 14.1; MS (MALDI) calcd for $C_{17}H_{16}N_2O_2Na$ $[M + Na]^+$ 303.111, found 303.115.



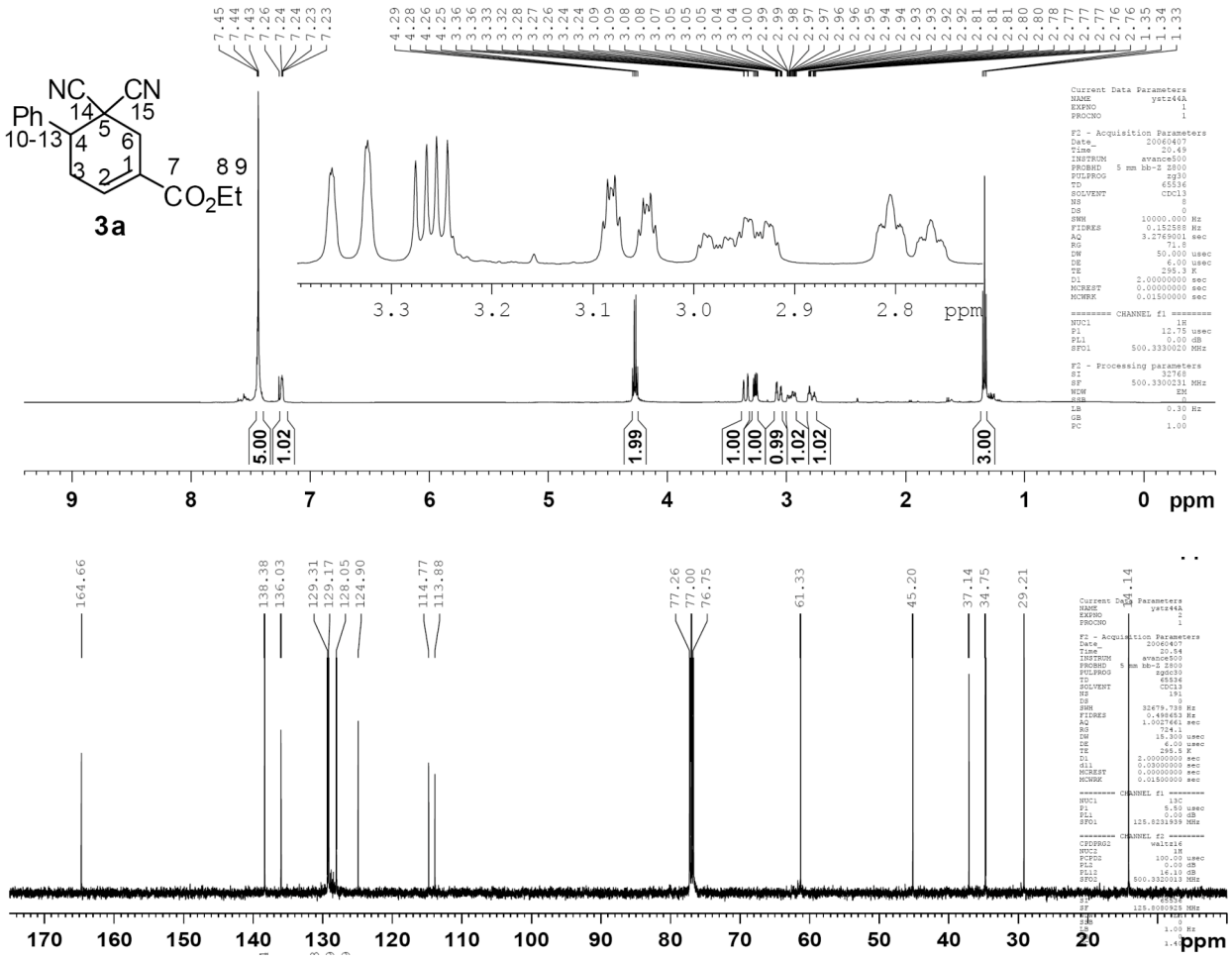
Ethyl 4,4-Dicyano-5-phenylcyclohex-1-enecarboxylate (4a). 93% yield as a foam: IR (film) ν_{max} 3065, 3035, 2984, 2252, 1714, 1660, 1550 1431, 1273, 1255, 1103, 732 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.48 (m, *5*H), 6.93 (m, 1H), 4.25 (qd, *J* = 7.1, 10 Hz, 2H), 3.24 (dd, *J* = 8.2, 8.2 Hz, 1H), 3.17 (ddt, *J* = 18.9, 5.1, 1.5 Hz, 1H), 3.07 (dq, *J* = 18.9, 3.0 Hz, 1H), 2.92–2.97 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.0, 136.3, 131.4, 130.3, 129.2, 129.1, 128.0, 114.8, 113.9, 61.2, 45.8, 36.6, 36.3, 27.7, 14.1; MS (MALDI) calcd for $C_{17}H_{16}N_2O_2Na$ $[M + Na]^+$ 303.111, found 303.132.

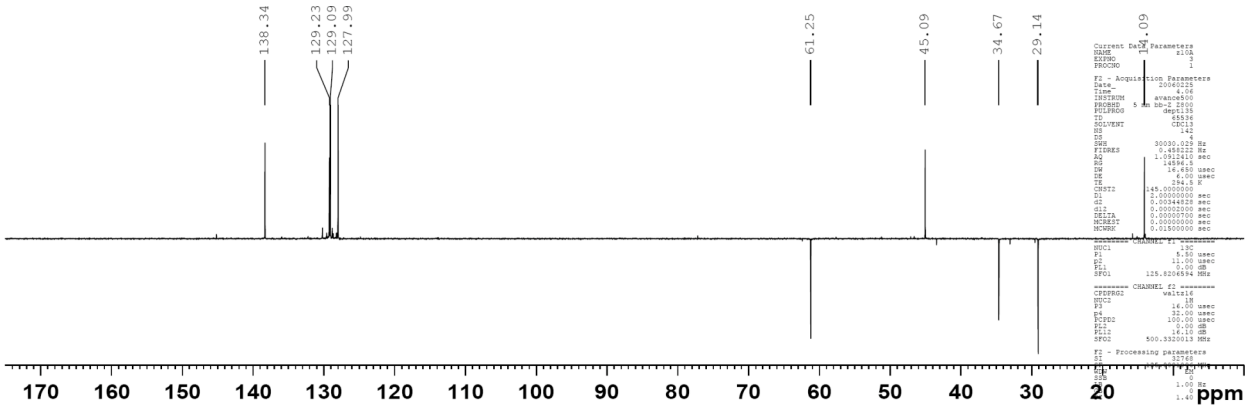
3. Structural Assignment of Cyclohexenes 3a and 4a Through 1D and 2D NMR Spectroscopic Analyses

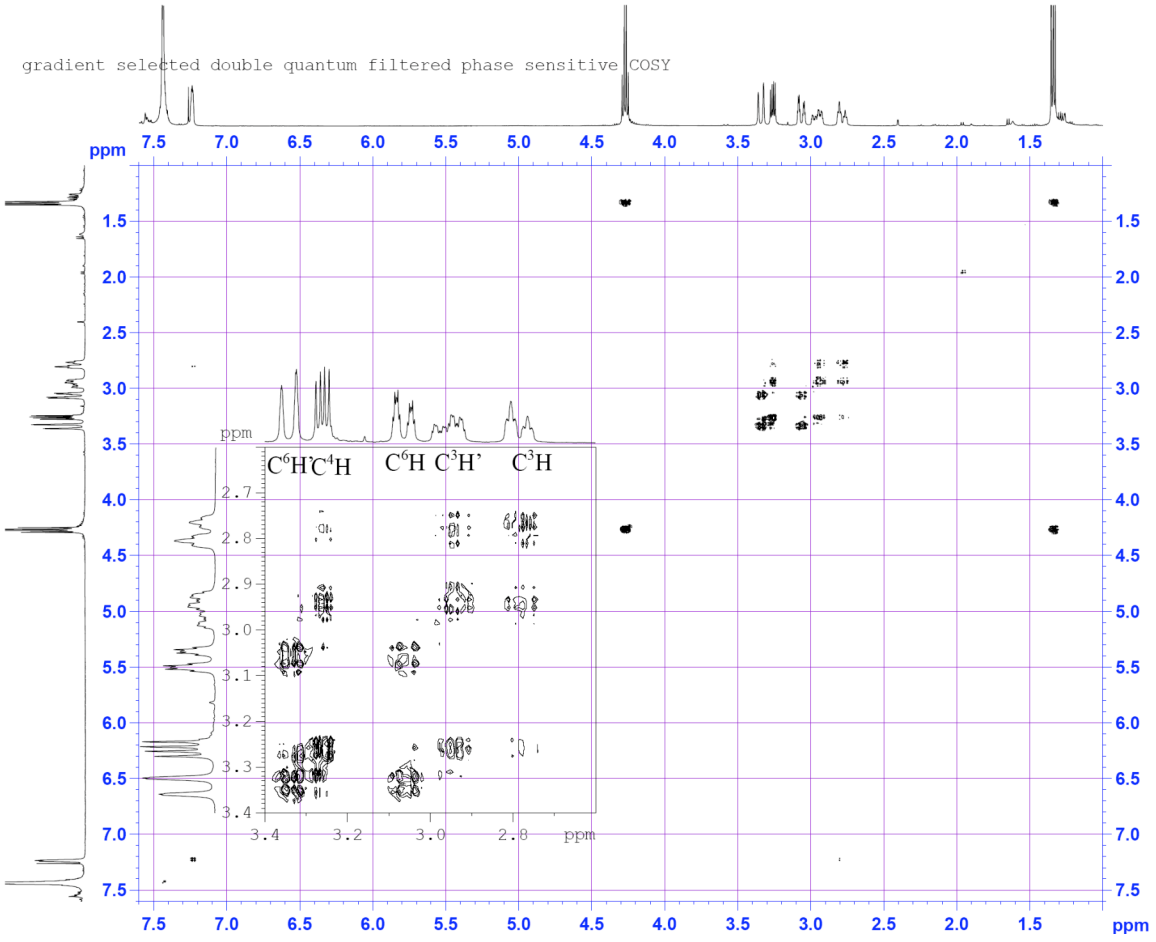
A. 1D and 2D NMR Spectra of Cyclohexene 3a

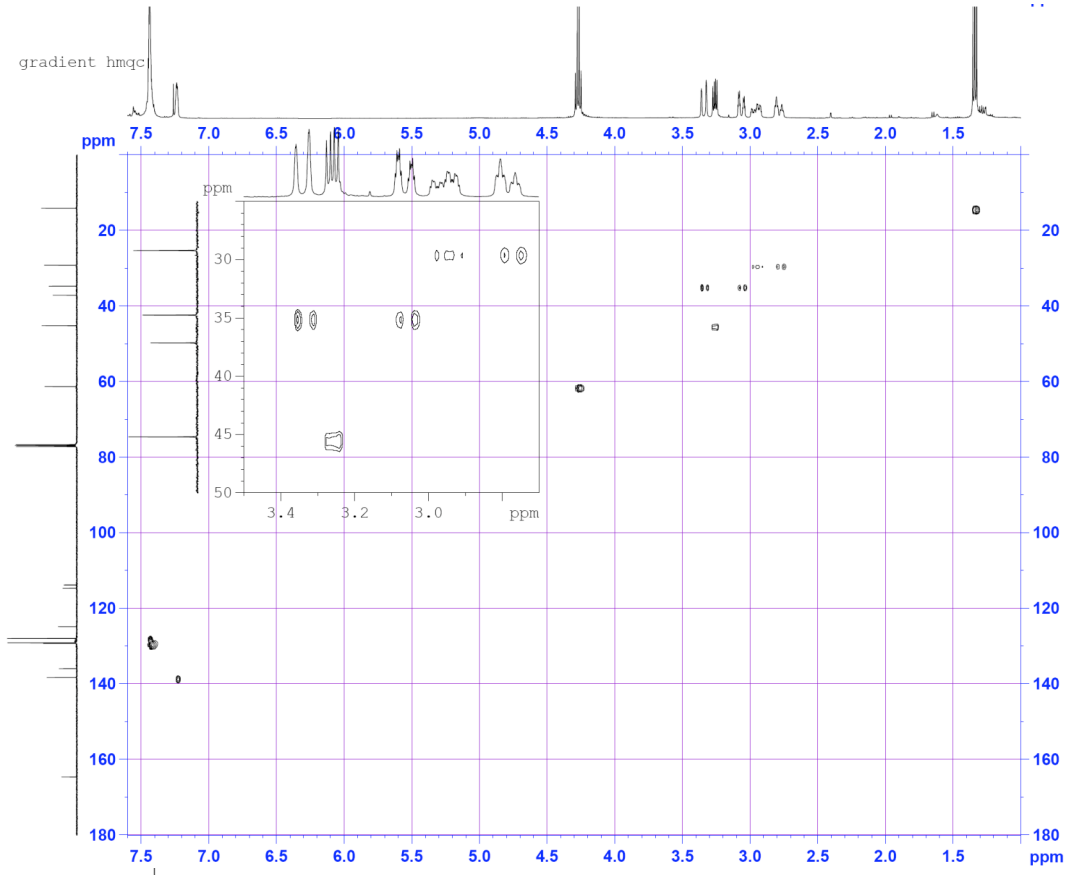
The structure of cyclohexene **3a** was assigned based on MS (MALDI) and ^{1}H, ^{13}C, ^{13}C-DEPT, 2D-COSY, HMQC, and HMBC NMR spectroscopy experiments and further confirmed through single-crystal X-ray crystallographic analysis of cyclohexene **3d**. MS (MALDI) of cyclohexene **3a** gave a molecular ion at *m*/*z* 303.115 (M + Na), in accordance with the molecular formula $C_{17}H_{16}N_2O_2$ with sodium incorporated. The ^{1}H NMR spectrum indicated a total of sixteen protons: five aromatic protons (7.3–7.5 ppm) belonging to the phenyl ring, one vinylic proton (7.23 ppm) for the α,β-unsaturated carboxylic ester, five protons for the ethyl carboxylic

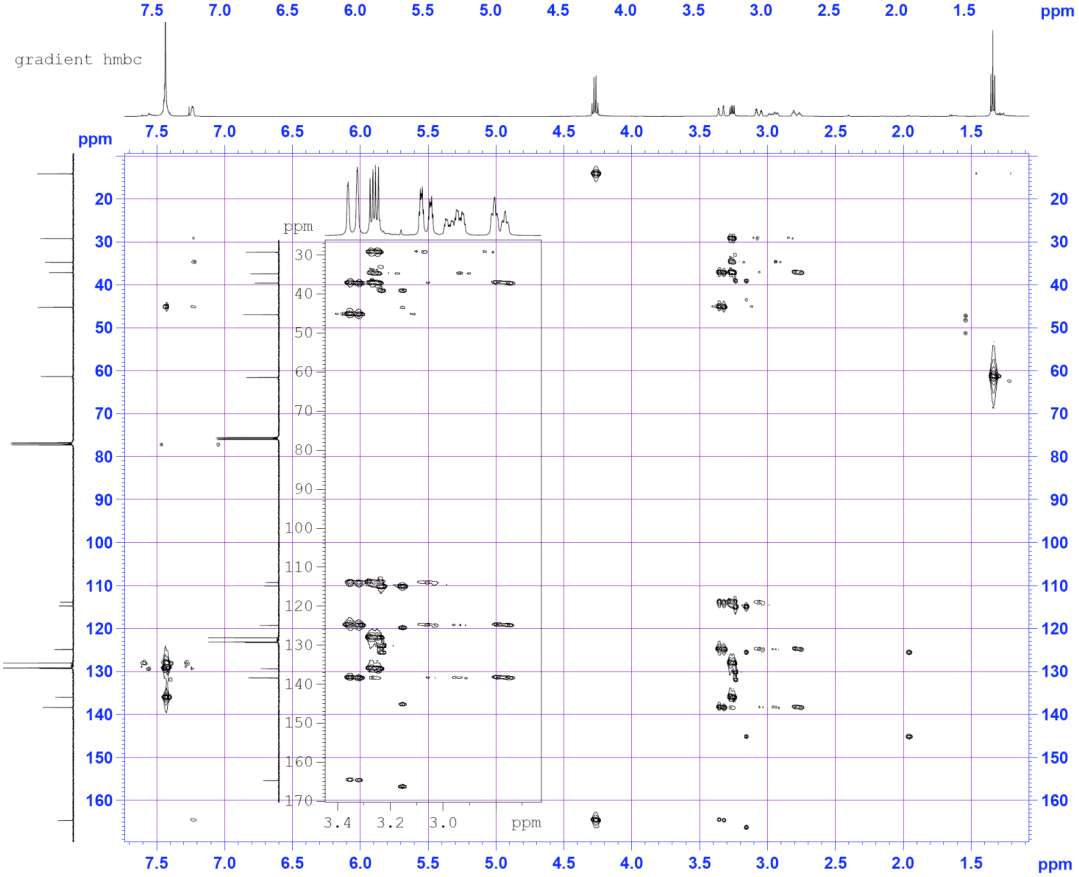
ester [4.3 (q) and 1.3 (t) ppm], and five aliphatic protons between 2 and 3 ppm for the five ring protons. ^{13}C NMR and ^{13}C DEPT experiments suggested 15 carbon atoms, including three methylene units (two in the cyclohexene ring and one for the ethyl ester). A 2D COSY experiment validated the cross-coupling of the ring protons for cyclohexene **3a**. Couplings were observed for the $C^2H–C^3H$, $C^3H–C^3H'$, $C^3H+C^3H'–C^4H$, and $C^6H–C^6H'$ units, consistent with the assigned structure **3a**. A 2D HMQC ($^1H–^{13}C$ correlation) experiment indicated that $C^3H–C^3H'$ coupling belonged to the C^3 atom and $C^6H–C^6H'$ coupling belonged to the C^6 atom of the cyclohexene **3a**.




138.34
129.23
129.09
127.99
61.25
45.09
34.67
29.14
77.09
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

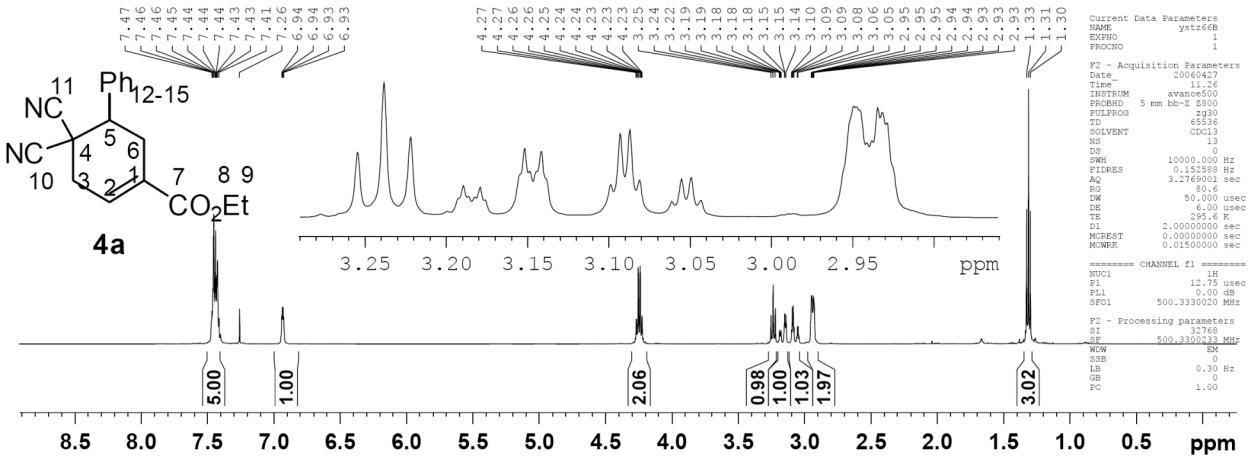

gradient selected double quantum filtered phase sensitive COSY
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
C⁶H-C⁴H
C⁶H C³H'
C³H
3.4 3.2 3.0 2.8 ppm

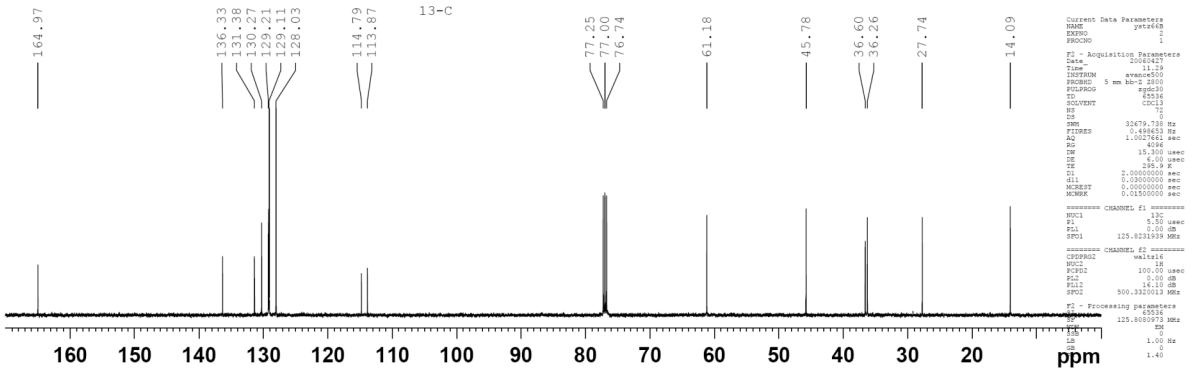

gradient hmqc
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
20 40 60 80 100 120 140 160 180
3.4 3.2 3.0

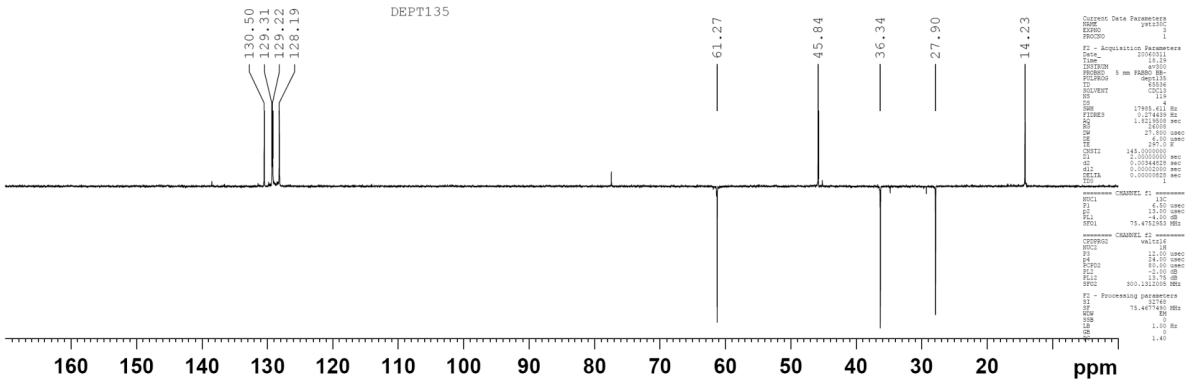

gradient hmbc
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
20 30 40 50 60 70 80 90 100 110 120 130 140 150 160
3.4 3.2 3.0

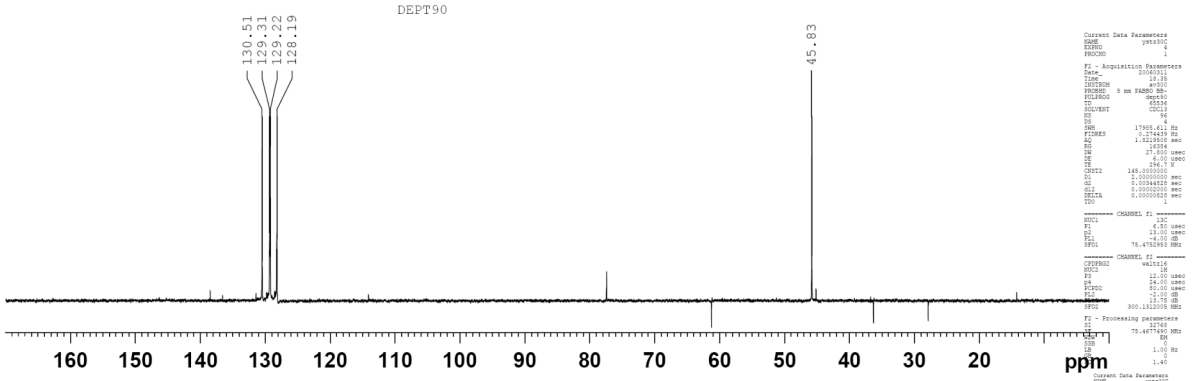
B. 1D and 2D NMR Spectra of Cyclohexene 4a

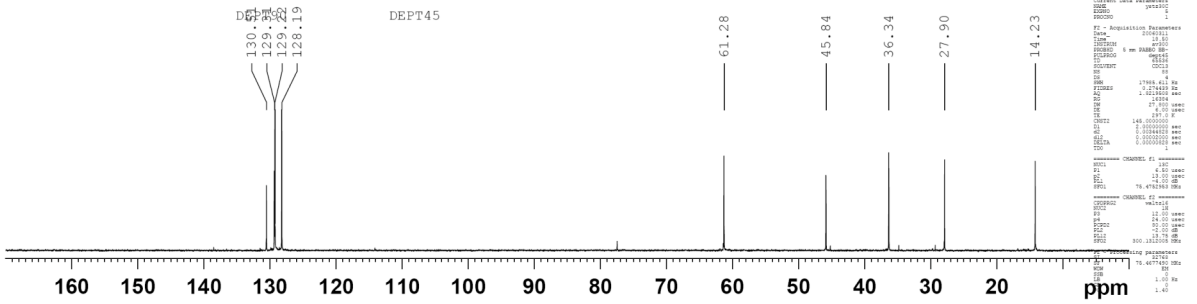
The structure of cyclohexene **4a** was also assigned based on MS (MALDI) and ^{1}H, ^{13}C, ^{13}C-DEPT, 2D-COSY, HMQC, and HMBC NMR spectroscopy experiments and further confirmed through single-crystal X-ray crystallographic analysis of cyclohexene **4f**. Similar to compound **3a**, the MALDI mass spectrum of cyclohexene **4a** gave a molecular ion at *m*/*z* 303.132 (M + Na), in accordance with the molecular formula $C_{17}H_{16}N_2O_2$ with sodium incorporation. The number of protons and carbons suggested by the ^{1}H NMR, ^{13}C NMR, and ^{13}C DEPT spectra of **4a** were similar to those of compound **3a**, although the ^{1}H NMR spectrum of **4a** exhibited (a) the C^{2}H vinylic proton at 6.93 ppm (vs. 7.23 ppm) for the α,β-unsaturated carboxylic ester and (b) very different coupling patterns of the five aliphatic protons (between 2 and 3 ppm) belonging to the cyclohexene ring. A 2D COSY experiment indicated the cross-coupling of the ring protons for cyclohexene **4a**. Couplings were observed for the C^{2}H–C^{3}H, C^{3}H–C^{3}H′, C^{5}H–C^{6}H+C^{6}H′, and C^{6}H–C^{6}H′ units. A 2D HMQC (^{1}H–^{13}C correlation) experiment indicated that the C^{3}H and C^{3}H′ protons belong to C^{3} and the C^{6}H and C^{6}H′ protons to C^{6}.

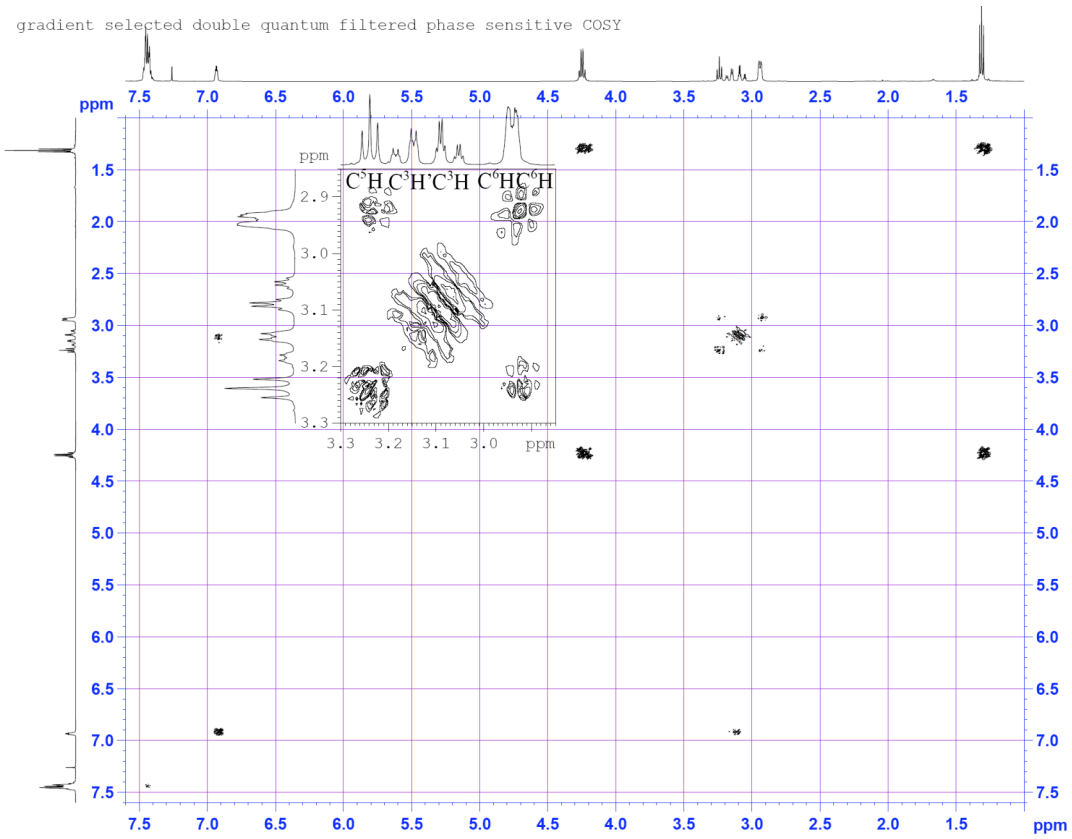


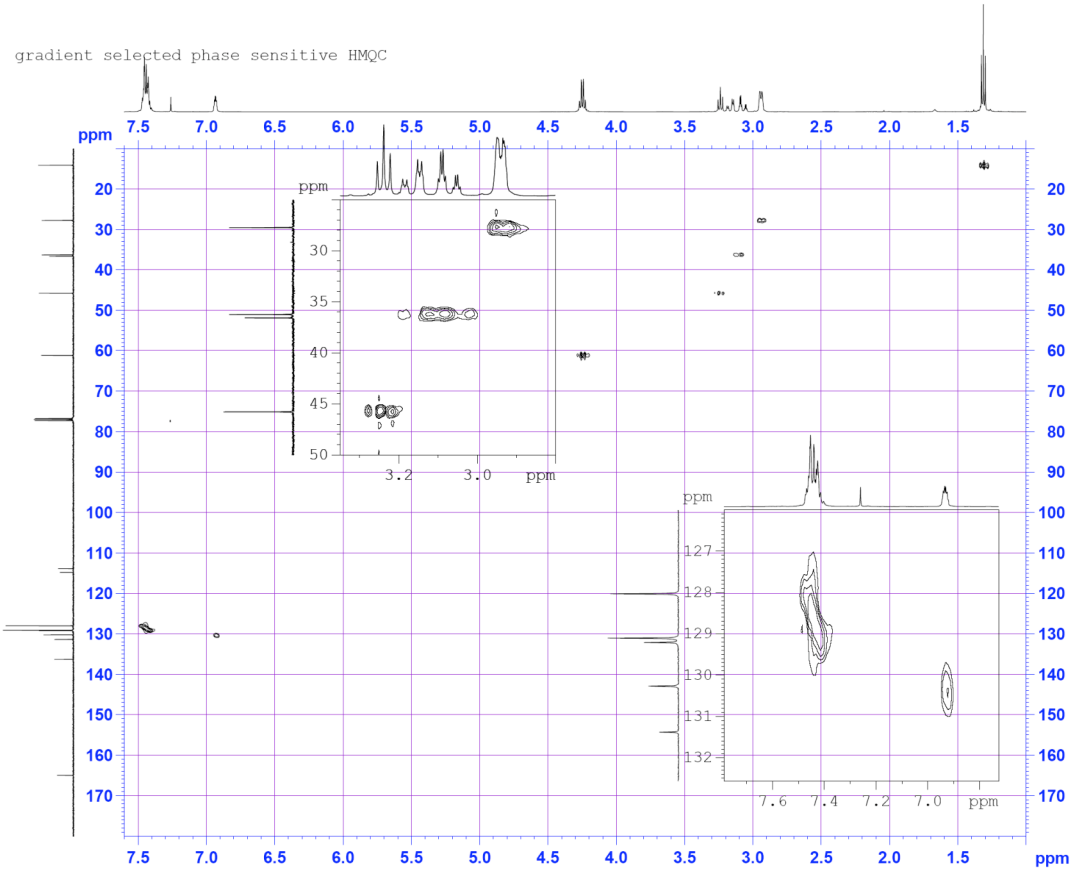

13-C
164.97
136.33
131.38
130.27
129.21
129.11
128.03
114.79
113.87
77.25
77.00
76.74
61.18
45.78
36.60
36.26
27.74
14.09
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

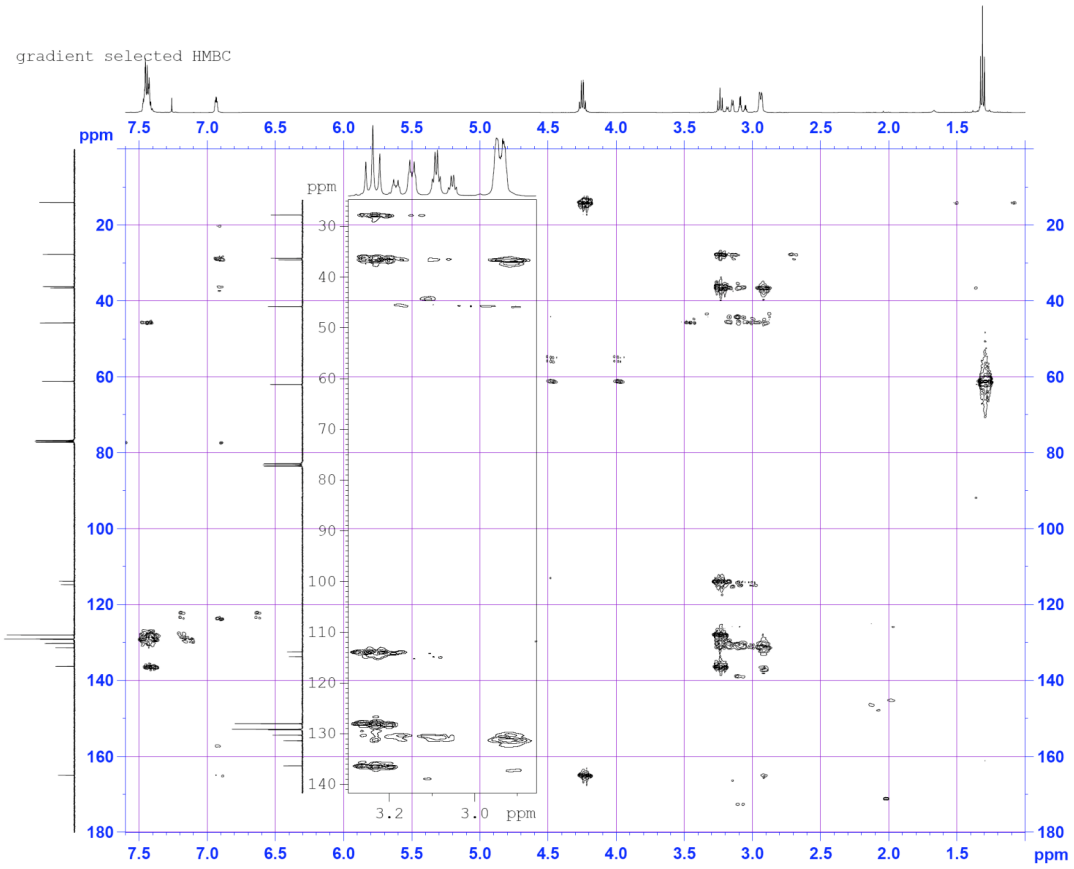

DEPT135
130.50
129.31
129.22
128.19
61.27
45.84
36.34
27.90
14.23
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


DEPT90
130.51
129.31
129.22
128.19
45.83
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

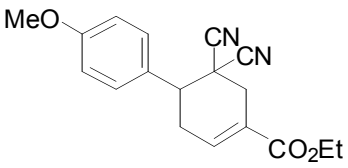

DEPT45
130.51
129.31
129.22
128.19
61.28
45.84
36.34
27.90
14.23
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


gradient selected double quantum filtered phase sensitive COSY
ppm
C5HC3H'C3H C6HC6H

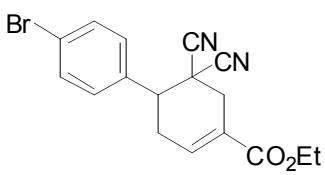

gradient selected phase sensitive HMQC
ppm



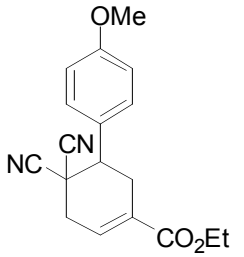
4. Characterization of Tetrasubstituted Cyclohexene Derivatives 3 and 4



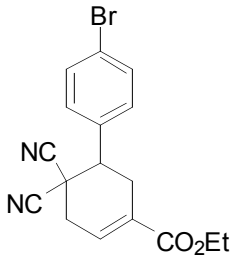
Ethyl 5,5-Dicyano-4-(4-methoxyphenyl)cyclohex-1-enecarboxylate (3b). 94% yield: IR (film) ν_{max} 2955, 2923, 2850, 2250, 2219, 1710, 1658, 1612, 1515, 1282, 1258, 1101, 1030 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, J = 8.8 Hz, 2H), 7.22 (m, 1H), 6.94 (d, J = 8.8 Hz, 2H), 4.26 (q, J = 7.1 Hz, 2H), 3.82 (s, 3H), 3.31 (d, J = 17.7 Hz, 1H), 3.22 (dd, J = 10.4, 5.4 Hz, 1H), 3.07 (dq, J = 18.9, 3.0 Hz, 1H), 2.92–2.97 (m, 2H), 1.31 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.0, 160.3, 136.3, 131.4, 130.3, 129.2, 129.1, 128.0, 114.8, 113.9, 61.2, 45.8, 36.6, 36.3, 27.7, 14.1; MS (MALDI) calcd for $C_{18}H_{18}N_2O_3Na$ [M + Na] 333.122, found 333.126.



Ethyl 4-(4-Bromophenyl)-5,5-dicyanocyclohex-1-enecarboxylate (3c). 86% yield: IR (film) ν_{max} 2980, 2918, 2850, 2250, 2224, 1711, 1660, 1263, 1235, 1103, 1075, 1012 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 8.5 Hz, 2H), 7.20 (m, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 3.34 (d, *J* = 17.7 Hz, 1H), 3.23 (dd, *J* = 10.2, 5.7 Hz, 1H), 3.04 (ddt, *J* = 17.7, 3.5, 2.3 Hz, 1H), 2.91 (ddm, *J* = 20.0, 10.2 Hz, 1H), 2.75 (dtm, *J* = 20.0, 5.0 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.4, 137.8, 135.0, 132.2, 129.6, 124.8, 123.3, 114.5, 113.5, 61.2, 44.4, 34.8, 34.4, 28.8, 14.0; MS (EI) calcd for $C_{17}H_{15}N_2O_2Br$ $[M]^+$ 358.0, found 358.0.

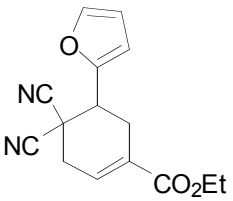


Ethyl 4,4-Dicyano-5-(4-methoxyphenyl)cyclohex-1-enecarboxylate (4b). 90% yield; IR (film) ν_{max} 3061, 2964, 2841, 2251, 1713, 1660, 1611, 1516, 1257, 1101, 1031, 833, 801, 737 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.92 (m, 1H), 4.25 (qd, *J* = 7.1, 1.3 Hz, 2H), 3.83 (s, 3H), 3.20 (t, *J* = 8.1 Hz, 1H), 3.15 (ddt, *J* = 18.9, 5.1, 1.6 Hz, 1H), 3.0 (dq, *J* = 18.8, 3.0 Hz, 1H), 2.90 (ddt, *J* = 8.1, 2.9, 1.4 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.1, 160.2, 131.5, 130.2, 129.2, 128.3, 115.0, 114.5, 114.0, 61.2, 55.3, 45.2, 37.0, 36.2, 27.8, 14.1; MS (MALDI) calcd for $C_{18}H_{18}N_2O_3Na$ $[M + Na]^+$ 333.122, found 333.124.

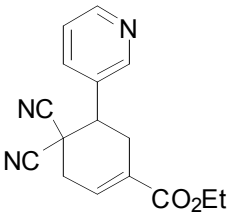


Ethyl 5-(4-Bromophenyl)-4,4-dicyanocyclohex-1-enecarboxylate (4c). 85% yield: IR (film) ν_{max} 3062, 2983, 2933, 2251, 1713, 1660, 1591, 1491, 1262, 1238, 1103, 1075, 1010, 826, 735 cm^{-1}; ^1H NMR (300z, $CDCl_3$) δ

7.58 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 6.93 (m, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 3.21 (dd, *J* = 9.8, 6.4 Hz, 1H), 3.14 (dt, *J* = 5.0, 1.63 Hz, 1H), 3.06 (dq, *J* = 18.9, 2.9 Hz, 1H), 2.85–2.95 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.8, 135.3, 132.3, 131.1, 130.2, 129.7, 123.4, 114.6, 113.6, 61.2, 45.2, 36.4, 36.1, 27.5, 14.1; MS (EI) calcd for $C_{17}H_{15}N_2O_2Br$ $[M]^+$ 358.0, found 358.0.

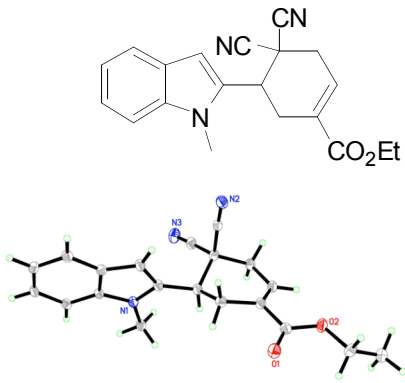


Ethyl 4,4-Dicyano-5-(furan-2-yl)cyclohex-1-enecarboxylate (4d). 88% yield: IR (film) ν_{max} 3058, 2984, 2934, 2250, 2222, 1713, 1656, 1591, 1491, 1447, 1385, 1264, 1102, 1076, 1015, 826, 737 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (dd, *J* = 1.8, 1.1 Hz, 1H), 6.90 (m, 1H), 6.48 (d, *J* = 3.3 Hz, 1H), 6.42 (dd, *J* = 3.3, 1.7 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 3.50 (dd, *J* = 10.2, 5.5 Hz, 1H), 3.12 (ddt, *J* = 18.9, 4.9, 1.4 Hz, 1H), 2.93–3.07 (m, 2H), 2.88 (dddt, *J* = 18.9, 10.2, 1.9 Hz, 1H), 1.32 (t, *J* = 7.13 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 164.9, 149.7, 143.4, 130.6, 130.2, 114.7, 113.5, 110.7, 109.1, 61.3, 39.6, 35.33, 35.31, 26.3, 14.1; MS (MALDI) calcd for $C_{15}H_{14}N_2O_3Na$ $[M + Na]^+$ 293.0902, found 293.0900.



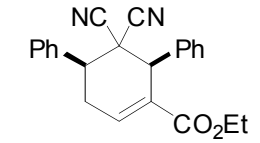
Ethyl 4,4-Dicyano-5-(pyridin-3-yl)cyclohex-1-enecarboxylate (4e). 80% yield; IR (film) ν_{max} 3036, 2983, 2915, 2251, 1712, 1660, 1592, 1429, 1259, 1104, 1027, 737, 714 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (s, 2H), 7.84 (dt, *J* = 8.0, 1.8 Hz, 1H), 7.14 (td, *J* = 8.7, 2.1 Hz, 1H), 6.90–6.96 (m, 1H), 4.23 (qd, *J* = 7.1, 0.8 Hz, 2H), 3.28 (dd, *J* = 9.6, 6.8 Hz, 1H), 3.17 (ddt, *J* = 18.9, 5.1, 1.4 Hz, 1H), 3.08 (dq, *J* = 18.9, 2.9 Hz, 1H), 2.88–2.95 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125

MHz, $CDCl_3$) δ 164.7, 150.6, 150.0, 135.0, 132.1, 131.0, 130.2, 123.8, 114.4, 113.4, 61.3, 43.5, 36.3, 36.0, 27.5, 14.0; MS (MALDI) calcd for $C_{16}H_{16}N_3O_2$ $[M + H]^+$ 282.124, found 282.129.



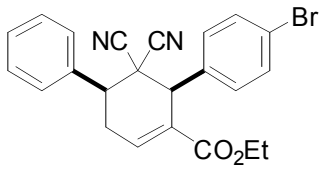
Ethyl 4,4-Dicyano-5-(1-methyl-1H-indol-2-yl)cyclohex-1-enecarboxylate (4f). 91% yield as a bright-orange solid: m.p. 203–204 °C; IR (film) ν_{max} 3058, 2980, 2934, 2252, 1714, 1661, 1469, 1277, 1256, 1102, 745, 730 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.29 (dd, *J* = 8.2, 7.4 Hz, 1H), 7.17 (dd, *J* = 7.9, 7.4 Hz, 1H), 6.96 (br s, 1H), 6.80 (s, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 3.63 (dd, *J* = 10.2, 5.4 Hz, 1H), 3.22 (br dd, *J* = 18.8, 4.4 Hz, 1H), 2.90–3.15 (m, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.8, 137.5, 135.0, 131.2, 130.3, 127.2, 122.5, 120.9, 120.2, 114.9, 113.7, 109.6, 101.3, 61.3, 37.2, 36.3, 35.8, 30.1, 28.9, 14.1; MS (EI) calcd for $C_{20}H_{19}N_3O_2$ $[M]^+$ 333.1, found 333.1.

5. Characterization of Pentasubstituted Cyclohexene Derivatives 3

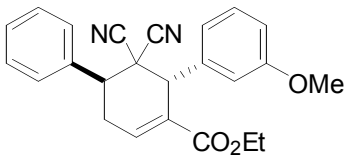


***cis*-Ethyl 5,5-Dicyano-4,6-diphenylcyclohex-1-enecarboxylate (3d).** 93% yield as a 82:18 mixture of cis and trans isomers: IR (film) ν_{max} 3032, 2960, 2924, 2853, 2247, 2215, 1719, 1656, 1603, 1495, 1455, 1371, 1257, 1103, 1021, 800, 733, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.35–7.52 (m, 10H), 7.22 (ddd, *J* = 6.1, 2.2, 2.0 Hz, 1H), 4.5 (dt, *J* = 4.1, 2.0 Hz, 1H), 4.00 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.90 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.45 (dd, *J* = 12.2, 4.4 Hz, 1H), 3.18 (dddd, *J* = 19.5, 12.2, 4.1, 2.2 Hz, 1H), 2.74 (dddd, *J* = 19.5, 6.2, 4.4, 2.0 Hz, 1H), 0.89 (t, *J* = 7.1

Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.4, 138.3, 136.0, 135.7, 130.5, 129.4, 129.2, 128.9, 128.82, 128.83, 128.3, 114.2, 112.5, 60.8, 51.6, 47.2, 47.1, 28.8, 13.6; MS (MALDI) calcd for $C_{23}H_{20}N_2O_2Na$ $[M + Na]^+$ 379.142, found 379.145.

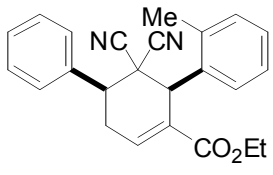


***cis*-Ethyl 6-(4-Bromophenyl)-5,5-dicyano-4-phenylcyclohex-1-enecarboxylate (3e).** 95% yield as a white solid, an 89:11 mixture of cis and trans isomers: m.p. 160–161 ºC (cis isomer): IR (film) ν_{max} 3033, 2982, 2253, 2217, 1716, 1655, 1489, 1267, 1253, 1012, 909, 731, 701 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27–7.57 (m, 9H), 7.25 (ddd, *J* = 6.2, 2.1, 2.1 Hz, 1H), 4.45–4.52 (m, 1H), 4.92–4.15 (m, 2H), 3.44 (dd, *J* = 12.2, 4.4Hz, 1H), 3.17 (dddd, *J* = 19.6, 12.2, 4.1, 2.1 Hz, 1H), 2.75 (dddd, *J* = 19.6, 6.2, 4.5, 1.8 Hz, 1H), 0.98 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.0, 139.0, 135.7, 134.9, 132.0, 129.8, 129.4, 129.2, 129.1, 128.2, 123.0, 114.0, 112.3, 61.0, 50.9, 47.0, 46.9, 28.7, 13.7; MS (EI) calcd for $C_{23}H_{19}N_2O_2Br$ $[M]^+$ 434, found 434.

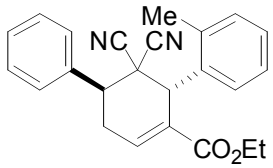


***trans*-Ethyl 5,5-Dicyano-6-(3-methoxyphenyl)-4-phenylcyclohex-1-enecarboxylate (3f).** 92% yield as a 78:22 mixture of cis and trans isomers: IR (film) ν_{max} 3061, 3034, 2981, 2934, 2840, 2247, 2226, 1713, 1658, 1602, 1491, 1457, 1273, 1248, 1158, 1100, 103 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (dd, *J* = 4.5, 2.8 Hz, 1H), 7.30–7.40 (m, 6H), 6.92–6.97 (m, 2H), 6.89–6.92 (m, 1H), 4.68 (s, 1H), 4.05–4.20 (m, 2H), 3.84 (s, 3H), 3.37 (dd, *J* = 11.4, 5.6 Hz, 1H), 3.05 (ddt, *J* = 20.5, 11.5, 2.1 Hz, 1H), 2.88 (dt, *J* = 20.5, 5.2 Hz, 1H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.3, 159.7, 139.1, 136.0, 135.9, 129.7, 129.2, 129.0,

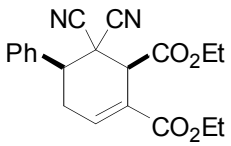
128.4, 127.8, 122.2, 116.3, 114.6, 114.2, 113.3, 61.2, 55.3, 48.6, 43.6, 40.2, 29.8, 13.9; MS (MALDI) calcd for $C_{24}H_{22}N_2O_3$ $[M]^+$ 409.1528, found 409.1525.



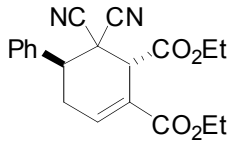
***cis*-Ethyl 5,5-Dicyano-6-(2-methoxyphenyl)-4-phenylcyclohex-1-enecarboxylate (3g).** 91% yield as a 64:36 mixture of cis and trans isomers: m.p. 179–180 °C; IR (film) ν_{max} 3065, 3030, 2981, 2931, 2250, 1718, 1655, 1493, 1456, 1370, 1283, 1253, 1216, 1112, 1051, 1020, 7612, 742, 724, 698 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.18–7.58 (m, 9H), 7.14 (ddd, *J* = 6.1, 2.3, 2.3 Hz, 1H), 4.90 (ddd, *J* = 4.1, 2.0, 2.0 Hz, 1H), 3.82–3.98 (m, 2H), 3.51 (dd, *J* = 12.2, 4.4 Hz, 1H), 3.22 (dddd, *J* = 19.5, 12.3, 4.2, 2.3 Hz, 1H), 2.74 (dddd, *J* = 19.5, 6.2, 4.4, 1.8 Hz, 1H), 2.62 (s, 3H), 0.88 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.3, 137.3, 137.2, 135.9, 134.2, 131.8, 131.1, 128.4, 129.1, 128.6, 128.4, 127.3, 126.4, 114.4, 112.9, 40.8, 47.7, 46.0, 45.9, 28.9, 19.9, 13.5; MS (EI) calcd for $C_{24}H_{22}N_2O_3$ $[M]^+$ 370, found 370.



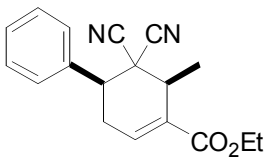
***trans*-Ethyl 5,5-Dicyano-6-(2-methoxyphenyl)-4-phenylcyclohex-1-enecarboxylate (3g).** IR (film) ν_{max} 3066, 3025, 2983, 2935, 2247, 1714, 1660, 1489, 1456, 1420, 1276, 1249, 1224, 1101, 1084, 1027, 757, 731, 667 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 7.4, 2.7 Hz, 1H), 7.06–7.4 (m, 9H), 5.05 (s, 1H), 4.02–4.15 (m, 2H), 3.53 (dd, *J* = 11.7, 5.6 Hz, 1H), 3.08 (dddd, *J* = 20.4, 11.7, 2.3, 2.3 Hz, 1H), 2.91 (ddd, *J* = 20.4, 5.3, 5.3 Hz, 1H), 2.65 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.3, 138.6, 138.4, 135.8, 133.6, 131.6, 129.2, 129.01, 129.00, 128.9, 128.5, 128.5, 126.0, 114.8, 113.0, 61.2, 44.1, 42.6, 40.0, 29.3, 20.3, 13.8; MS (EI) calcd for $C_{24}H_{22}N_2O_3$ $[M]^+$ 370, found 370.



***cis*-Diethyl 6,6-Dicyano-5-phenylcyclohex-2-ene-1,2-dicarboxylate (3h)**. 96% yield as a 66:33 mixture of cis and trans isomers: IR (film) ν_{max} 3065, 3036, 2984, 2919, 2249, 1739, 1712, 1660, 1451, 1373, 1258, 1187, 1102, 1031, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40–7.52 (m, 5H), 7.25 (ddd, *J* = 6.1, 2.1, 2.0 Hz, 1H), 4.19–4.4 (m, 4H), 4.17 (dt, *J* = 3.6, 1.7 Hz, 1H), 3.27 (dd, *J* = 11.9, 4.5 Hz, 1H), 3.07 (dddd, *J* = 19.7, 12.0, 3.6, 2.2 Hz, 1H), 2.70 (ddd, *J* = 19.7, 5.3, 1.6 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.3, 164.5, 139.2, 135.0, 129.5, 129.1, 128.4, 128.5, 113.2, 111.6, 62.6, 61.4, 50.9, 46.4, 41.1, 28.6, 14.0, 13.8; MS (MALDI) calcd for $C_{20}H_{20}N_2O_2Na$ $[M + Na]^+$ 375.132, found 375.136.

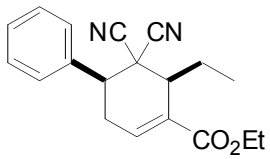


***trans*-Diethyl 6,6-Dicyano-5-phenylcyclohex-2-ene-1,2-dicarboxylate (3h)**. IR (film) ν_{max} 2984, 2934, 2252, 17412, 1712, 1423, 1303, 1259, 1187, 1127 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.37–7.50 (m, 5H), 7.35 (dd, *J* = 4.7, 2.4 Hz, 1H), 4.15–4.4 (m, 5H), 3.80 (dd, *J* = 11.2, 5.3 Hz, 1H), 2.94 (ddm, *J* = 20.6, 11.5 Hz, 1H), 2.8 (dt, *J* = 20.4, 5.2 Hz, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.9, 164.0, 141.0, 135.5, 129.2, 129.0, 128.2, 124.2, 112.8, 112.4, 62.7, 61.4, 47.9, 41.0, 39.7, 29.1, 13.8, 13.8; MS (MALDI) calcd for $C_{20}H_{20}N_2O_2Na$ $[M + Na]^+$ 375.132, found 375.137.

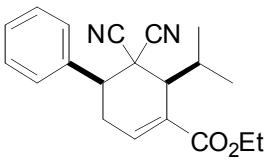


***cis*-Ethyl 5,5-Dicyano-6-methyl-4-phenylcyclohex-1-enecarboxylate (3i)**. 95% yield as an 80:20 mixture of cis and trans isomers: m.p. 92–93 °C; IR (film) ν_{max} 3062, 3033, 2976, 2935, 2229, 1728, 1640, 1583, 1453, 1372, 1247, 1185, 1105, 1025, 766, 738, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40–7.50 (m,

5H), 7.01 (ddd, *J* = 6.0, 2.1, 2.1 Hz, 1H), 4.20–4.30 (m, 2H), 3.32–3.40 (m, 1H), 3.22 (dd, *J* = 12.1, 4.6 Hz, 1H), 2.99 (dddd, *J* = 19.5, 12.2, 3.8, 2.3 Hz, 1H), 2.62 (dddd, *J* = 19.5, 6.1, 4.5, 1.7 Hz, 1H), 1.62 (d, *J* = 7.0 Hz, 3H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.4, 136.8, 136.3, 131.0, 129.2, 129.1, 128.2, 114.9, 112.6, 61.1, 46.3, 46.0, 40.5, 28.8, 16.2, 14.1; MS (EI) calcd for $C_{18}H_{18}N_2O_2$ $[M]^+$ 294 found 294.

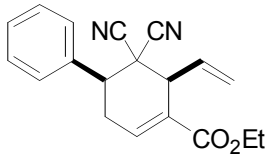


***cis*-Ethyl 5,5-Dicyano-6-ethyl-4-phenylcyclohex-1-enecarboxylate (3j).** 98% yield as a 92:8 mixture of cis and trans isomers: IR (film) ν_{max} 2973, 2936, 2877, 2248, 2228, 1715, 1456, 1374, 1246, 766, 735, 701 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40–7.50 (m, 5H), 7.00 (ddd, *J* = 6.4, 2.0, 2.0 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 3.26–3.34 (m, 1H), 3.20 (dd, *J* = 12.0, 4.2 Hz, 1H), 3.00 (dddd, *J* = 19.0, 12.0, 3.6, 2.2 Hz, 1H), 2.59 (dddd, *J* = 19.0, 6.3, 4.3, 1.8 Hz, 1H), 2.12 (dqd, *J* = 15.0, 7.6, 3.1 Hz, 1H), 1.91 (dqd, *J* = 15.0, 7.6, 1.3 Hz, 1H), 1.35 (t, *J* = 7.1 Hz, 3H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 165.9, 136.7, 136.0, 131.5, 129.3, 128.0, 128.5, 115.5, 113, 61.2, 46.8, 46.2, 44.0, 28.3, 25.3, 14.1, 12.3; MS (EI) calcd for $C_{19}H_{20}N_2O_2$ $[M]^+$ 308, found 308.

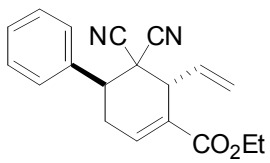


***cis*-Ethyl 5,5-Dicyano-6-isopropyl-4-phenylcyclohex-1-enecarboxylate (3k)**. 77% yield as a 34:66 mixture of cis and trans isomers: IR (film) ν_{max} 3035, 2973, 2935, 2248, 1714, 1641, 1459, 1370, 1245, 1097, 1048, 738, 700 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.4–7.5 (m, 5H), 6.97 (ddd, *J* = 6.5, 2.0, 2.0 Hz, 1H), 4.2–4.35 (m, 2H), 3.38–3.44 (m, 1H), 3.20 (dd, *J* = 11.8, 4.2 Hz, 1H), 3.02 (dddd, *J* = 19.0, 11.9, 3.7, 2.3 Hz, 1H), 2.58 (dddd, *J* = 19.0, 6.4, 4.4, 1.8 Hz, 1H), 2.46 (dqd, *J* = 7.1, 7.1, 2.7 Hz, 1H), 1.35 (t, *J* = 7.1 Hz, 3H), 1.30 (d, *J* = 7.1 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz,

$CDCl_3$) δ 166.9, 136.8, 136.0, 131.3, 129.2, 129.0, 128.5, 115.6, 113.8, 61.2, 50.2, 46.8, 44.6, 31.4, 28.1, 22.3, 18.8, 14.1; MS (MALDI) calcd for $C_{20}H_{22}N_2O_2Na$ $[M + Na]^+$ 345.1579, found 345.1589.

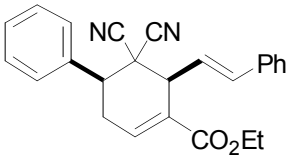


***cis*-Ethyl 5,5-Dicyano-4-phenyl-6-vinylcyclohex-1-enecarboxylate (3l)**. 94% yield as a 91:9 mixture of cis and trans isomers: IR (film) ν_{max} 3064, 3035, 2984, 2933, 2907, 2250, 1717, 1653, 1496, 1455, 1422, 1371, 1256, 1109, 1050, 733, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.38–7.5 (m, 5H), 7.10 (ddd, *J* = 6.0, 2.2, 2.2 Hz, 1H), 5.86 (ddd, *J* = 17.0, 8.9, 10.1 Hz, 1H), 5.58 (d, *J* = 17.0 Hz, 1H), 5.52 (d, *J* = 10.1 Hz, 1H), 4.16–4.28 (m, 2H), 3.82–3.90 (m, 1H), 3.27 (dd, *J* = 12.0, 4.6 Hz, 1H), 3.03 (dddd, *J* = 19.6, 12.0, 3.9, 2.2 Hz, 1H), 2.67 (dddd, *J* = 19.6, 6.1, 4.5, 1.7 Hz, 1H), 1.29 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.1, 137.9, 136.0, 132.2, 129.3, 129.1, 129.0, 128.2, 122.8, 114.3, 112.4, 61.1, 49.2, 45.9, 45.7, 28.8, 14.0; MS (MALDI) calcd for $C_{19}H_{18}N_2O_2Na$ $[M + Na]^+$ 329.127, found 329.106.

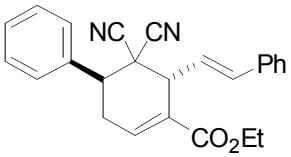


***trans*-Ethyl 5,5-Dicyano-4-phenyl-6-vinylcyclohex-1-enecarboxylate (3l)**. IR (film) ν_{max} 3064, 3034, 2983, 2920, 2851, 2249, 1714, 1656, 1452, 1417, 1374, 1274, 1248, 1101, 1069, 1027, 761, 700 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.38–7.48 (m, 5H), 7.32 (dd, *J* = 4.7, 2.8 Hz, 1H), 6.10 (ddd, *J* = 17.0, 10.3, 6.5 Hz, 1H), 5.60 (d, *J* = 10.3 Hz, 1H), 5.33 (d, *J* = 17.0 Hz, 1H), 4.20–4.31 (m 2H), 4.19 (d, *J* = 6.7 Hz, 1H), 3.26 (dd, *J* = 11.6, 5.6 Hz, 1H), 2.95 (dddd, *J* = 20.4, 11.6, 2.6, 1.7 Hz, 1H), 2.74 (ddd, *J* = 20.4, 5.2, 5.2 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$)

δ 164.5, 139.4, 136.0, 132.4, 129.3, 129.2, 128.3, 127.1, 123.6, 114.2, 113.3, 61.4, 45.6, 41.9, 40.7, 29.9, 14.1; MS (MALDI) calcd for $C_{19}H_{19}N_2O_2$ $[M + H]^+$ 307.145 found 307.145.

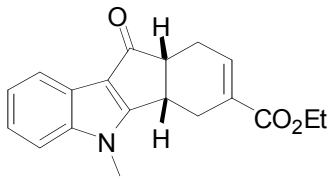


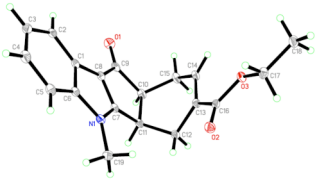
***cis*-Ethyl 5,5-Dicyano-4-phenyl-6-styrylcyclohex-1-enecarboxylate (3m)**. 93% yield as a 91:9 mixture of cis and trans isomers: IR (film) ν_{max} 3061, 3032, 2982, 2926, 2249, 1716, 1650, 1451, 1371, 1255, 1108, 1052, 1022, 967, 737, 697 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.27–7.55 (m, 10H), 7.13–7.20 (m, 1H), 6.90 (d, *J* = 15.7 Hz, 1H), 6.19 (dd, *J* = 15.6, 9.2 Hz, 1H), 4.10–4.30 (m, 2H), 4.06 (d, *J* = 8.6 Hz, 1H), 3.35 (dd, *J* = 11.8, 4.4 Hz, 1H), 3.0–3.2 (m, 1H), 2.7 (ddd, *J* = 19.5, 4.3, 4.3 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.1, 137.8, 137.4, 135.9, 135.7, 129.2, 129.1, 129.0, 128.5, 128.2, 128.1, 126.7, 122.8, 114.3, 112.4, 60.9, 48.6, 45.8, 45.7, 28.7, 14.0; MS (EI) calcd for $C_{25}H_{22}N_2O_2$ $[M]^+$ 382.2, found 382.2.



***trans*-Ethyl 5,5-Dicyano-4-phenyl-6-styrylcyclohex-1-enecarboxylate (3m)**. IR (film) ν_{max} 3060, 3031, 2981, 2925, 2853, 2247, 1713, 1653, 1494, 1450, 1374, 1276, 1248, 1097, 1026, 969, 757, 699 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.30–7.50 (m, 11H), 6.60 (d, *J* = 15.8 Hz, 1H), 6.39 (dd, *J* = 15.8, 6.9 Hz, 1H), 4.36 (d, *J* = 6.8 Hz, 1H), 4.17–4.30 (m, 2H), 3.33 (dd, *J* = 11.7, 5.6 Hz, 1H), 2.99 (dddd, *J* = 20.5, 11.6, 2.5, 1.5 Hz, 1H), 2.80 (dt, *J* = 20.5, 5.2 Hz, 1H), 1.30 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.5, 139.4, 138.0, 136.0, 135.4, 129.3, 129.2, 128.74, 128.72, 128.3, 127.4, 126.9, 122.9, 114.2, 113.4, 61.4, 45.3, 42.4, 40.8, 29.9, 14.1; MS (EI) calcd for $C_{25}H_{22}N_2O_2$ $[M]^+$ 382.2, found 382.2.

6. Synthesis of Tetracycle 12

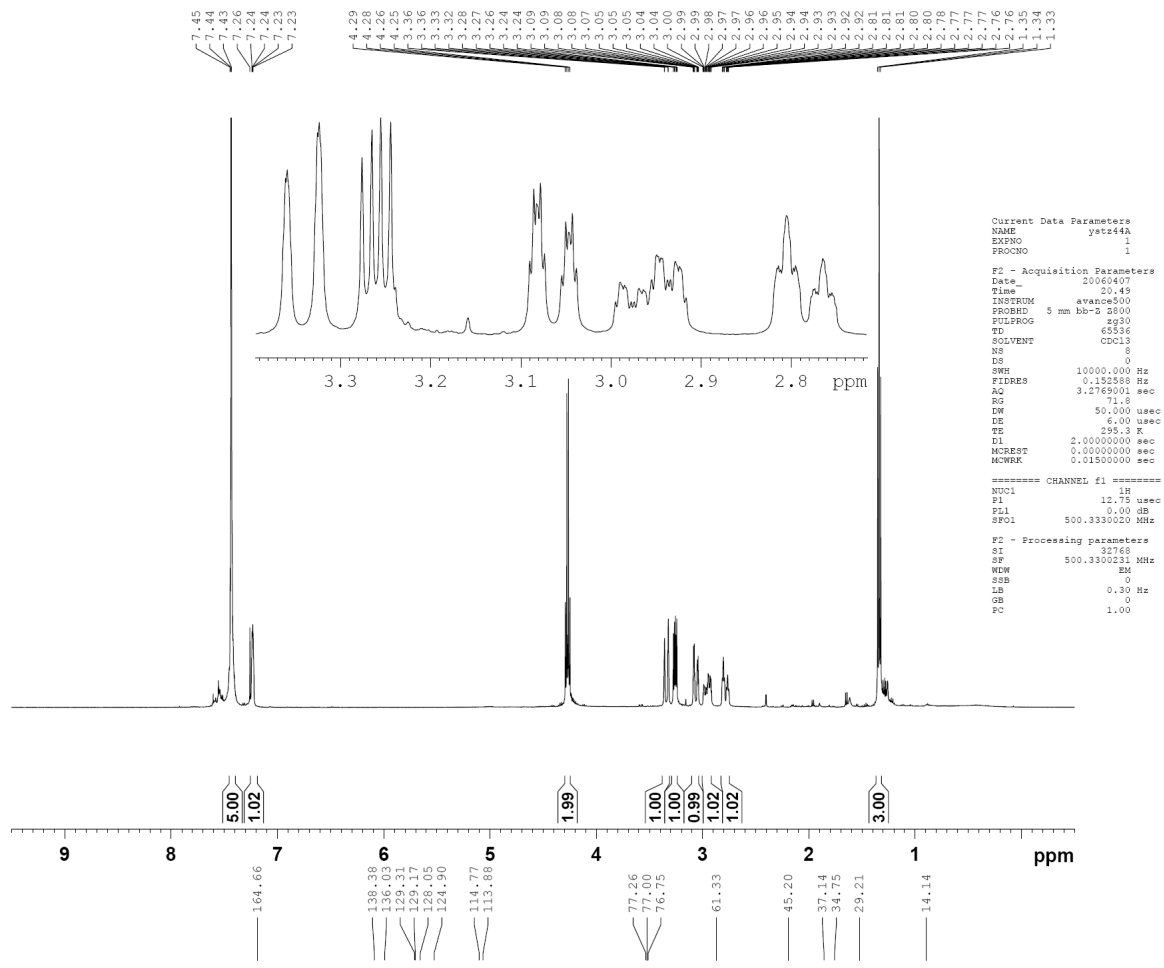


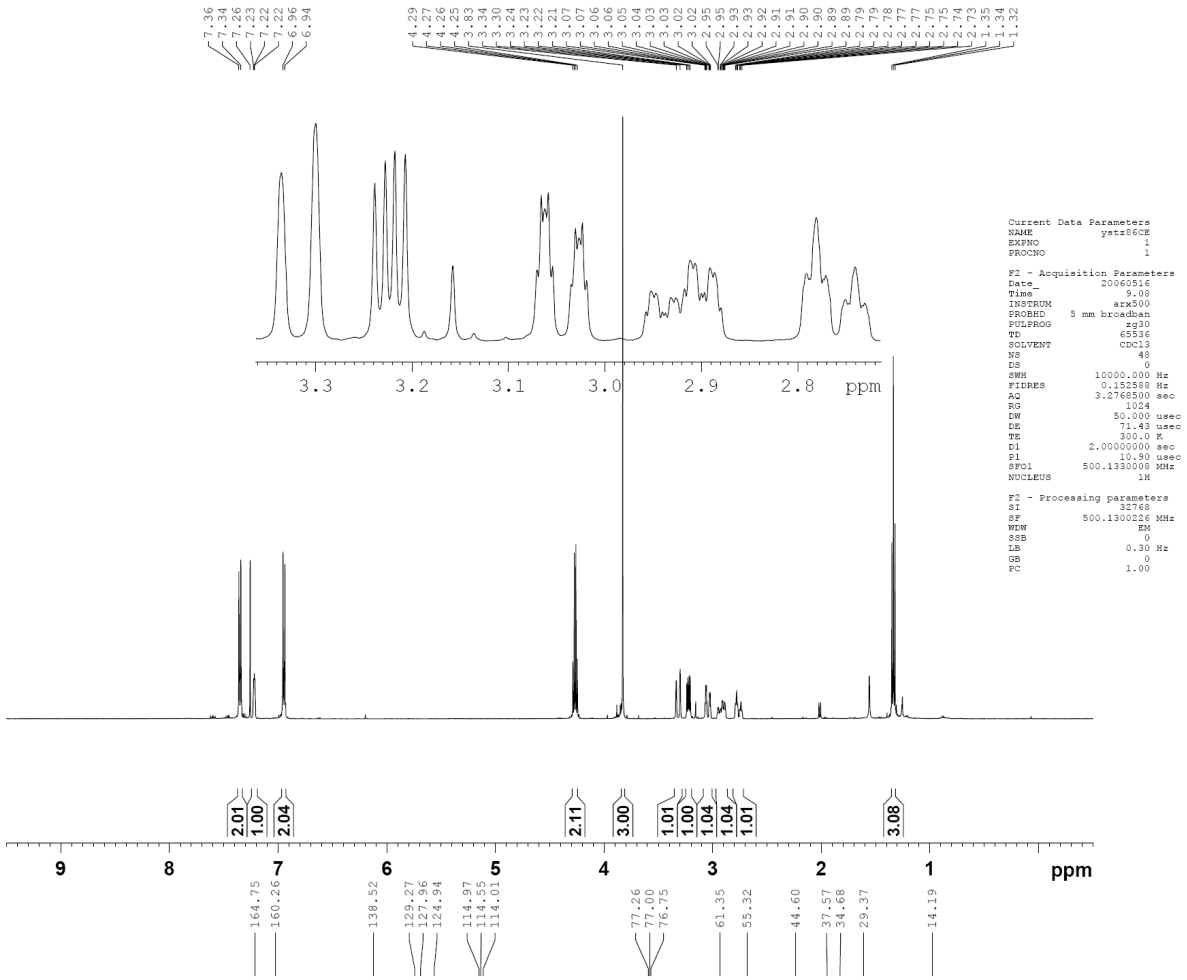


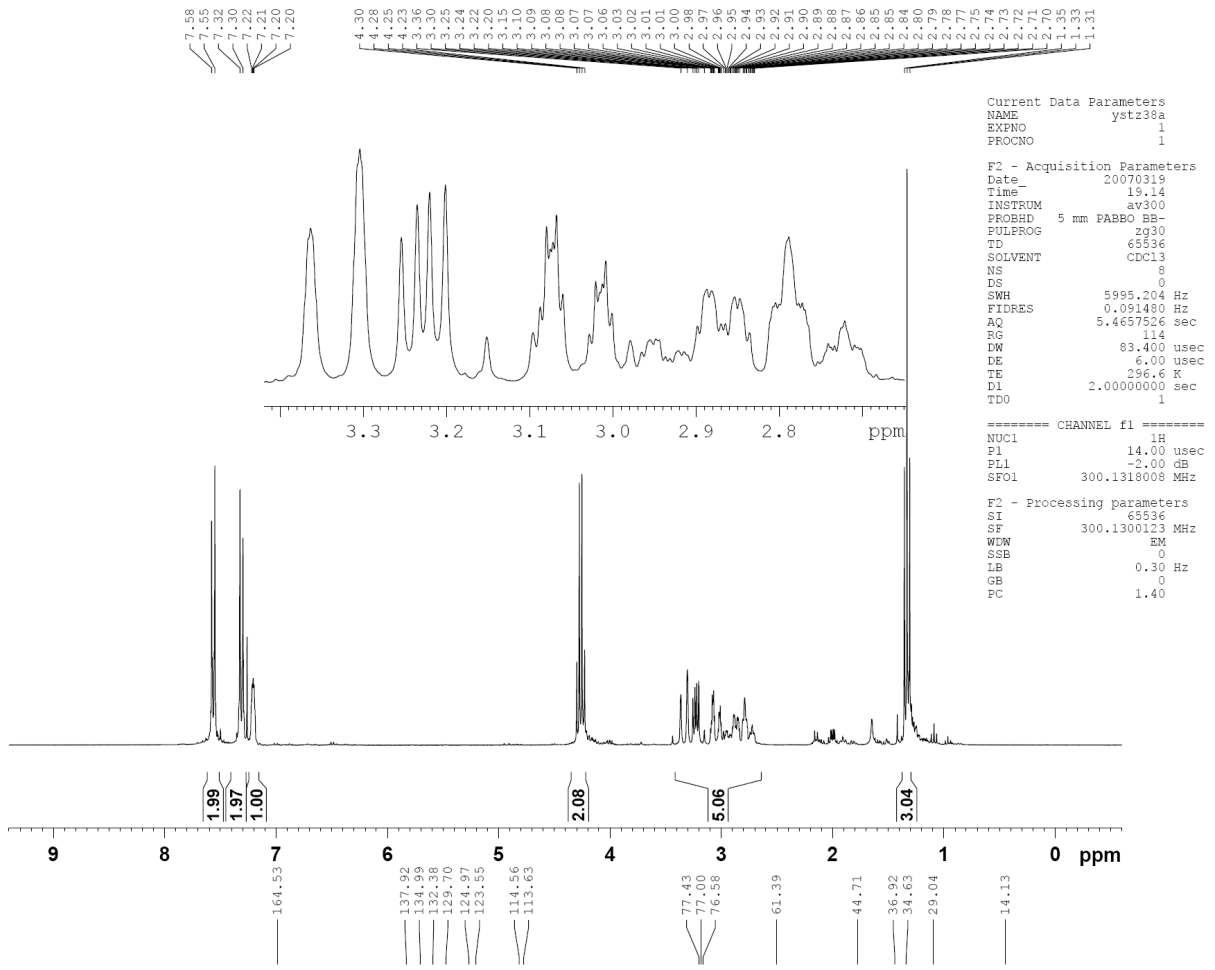
Aqueous hydrochloric acid (37%, 20 mL) was added over 2 min to a solution of the cyclohexene **4f** (1.00 mmol) in ethyl acetate (2 mL) at 0 °C, followed by the slow addition of sulfuric acid (1 mL). The solution was warmed slowly to room temperature and then heated under reflux. After the reaction had reached completion (ca. 2 h), the resulting mixture was cooled to 0 °C and diluted with water (10 mL). Aqueous sodium hydroxide (2 N) was added slowly until the pH of the solution reached 5. The combined mixture was extracted with ethyl acetate (2 × 100 mL). The combined organic phases were washed sequentially with water (20 mL) and brine (20 mL) and then dried (Na_2SO_4). After evaporation of the solvents, the crude product was identified as the hydrolysis/monodecarboxylation product of the malononitrile, i.e., the corresponding monocarboxylic acid. The crude mixture was dissolved in ethanol (40 mL) and then sulfuric acid (1 mL) was added slowly at 0 °C. The solution was heated under reflux until the reaction reached completion. After cooling the mixture to ambient temperature, the solvent was evaporated. The crude product was dissolved in ethyl acetate (30 mL), washed sequentially with water (10 mL), aqueous sodium hydroxide (10 mL, 0.2 N), saturated aqueous sodium bicarbonate (2 × 20 mL), and brine (10 mL), and then dried (Na_2SO_4). After evaporation of the solvents, the crude product was purified through flash column chromatography (silica gel, 70% ethyl acetate in hexane) to afford the fused tetracycle **12** (262 mg, 85%). IR (film) ν_{max} 2924, 2854, 1704, 1682, 1532, 1480, 1453, 1272, 1249, 1096 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, J = 7.6 Hz, 1H), 7.24–7.35 (m, 3H), 7.16 (ddd, J = 6.2, 4.8, 1.4 Hz, 1H), 4.02–4.18 (m, 2H), 3.86 (s, 3H), 3.75 (td, J = 6.9, 4.9 Hz, 1H), 3.28 (td, J = 7.4, 4.7 Hz, 1H), 2.87 (dd, J = 15.2, 4.9

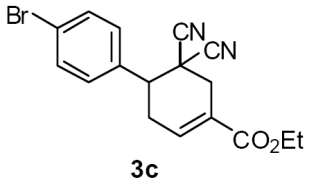
Hz, 1H), 2.69–2.82 (m, 2H), 2.51 (dddd, J = 15.9, 7.8, 4.7, 1.5 Hz, 1H), 1.23 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.7, 168.0, 165.9, 143.4, 140.4, 130.5, 123.6, 122.4, 121.2, 121.1, 120.1, 110.1, 60.6, 51.0, 33.7, 31.0, 26.1, 25.2, 14.1; MS (MALDI) calcd for $C_{19}H_{20}N_3O_2$ $[M + H]^+$ 310.144, found 310.148.

7. ^{1}H and ^{13}C NMR Spectra of Products


7.45 7.44 7.43 7.26 7.24 7.24 7.23 7.23
4.29 4.28 4.26 4.25 3.36 3.36 3.33 3.32 3.28 3.27 3.26 3.24 3.24 3.09 3.09 3.08 3.08 3.07 3.05 3.05 3.04 3.04 3.00 2.99 2.99 2.98 2.97 2.96 2.96 2.95 2.94 2.94 2.93 2.93 2.92 2.92 2.81 2.81 2.80 2.80 2.78 2.77 2.77 2.76 2.76 1.35 1.34 1.33
3.3 3.2 3.1 3.0 2.9 2.8 ppm
Current Data Parameters
NAME ystz44A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060407
Time 20.49
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 71.8
DW 50.000 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300231 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.00 1.02 1.99 1.00 1.00 0.99 1.02 1.02 3.00
9 8 7 6 5 4 3 2 1 ppm
164.66 138.38 136.03 129.31 129.17 128.05 124.90 114.77 113.88 77.26 77.00 76.75 61.33 45.20 37.14 34.75 29.21 14.14
NC CN Ph CO2Et
3a
Current Data Parameters
NAME ystz44A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060407
Time 20.54
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 191
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 724.1
DW 15.300 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080925 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

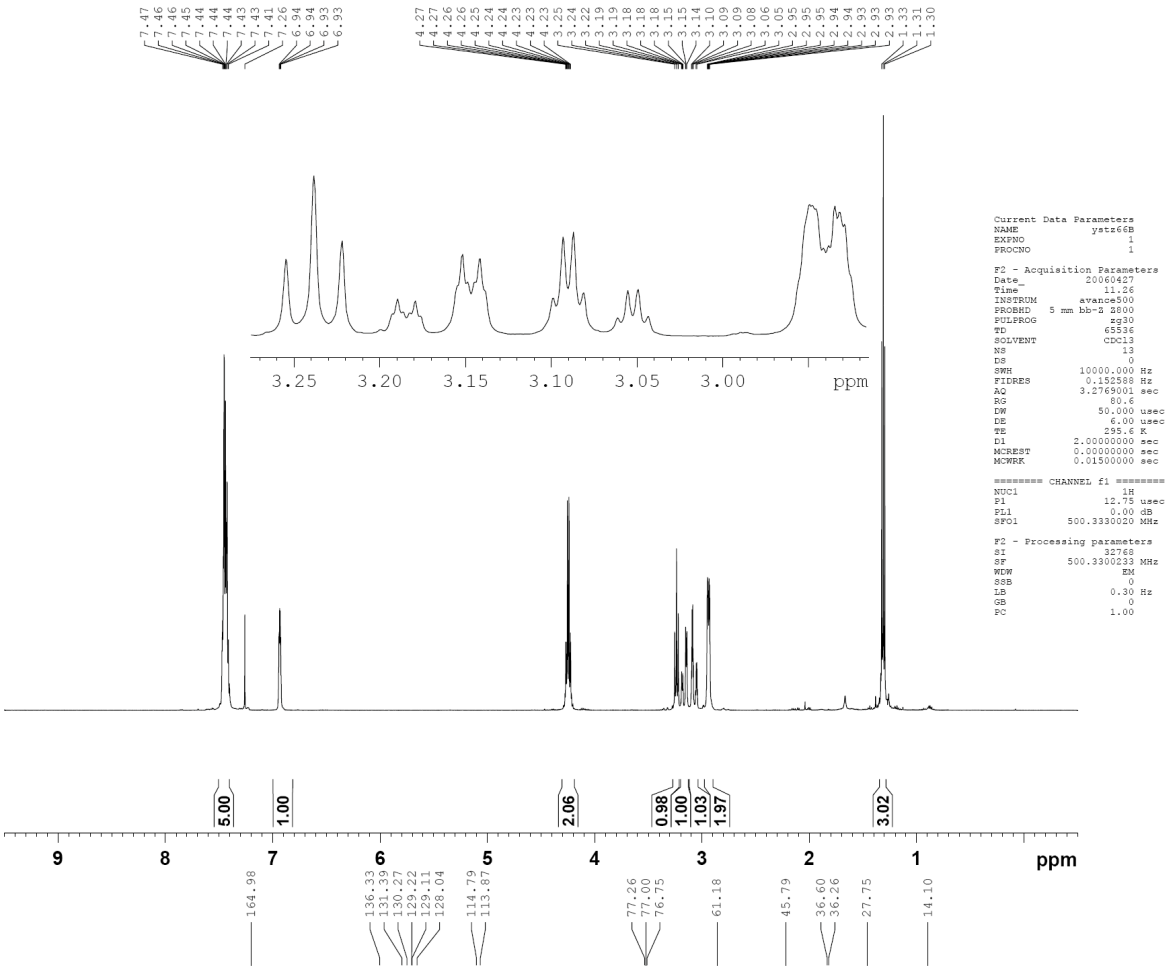

7.36
7.34
7.26
7.23
7.22
7.22
6.96
6.94
4.29
4.27
4.26
4.25
3.83
3.34
3.30
3.24
3.23
3.22
3.21
3.07
3.07
3.06
3.06
3.05
3.04
3.03
3.03
3.02
3.02
2.95
2.95
2.93
2.92
2.91
2.91
2.90
2.90
2.89
2.89
2.79
2.79
2.78
2.77
2.77
2.75
2.75
2.74
2.73
1.35
1.34
1.32
3.3
3.2
3.1
3.0
2.9
2.8
ppm
Current Data Parameters
NAME ystz86CE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060516
Time 9.08
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 48
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 10.90 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 500.1300226 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.01
1.00
2.04
2.11
3.00
1.01
1.00
1.04
1.04
1.01
3.08
9
8
7
6
5
4
3
2
1
ppm
164.75
160.26
138.52
129.27
127.96
124.94
114.97
114.55
114.01
77.26
77.00
76.75
61.35
55.32
44.60
37.57
34.68
29.37
14.19
MeO
CN
CN
CO2Et
3b
Current Data Parameters
NAME ystz86CE
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060516
Time 9.16
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 518
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 7.00 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 - Processing parameters
SI 32768
SF 125.7577939 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

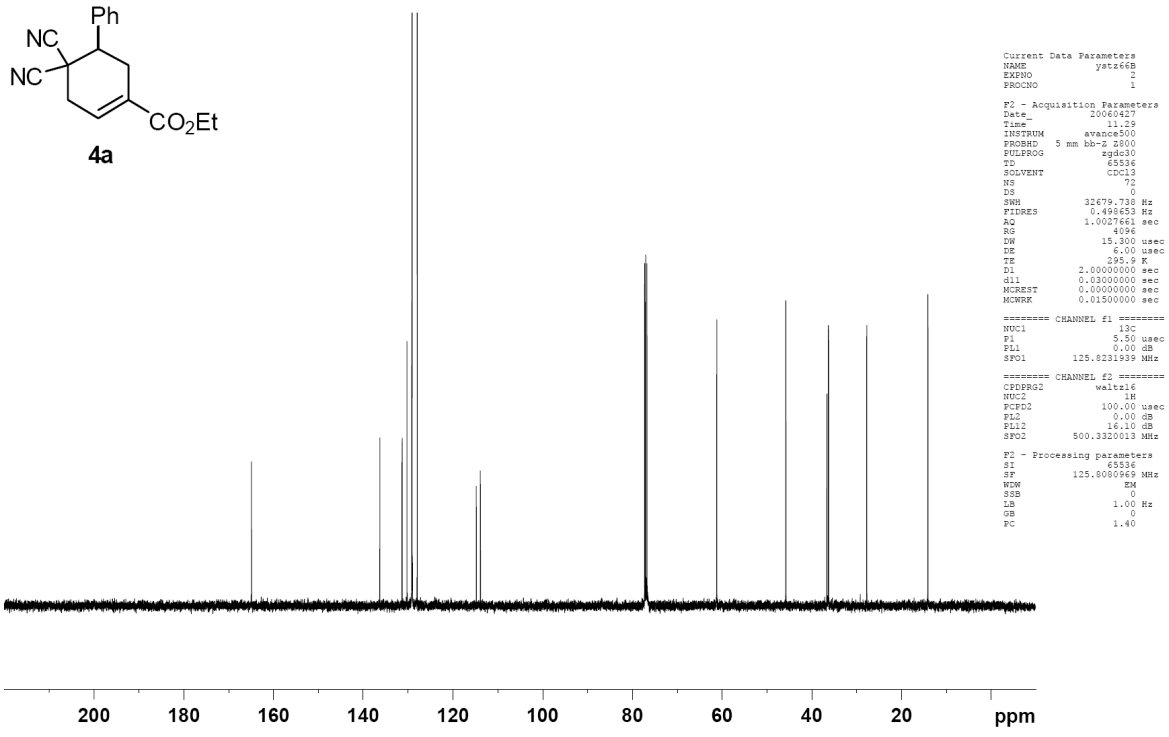

7.58 7.55 7.32 7.30 7.22 7.21 7.20 7.20
4.30 4.28 4.25 4.23 3.36 3.30 3.25 3.24 3.22 3.20 3.15 3.10 3.09 3.08 3.08 3.07 3.06 3.03 3.02 3.01 3.01 3.00 2.98 2.97 2.96 2.95 2.94 2.93 2.92 2.91 2.90 2.89 2.88 2.87 2.86 2.85 2.85 2.84 2.80 2.79 2.78 2.77 2.75 2.74 2.73 2.72 2.71 2.70 1.35 1.33 1.31
Current Data Parameters
NAME ystz38a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070319
Time 19.14
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 114
DW 83.400 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 65536
SF 300.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
3.3 3.2 3.1 3.0 2.9 2.8 ppm
1.99 1.97 1.00 2.08 5.06 3.04
9 8 7 6 5 4 3 2 1 0 ppm

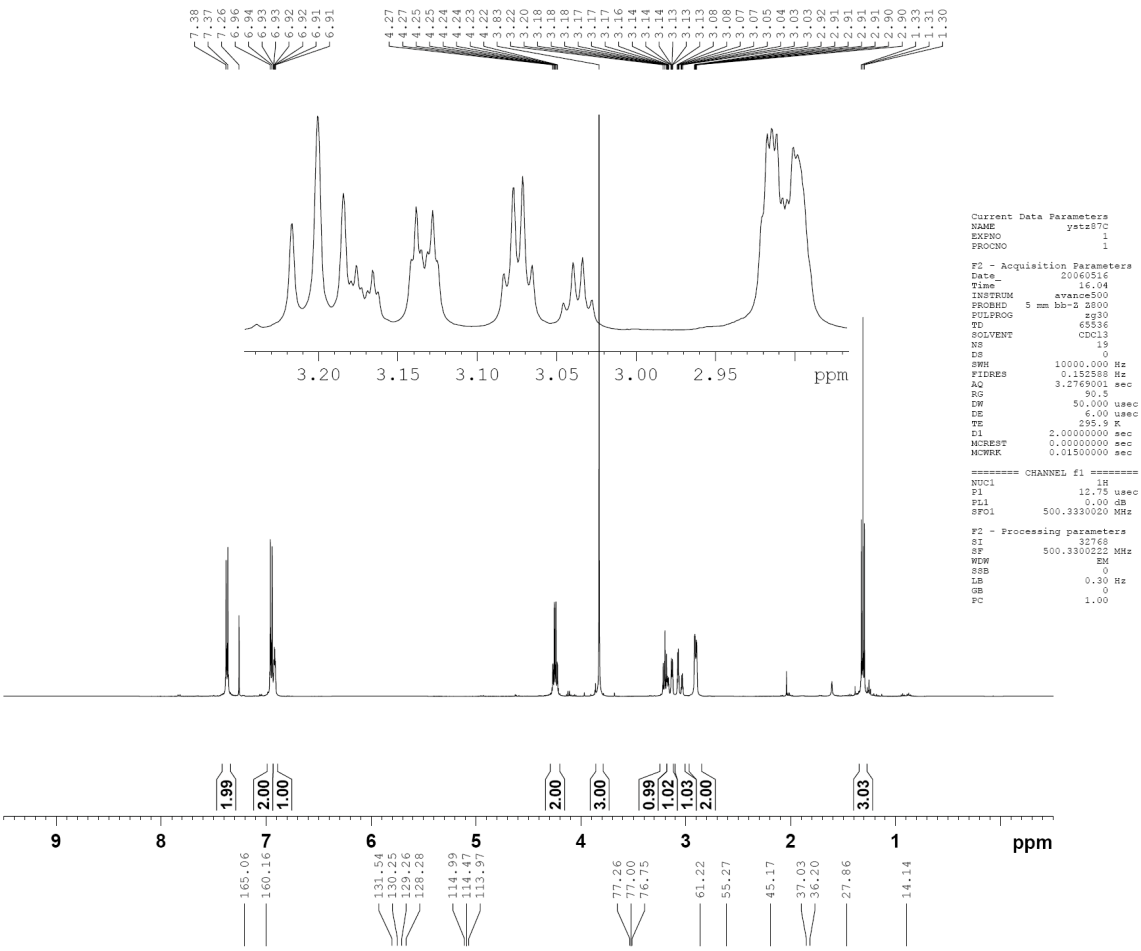

Br
CN
CN
CO2Et

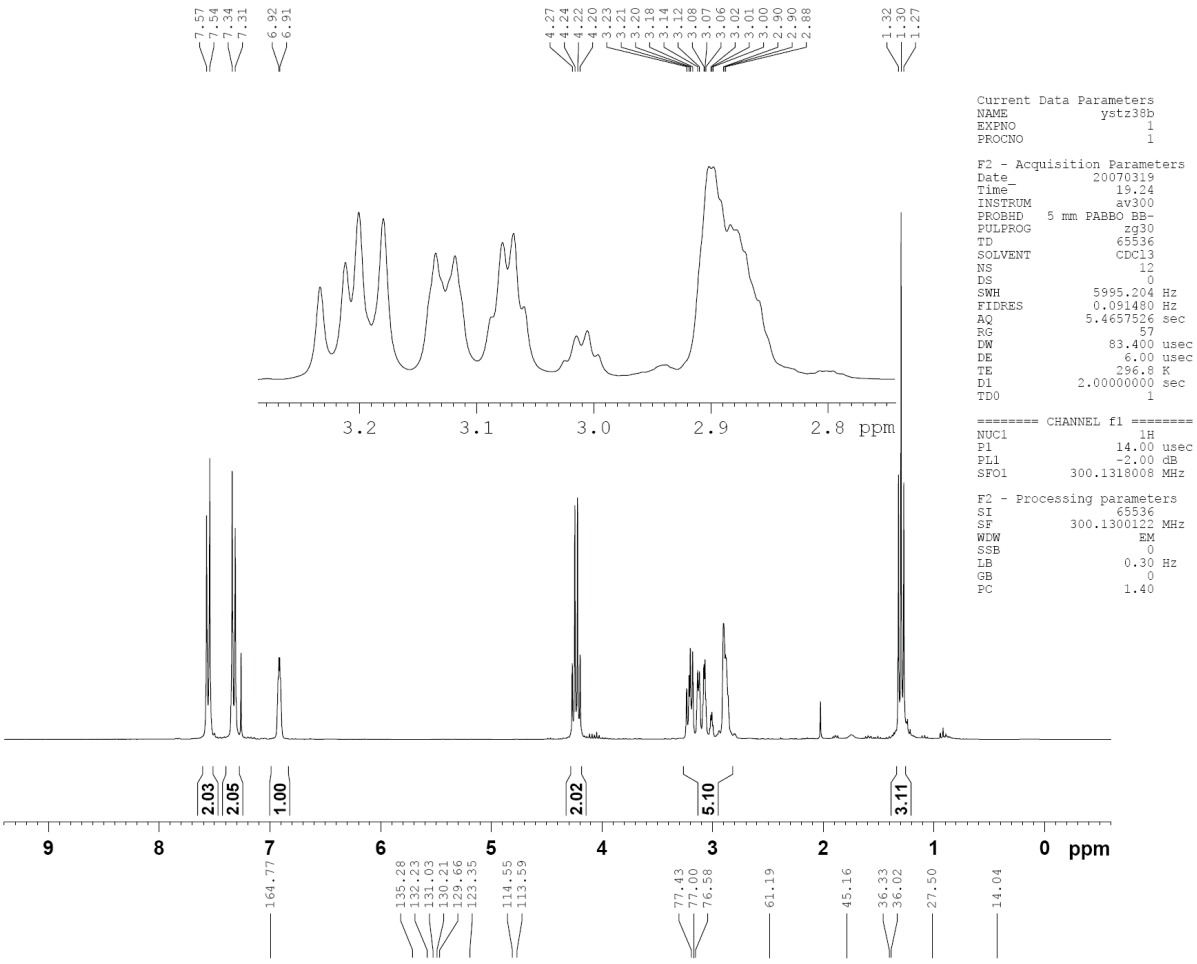
3c

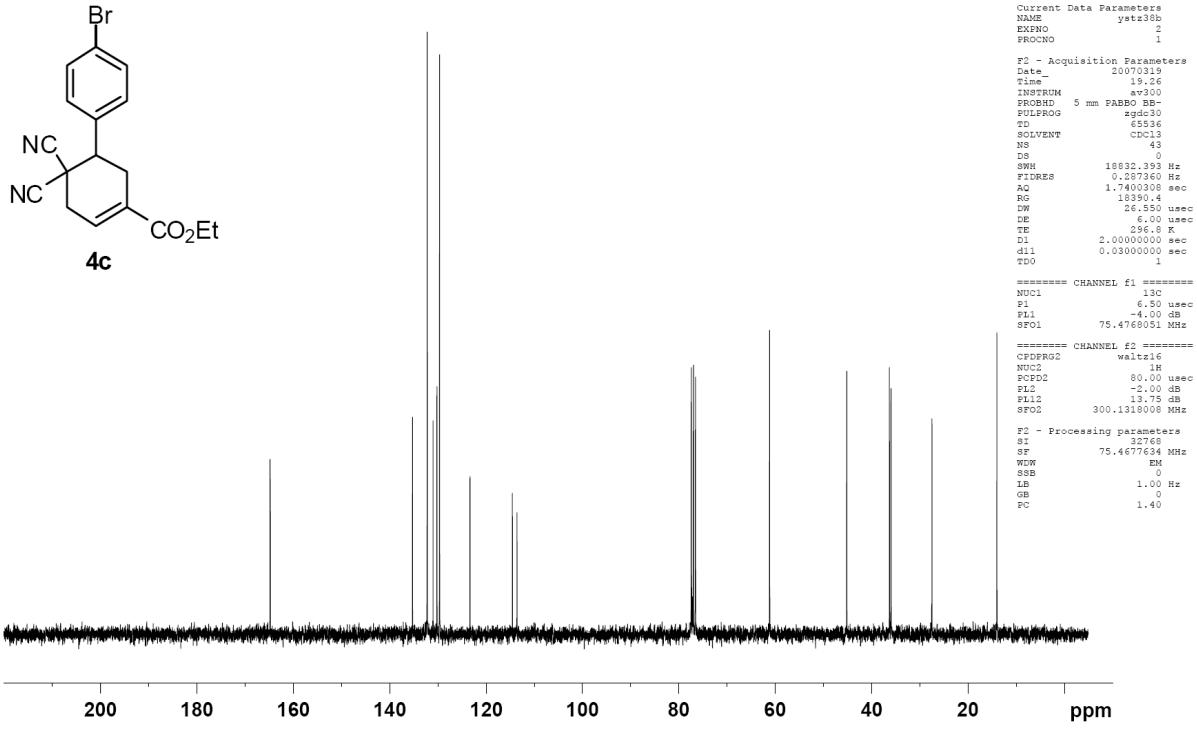
164.53
137.92 134.99 132.38 129.70 124.97 123.55
114.56 113.63
77.43 77.00 76.58
61.39
44.71
36.92 34.63
29.04
14.13
Current Data Parameters
NAME ystz38a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070319
Time 19.19
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 56
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 13004
DW 26.550 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 -4.00 dB
SFO1 75.4768051 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.75 dB
SFO2 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 75.4677564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

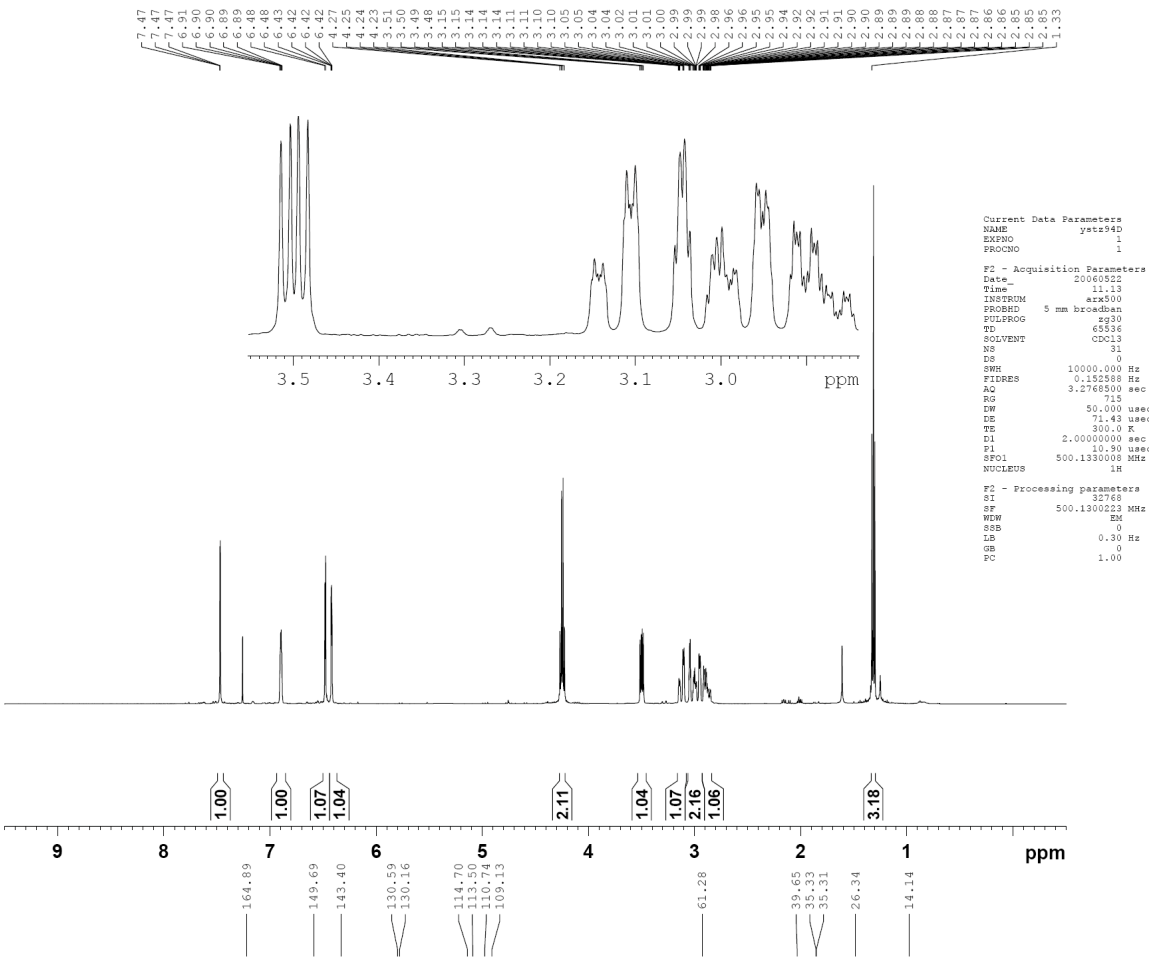

7.47 7.46 7.46 7.45 7.44 7.44 7.44 7.43 7.43 7.41 7.26 6.94 6.94 6.93 6.93
4.27 4.27 4.26 4.26 4.25 4.24 4.24 4.23 4.23 4.23 3.25 3.24 3.22 3.19 3.19 3.18 3.18 3.18 3.15 3.15 3.14 3.10 3.09 3.08 3.06 3.05 2.95 2.95 2.94 2.94 2.93 2.93 1.33 1.31 1.30
3.25 3.20 3.15 3.10 3.05 3.00 ppm
Current Data Parameters
NAME ystz66B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060427
Time 11.26
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300233 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.00 1.00 2.06 0.98 1.00 1.03 1.97 3.02
9 8 7 6 5 4 3 2 1 ppm
164.98 136.33 131.39 130.27 129.22 129.11 128.04 114.79 113.87 77.26 77.00 76.75 61.18 45.79 36.60 36.26 27.75 14.10

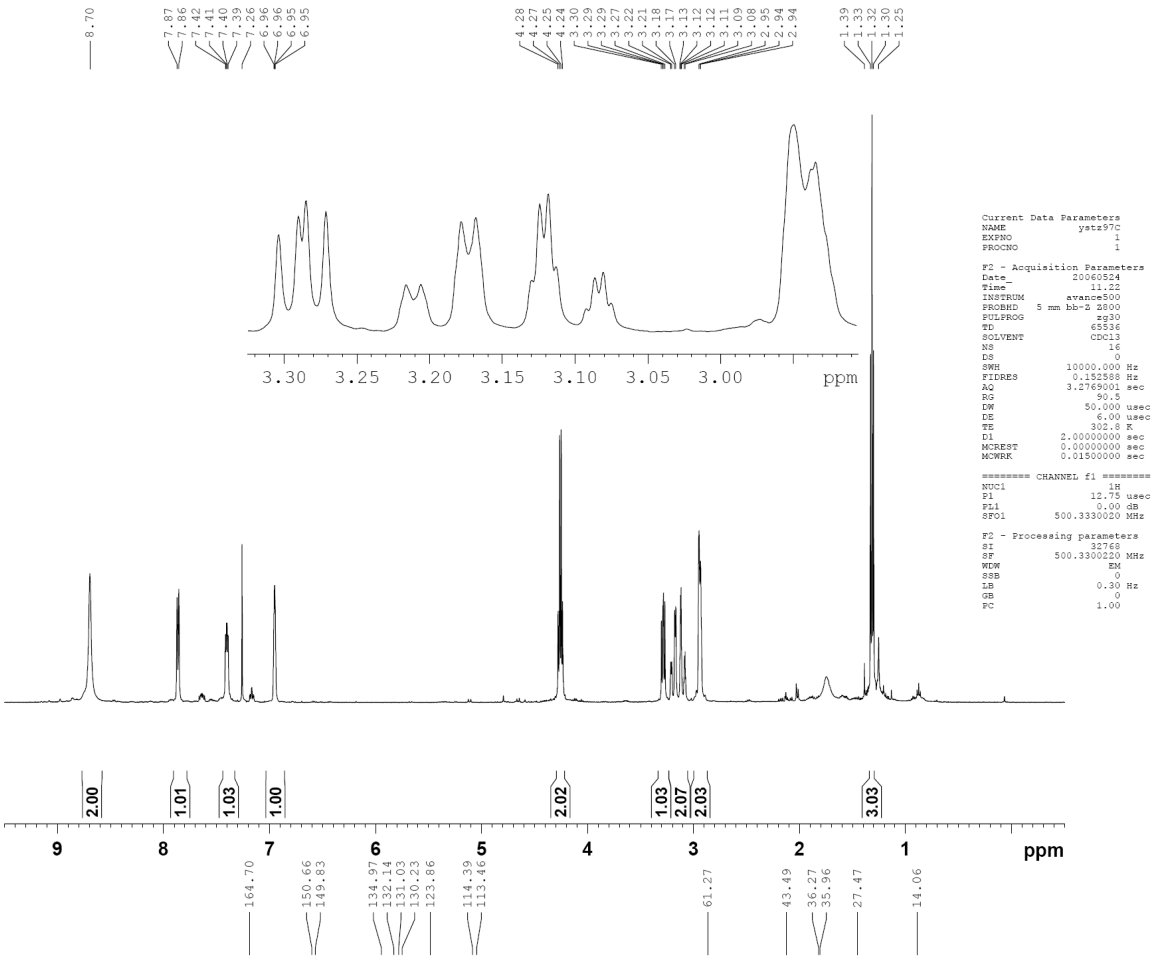

Ph
NC
NC
CO2Et
4a
Current Data Parameters
NAME ystz66B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060427
Time 11.29
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 72
DS 0
SWH 32679.738 Hz
FIDRES 0.498652 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


7.38 7.37 7.26 6.96 6.94 6.93 6.93 6.92 6.92 6.91 6.91
4.27 4.27 4.25 4.25 4.24 4.24 4.23 4.22 3.83 3.22 3.20 3.18 3.18 3.17 3.17 3.16 3.14 3.14 3.14 3.13 3.13 3.13 3.08 3.08 3.07 3.07 3.05 3.04 3.03 3.03 2.92 2.91 2.91 2.91 2.90 2.90 1.33 1.31 1.30
3.20 3.15 3.10 3.05 3.00 2.95 ppm
Current Data Parameters
NAME ystz87c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060516
Time 16.04
INSTRUM avance500
PROBHD 5 mm bb-2 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 19
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.99 2.00 1.00 2.00 3.00 0.99 1.02 1.03 2.00 3.03
9 8 7 6 5 4 3 2 1 ppm
165.06 160.16 131.54 130.25 129.26 128.28 114.99 114.47 113.97 77.26 77.00 76.75 61.22 55.27 45.17 37.03 36.20 27.86 14.14
OMe
CN
NC
CO2Et
4b
Current Data Parameters
NAME ystz87c
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060516
Time 16.08
INSTRUM avance500
PROBHD 5 mm bb-2 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 66
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080893 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


7.57
7.54
7.34
7.31
6.92
6.91
4.27
4.24
4.22
4.20
3.23
3.21
3.20
3.18
3.14
3.12
3.08
3.07
3.06
3.02
3.01
3.00
2.90
2.90
2.88
1.32
1.30
1.27
3.2
3.1
3.0
2.9
2.8 ppm
2.03
2.05
1.00
2.02
5.10
3.11
9
8
7
6
5
4
3
2
1
0 ppm
164.77
135.28
132.23
131.03
130.21
129.66
123.35
114.55
113.59
77.43
77.00
76.58
61.19
45.16
36.33
36.02
27.50
14.04
Current Data Parameters
NAME ystz38b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070319
Time_ 19.24
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 57
DW 83.400 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 65536
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


Br
NC
NC
CO2Et
4c
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME ystz38b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070319
Time_ 19.26
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 43
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 18390.4
DW 26.550 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 6.50 usec
PL1 -4.00 dB
SFO1 75.4768051 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.75 dB
SFO2 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 75.4677634 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.47 7.47 7.47 6.91 6.90 6.90 6.89 6.89 6.48 6.48 6.43 6.42 6.42 6.42 4.27 4.25 4.24 4.23 3.51 3.50 3.49 3.48 3.15 3.15 3.14 3.14 3.14 3.11 3.11 3.10 3.10 3.05 3.05 3.04 3.04 3.02 3.01 3.01 3.00 2.99 2.99 2.99 2.98 2.96 2.96 2.95 2.95 2.94 2.92 2.92 2.91 2.91 2.90 2.90 2.89 2.89 2.88 2.88 2.87 2.87 2.87 2.86 2.86 2.85 2.85 2.85 2.82 1.33
3.5 3.4 3.3 3.2 3.1 3.0 ppm
Current Data Parameters
NAME ystz94D
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060522
Time 11.13
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 31
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 715
DW 50.000 use
DE 71.43 use
TE 300.0 K
D1 2.00000000 sec
P1 10.90 use
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 500.1300223 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00 1.00 1.07 1.04 2.11 1.04 1.07 2.16 1.06 3.18
9 8 7 6 5 4 3 2 1 ppm
164.89 149.69 143.40 130.59 130.16 114.70 113.50 110.74 109.13 61.28 39.65 35.33 35.31 26.34 14.14
O NC NC CO2Et
4d
Current Data Parameters
NAME ystz94D
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060522
Time 11.18
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 85
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 use
DE 20.00 use
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 use
D1 2.00000000 sec
P1 7.00 use
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 - Processing parameters
SI 32768
SF 125.7577980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

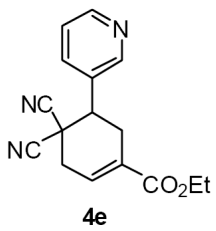


Current Data Parameters
NAME ystz97C
EXPNO 1
PROCNO 1

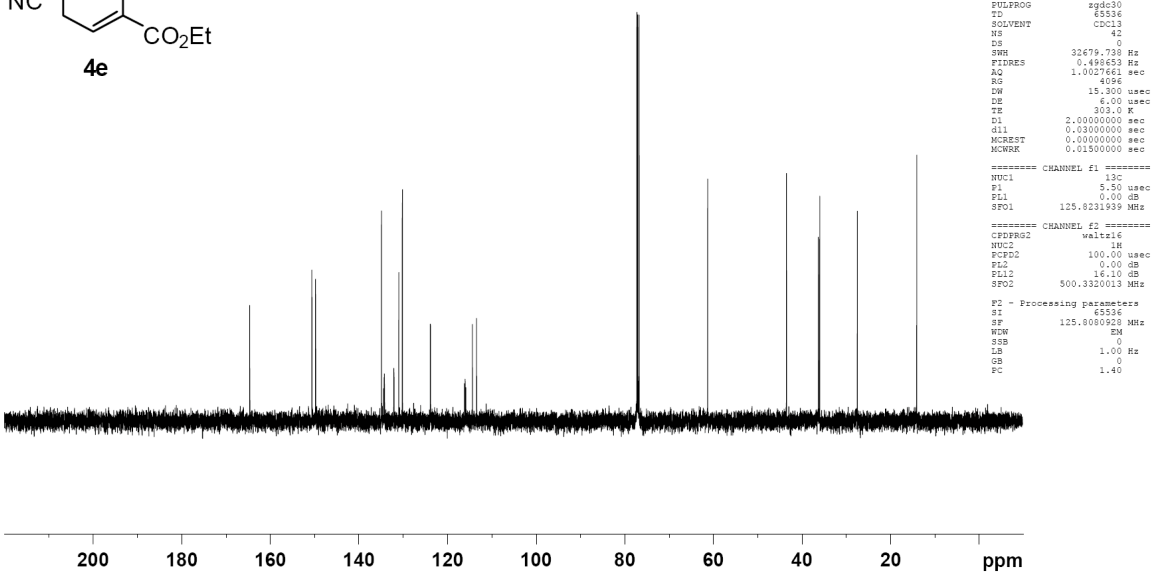
F2 - Acquisition Parameters
Date_ 20060524
Time 11.22
INSTRUM avance500
PROBHD 5 mm bb-2 Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 302.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4e



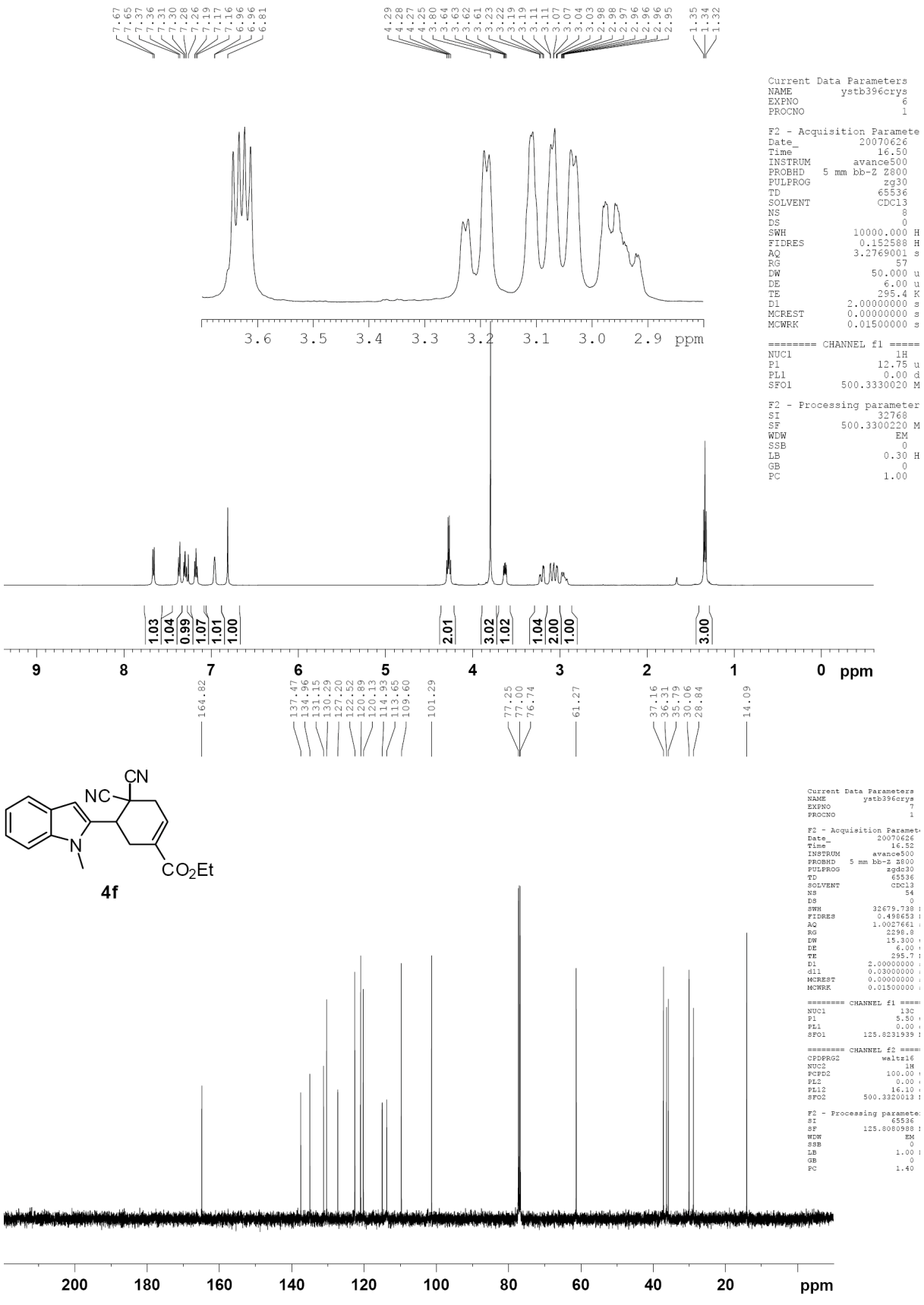
Current Data Parameters
NAME ystz97A
EXPNO 2
PROCNO 1

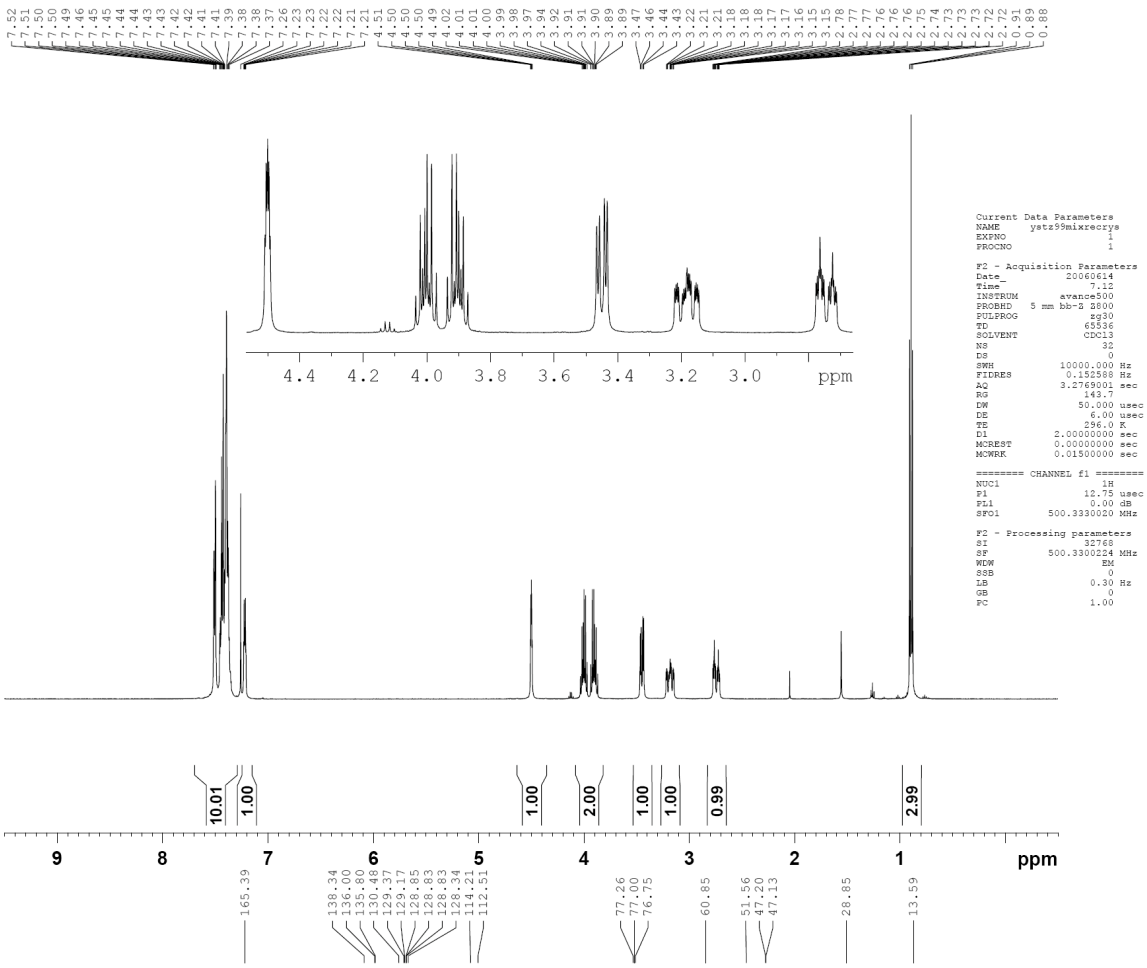
F2 - Acquisition Parameters
Date_ 20060524
Time 11.15
INSTRUM avance500
PROBHD 5 mm bb-2 Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 42
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 303.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

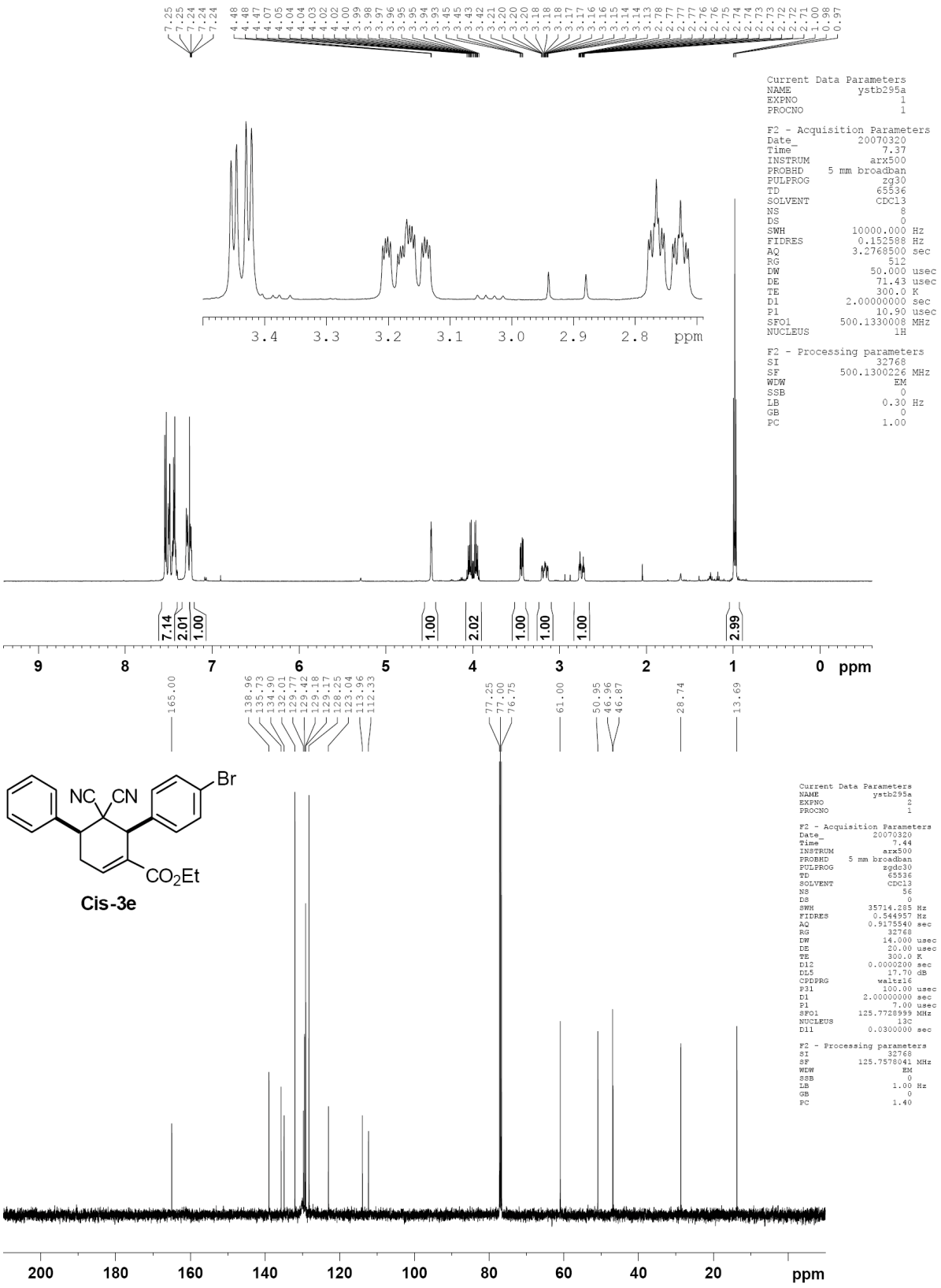
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

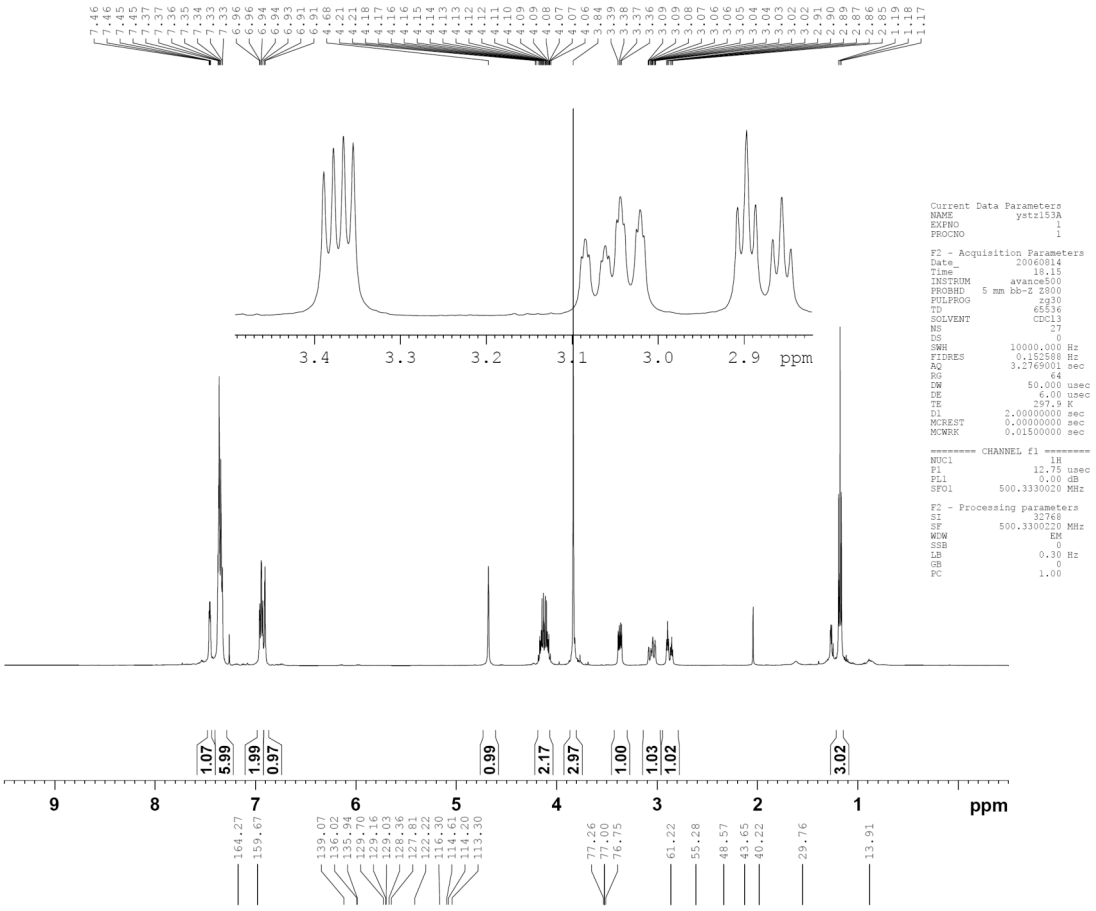
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

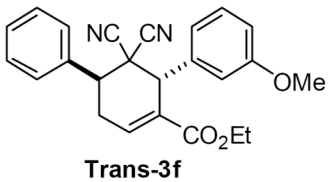
F2 - Processing parameters
SI 65536
SF 125.8080928 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.67 7.65 7.37 7.36 7.31 7.30 7.28 7.26 7.19 7.17 7.16 6.96 6.96 6.81
4.29 4.28 4.27 4.25 3.80 3.64 3.63 3.62 3.61 3.23 3.22 3.19 3.19 3.11 3.07 3.07 3.04 3.03 2.98 2.98 2.97 2.96 2.96 2.96 2.95
1.35 1.34 1.32
Current Data Parameters
NAME ystb396crys
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070626
Time 16.50
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 s
RG 57
DW 50.000 us
DE 6.00 us
TE 295.4 K
D1 2.00000000 s
MCREST 0.00000000 s
MCWRK 0.01500000 s
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 us
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.6 3.5 3.4 3.3 3.2 3.1 3.0 2.9 ppm
1.03 1.04 0.99 1.07 1.01 1.00 2.01 3.02 1.02 1.04 2.00 1.00 3.00
9 8 7 6 5 4 3 2 1 0 ppm
164.82 137.47 134.96 131.15 130.29 127.20 122.52 120.89 120.13 114.93 113.65 109.60 101.29 77.25 77.00 76.74 61.27 37.16 36.31 35.79 30.06 28.84 14.09
CN
NC
N
CO2Et
4f
Current Data Parameters
NAME ystb396crys
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070626
Time 16.52
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 54
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 s
RG 2298.8
DW 15.300 us
DE 6.00 us
TE 295.7 K
D1 2.00000000 s
d11 0.03000000 s
MCREST 0.00000000 s
MCWRK 0.01500000 s
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 us
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080988 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

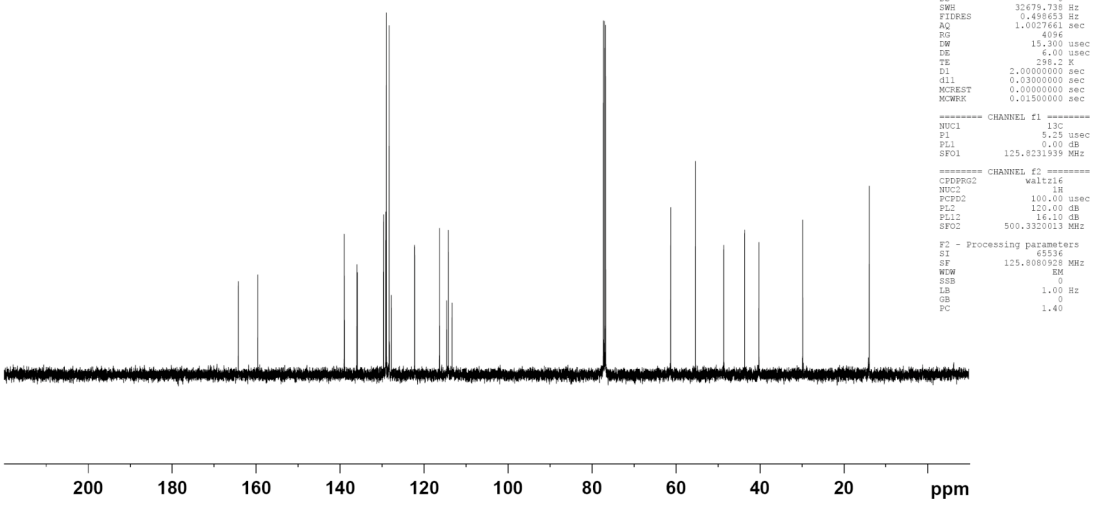

7.52 7.51 7.50 7.50 7.49 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.39 7.38 7.38 7.37 7.26 7.23 7.22 7.21 7.21
4.51 4.50 4.50 4.49 4.02 4.01 4.01 4.00 3.99 3.98 3.97 3.94 3.92 3.91 3.91 3.90 3.89 3.89 3.47 3.46 3.44 3.43 3.22 3.21 3.21 3.18 3.18 3.18 3.17 3.17 3.16 3.15 3.15 2.78 2.77 2.76 2.76 2.76 2.75 2.74 2.73 2.73 2.72 2.72 2.72 0.91 0.89 0.88
4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 ppm
Current Data Parameters
NAME ystz99mixrecrys
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060614
Time 7.12
INSTRUM avance500
PROBHD 5 mm bb-S 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300224 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.01 1.00 1.00 2.00 1.00 1.00 0.99 2.99
9 8 7 6 5 4 3 2 1 ppm
165.39 138.34 136.00 135.80 130.48 129.37 129.17 128.85 128.83 128.83 128.24 114.21 112.51 77.26 77.00 76.75 60.85 51.56 47.20 47.13 28.85 13.59
NC CN Ph Ph CO2Et
Cis-3d
Current Data Parameters
NAME ystz99mixrecrys
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060614
Time 15.12
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 635
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1149.4
DW 15.300 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080869 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

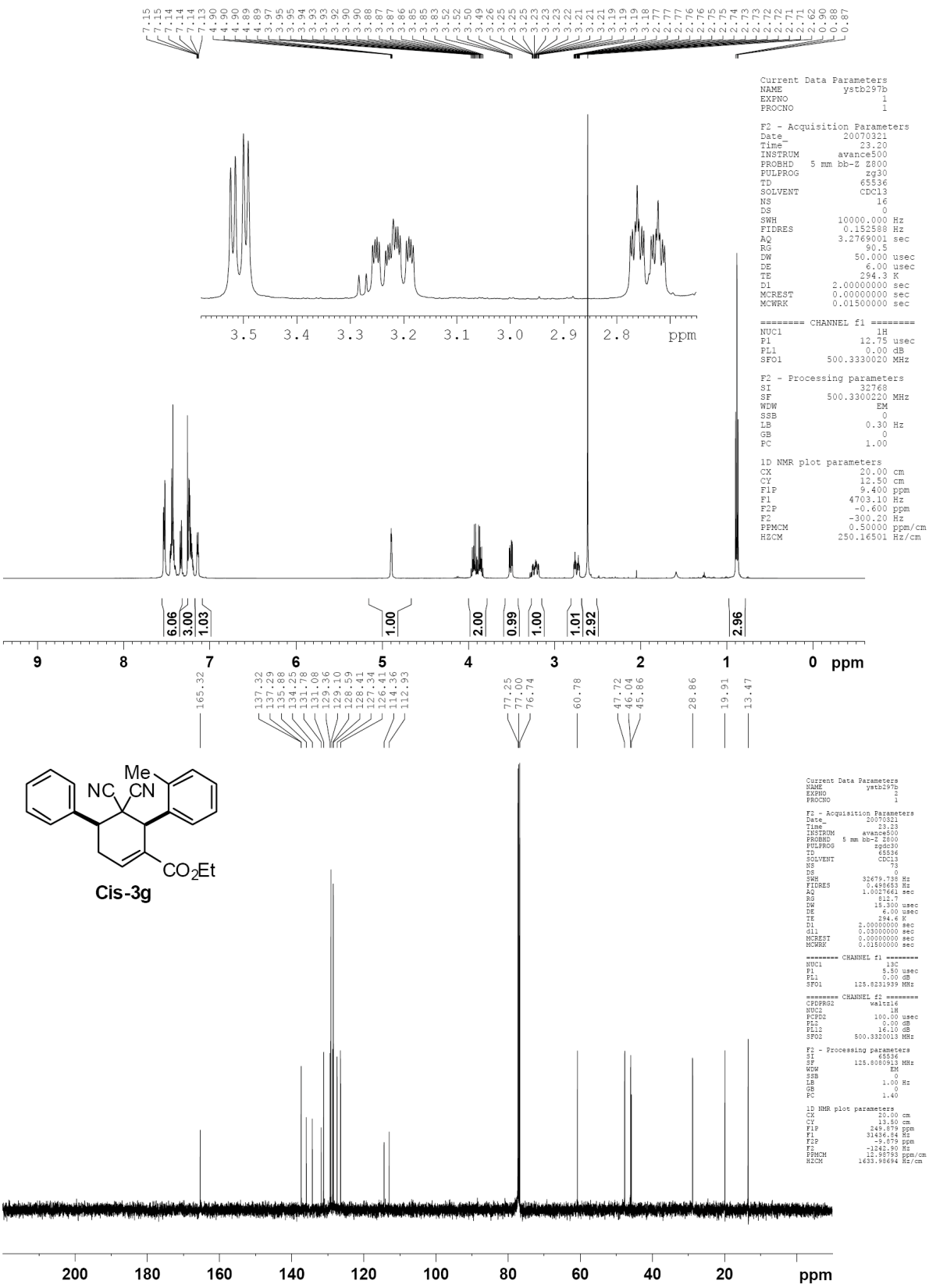

Current Data Parameters
NAME ystb295a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070320
Time 7.37
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 512
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 10.90 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 500.1300226 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.14
2.01
1.00
1.00
2.02
1.00
1.00
1.00
2.99
ppm
NC
CN
Br
CO2Et
Cis-3e
Current Data Parameters
NAME ystb295a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070320
Time 7.44
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 56
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 7.00 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 - Processing parameters
SI 32768
SF 125.7578041 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

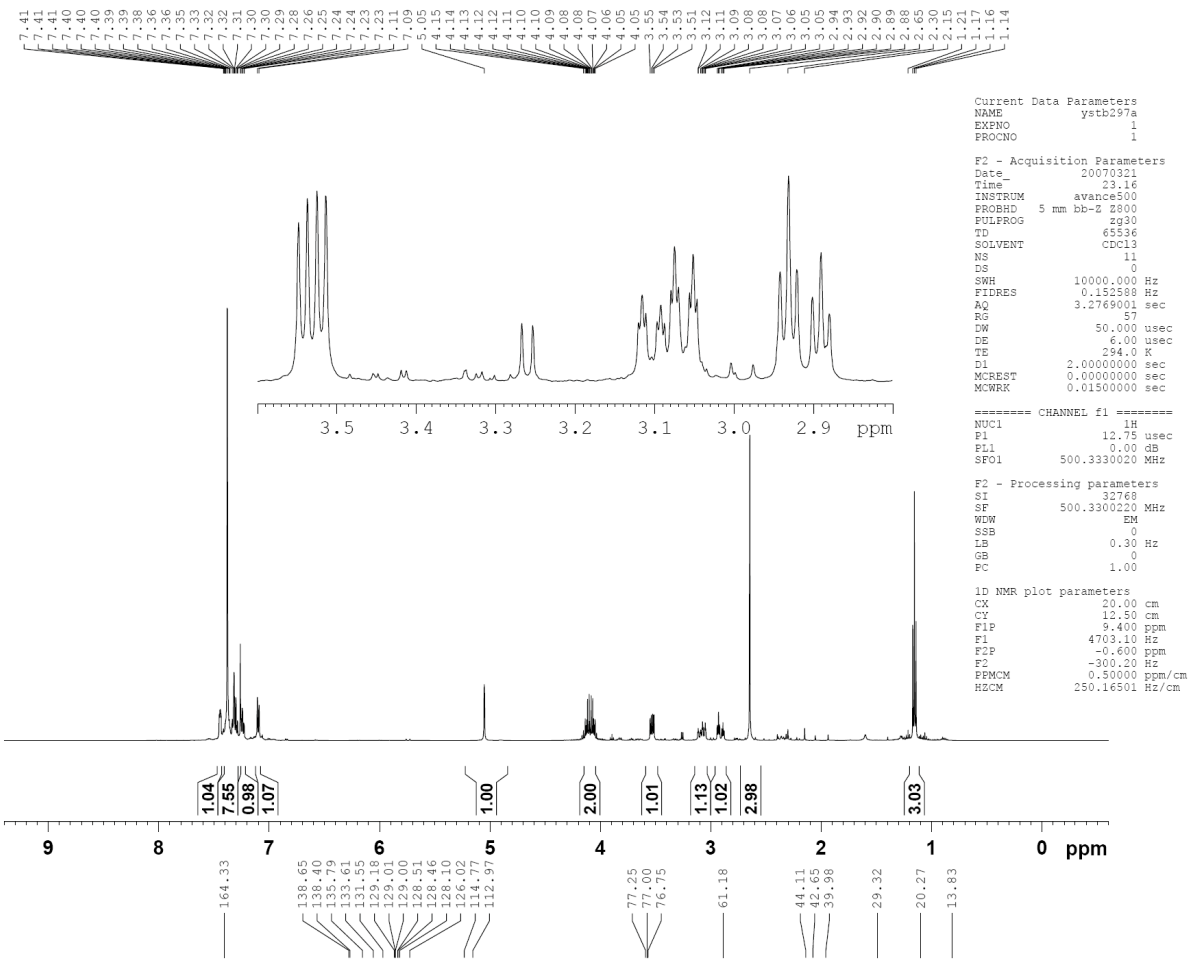

7.46 7.46 7.45 7.45 7.37 7.37 7.36 7.35 7.34 7.33 7.33 6.96 6.96 6.94 6.94 6.93 6.91 6.91 4.68 4.21 4.21 4.18 4.17 4.16 4.16 4.15 4.14 4.13 4.13 4.12 4.12 4.11 4.10 4.09 4.09 4.08 4.07 4.07 4.06 3.84 3.39 3.38 3.37 3.36 3.09 3.09 3.08 3.07 3.06 3.05 3.04 3.04 3.03 3.02 3.02 2.91 2.90 2.89 2.87 2.86 2.85 1.19 1.18 1.17
3.4 3.3 3.2 3.1 3.0 2.9 ppm
Current Data Parameters
NAME ystz153A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060814
Time_ 18.15
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 27
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 64
DW 50.000 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.07 5.99 1.99 0.97 0.99 2.17 2.97 1.00 1.03 1.02 3.02
9 8 7 6 5 4 3 2 1 ppm
164.27 159.67 139.07 136.02 135.94 129.70 129.16 129.03 128.36 127.81 122.22 116.30 114.61 114.20 113.30 77.26 77.00 76.75 61.22 55.28 48.57 43.65 40.22 29.76 13.91

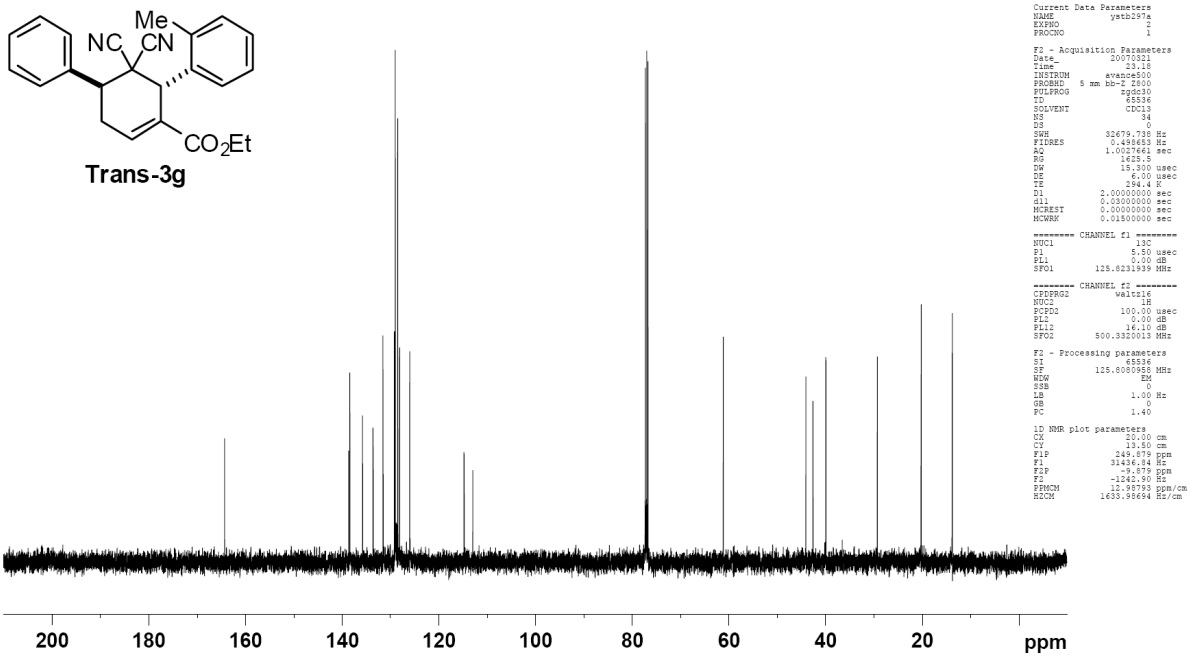

NC
CN
OMe
CO2Et

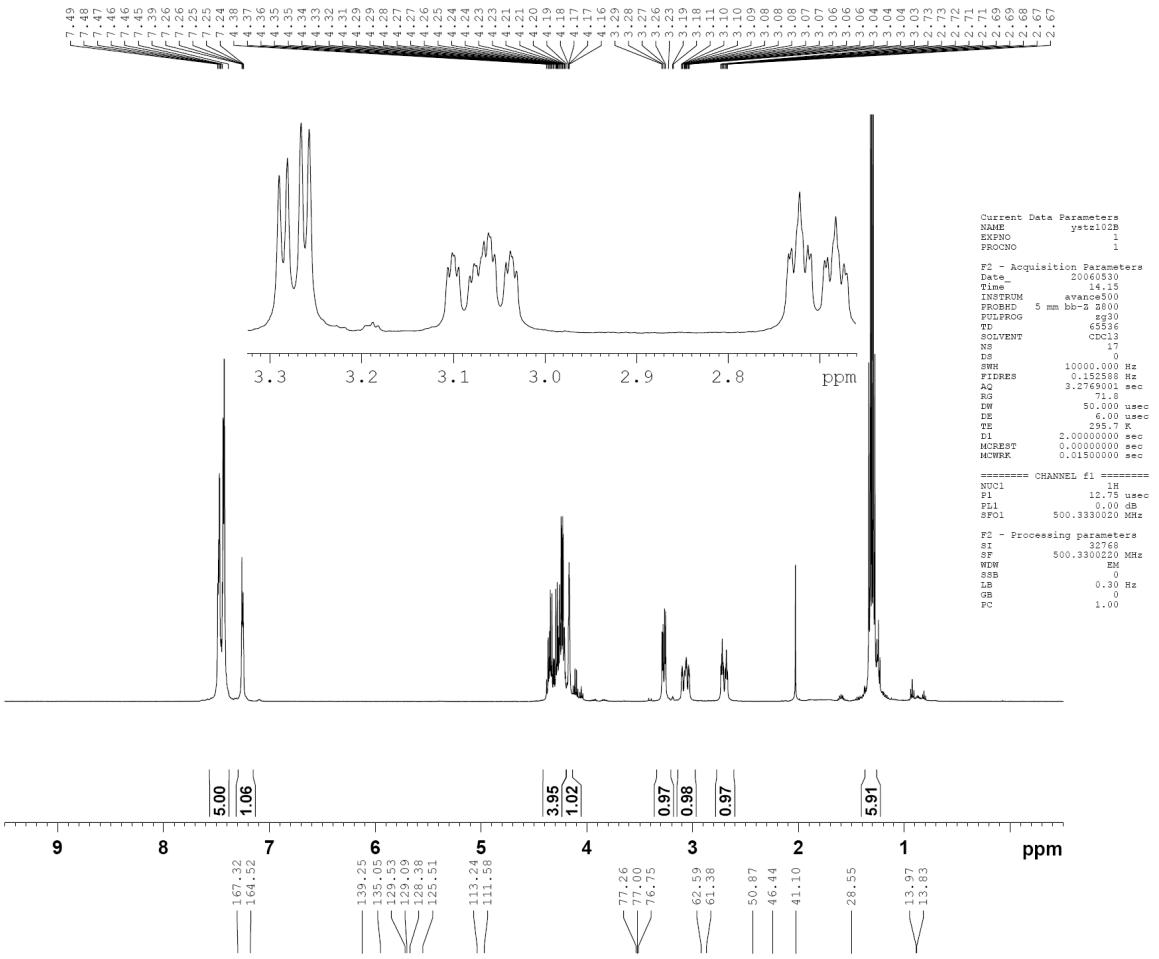
Trans-3f

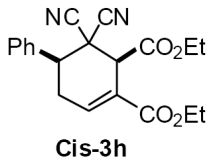

Current Data Parameters
NAME ystz153A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060814
Time_ 18.19
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 49
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080928 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


Me
NC
CN
CO2Et
Cis-3g
ppm

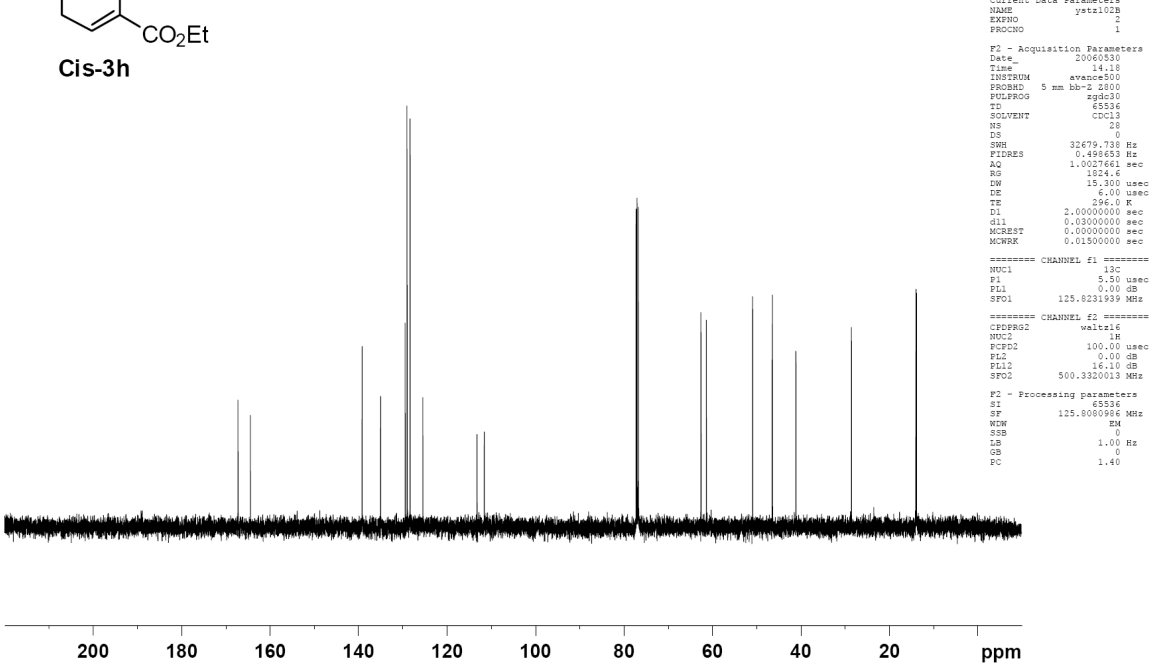

7.41 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.38 7.36 7.36 7.35 7.33 7.32 7.32 7.31 7.30 7.30 7.29 7.28 7.26 7.25 7.24 7.24 7.23 7.23 7.11 7.09 5.05 4.15 4.14 4.13 4.12 4.12 4.11 4.10 4.10 4.09 4.08 4.08 4.07 4.06 4.05 4.05 3.55 3.54 3.53 3.51 3.12 3.11 3.09 3.08 3.08 3.07 3.06 3.05 3.05 2.94 2.93 2.92 2.90 2.89 2.88 2.65 2.30 2.15 1.21 1.17 1.16 1.14
Current Data Parameters
NAME ystb297a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070321
Time 23.16
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 294.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 9.400 ppm
F1 4703.10 Hz
F2P -0.600 ppm
F2 -300.20 Hz
PPMCM 0.50000 ppm/cm
HZCM 250.16501 Hz/cm
3.5 3.4 3.3 3.2 3.1 3.0 2.9 ppm
1.04 7.55 0.98 1.07 1.00 2.00 1.01 1.13 1.02 2.98 3.03
9 8 7 6 5 4 3 2 1 0 ppm
164.33 138.65 138.40 135.79 133.61 131.55 129.18 129.01 129.00 128.51 128.46 128.10 126.02 114.77 112.97 77.25 77.00 76.75 61.18 44.11 42.65 39.98 29.32 20.27 13.83

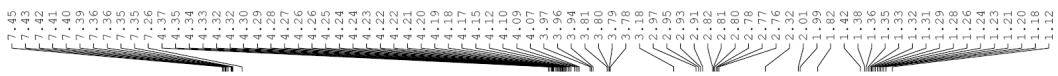

Me
NC
CN
CO2Et
Trans-3g
Current Data Parameters
NAME ystb297a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070321
Time 23.16
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 34
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1625.5
DW 15.300 usec
DE 6.00 usec
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080958 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 13.50 cm
F1P 249.879 ppm
F1 31436.84 Hz
F2P -9.879 ppm
F2 -1242.90 Hz
PPMCM 12.98793 ppm/cm
HZCM 1633.98694 Hz/cm
200 180 160 140 120 100 80 60 40 20 ppm

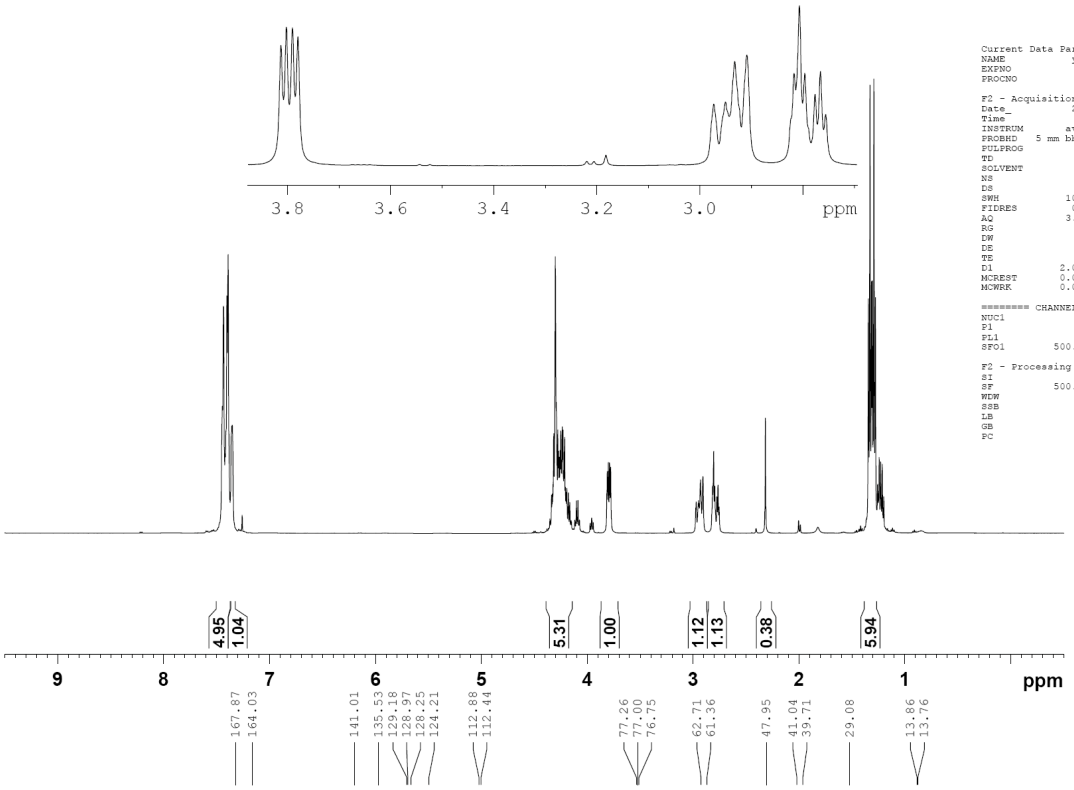

7.49 7.48 7.47 7.46 7.46 7.45 7.39 7.26 7.26 7.25 7.25 7.24
4.38 4.37 4.36 4.35 4.35 4.34 4.33 4.32 4.31 4.29 4.29 4.28 4.27 4.27 4.26 4.25 4.24 4.24 4.23 4.23 4.21 4.21 4.20 4.19 4.18 4.17 4.17 4.16
3.29 3.28 3.27 3.26 3.23 3.19 3.18 3.11 3.10 3.10 3.09 3.08 3.08 3.07 3.07 3.06 3.06 3.06 3.04 3.04 3.04 3.03 2.73 2.72 2.71 2.71 2.69 2.69 2.68 2.67 2.67
3.3 3.2 3.1 3.0 2.9 2.8 ppm
Current Data Parameters
NAME ystz102B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060530
Time 14.15
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 71.8
DW 50.000 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.00 1.06 3.95 1.02 0.97 0.98 0.97 5.91
9 8 7 6 5 4 3 2 1 ppm
167.32 164.52 139.25 135.05 129.53 129.09 128.38 125.51 113.24 111.58 77.26 77.00 76.75 62.59 61.38 50.87 46.44 41.10 28.55 13.97 13.83


NC CN
Ph
CO2Et
CO2Et

Cis-3h


Current Data Parameters
NAME ystz102B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060530
Time 14.18
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 28
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1824.6
DW 15.300 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080986 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm



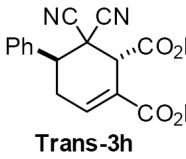


Current Data Parameters
NAME ystz102A
EXPNO 1
PROCNO 1

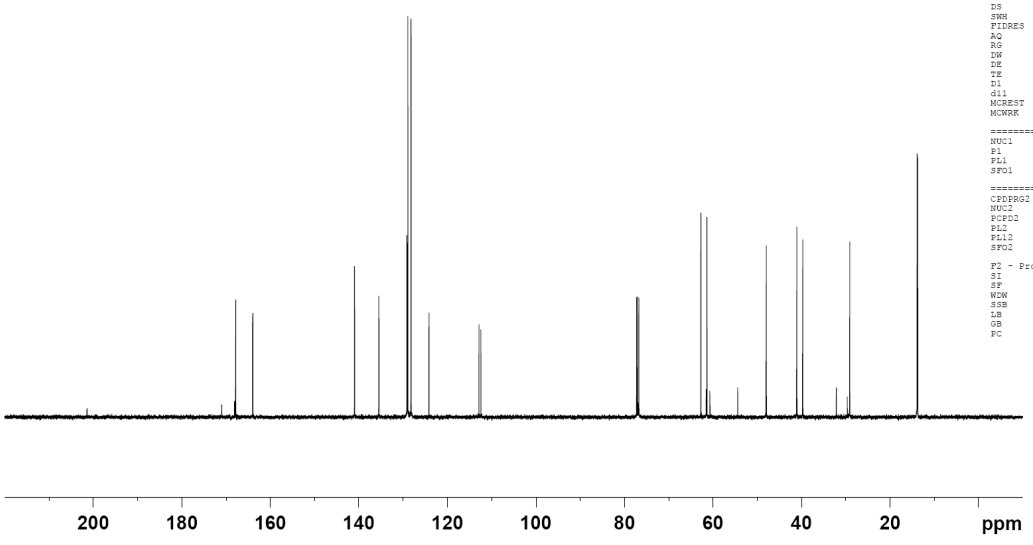
F2 - Acquisition Parameters
Date_ 20060530
Time 15.11
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 35.9
DW 50.000 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Trans-3h



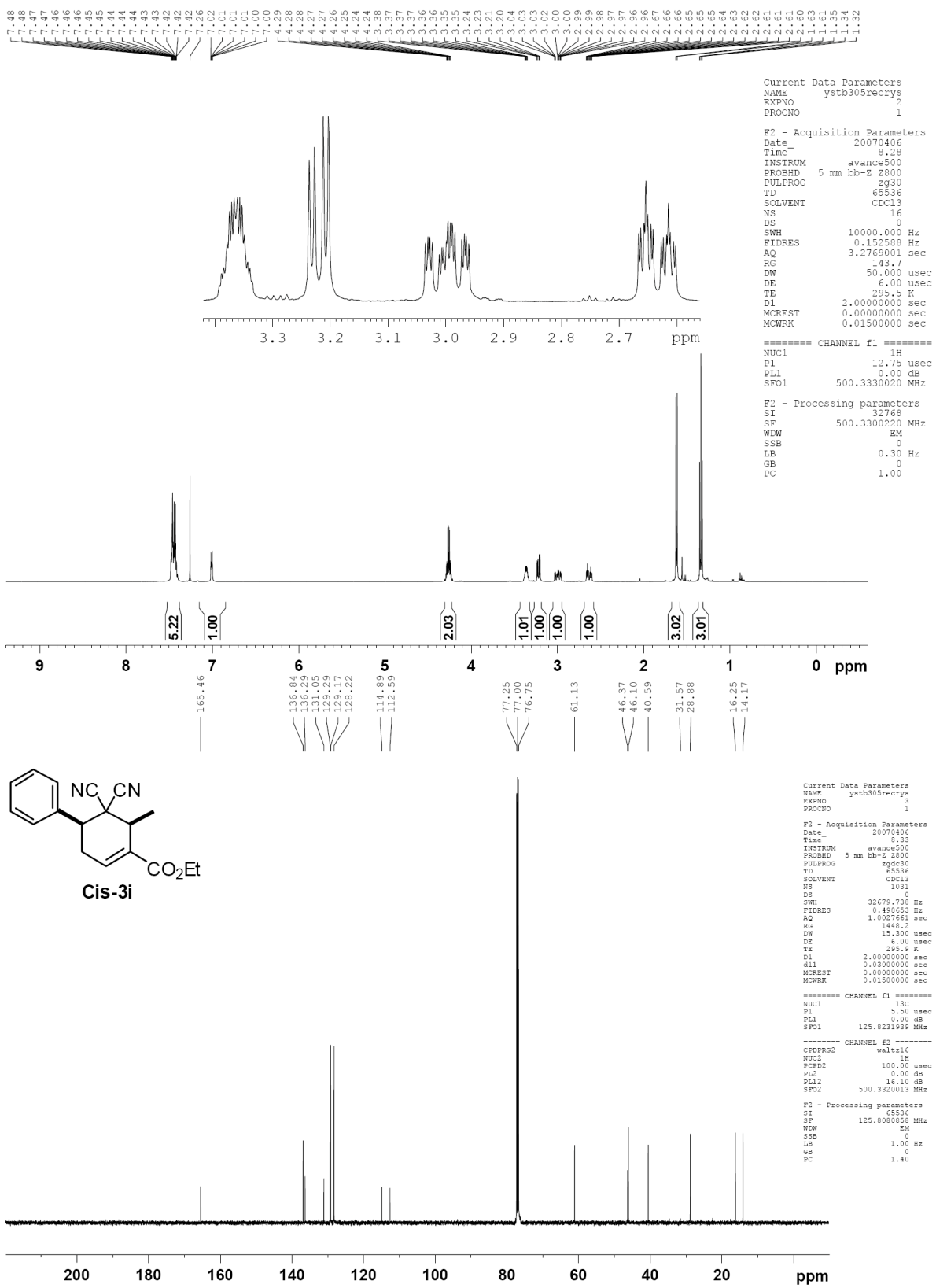
Current Data Parameters
NAME ystz102A
EXPNO 2
PROCNO 1

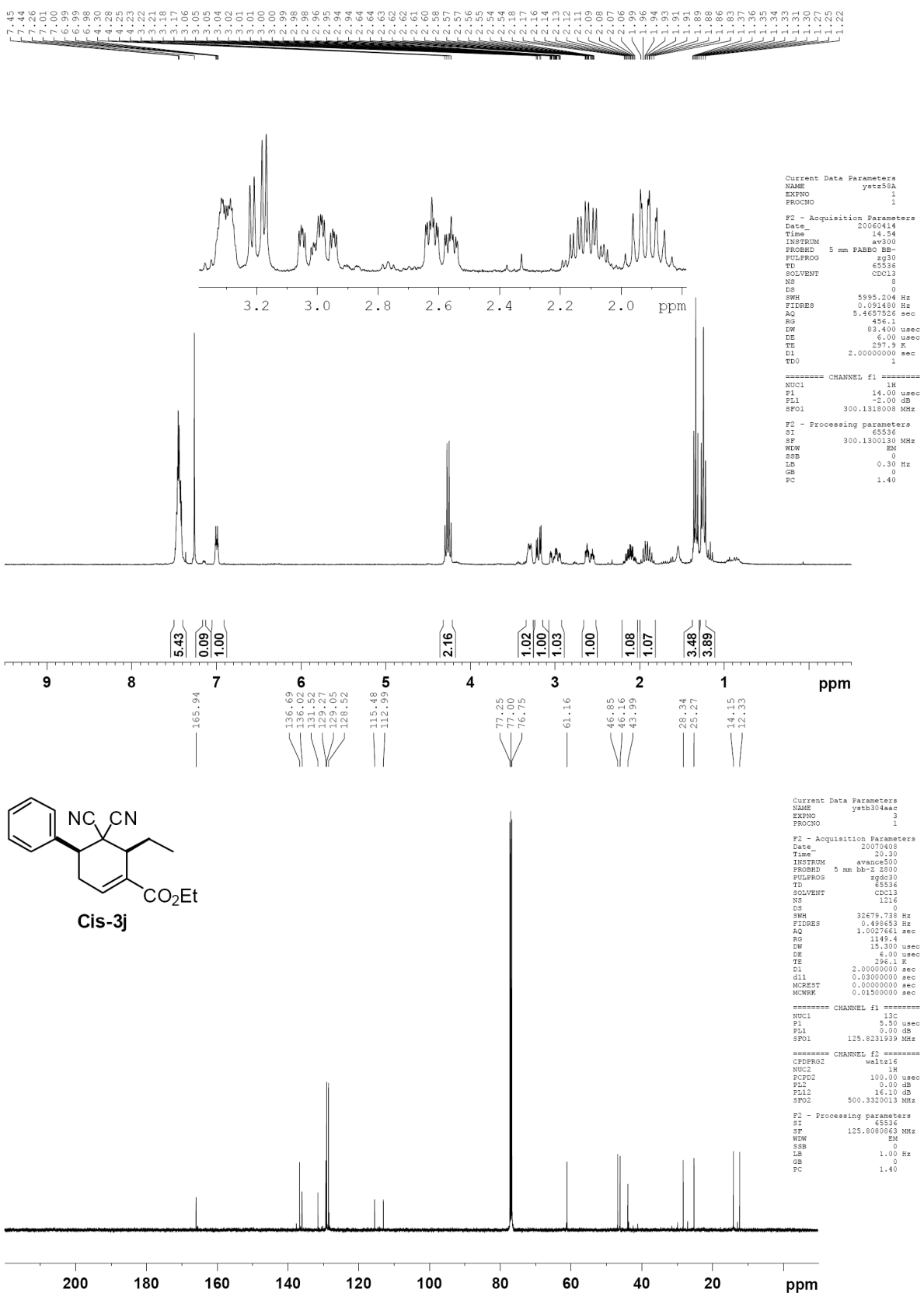
F2 - Acquisition Parameters
Date_ 20060530
Time 15.14
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 60
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

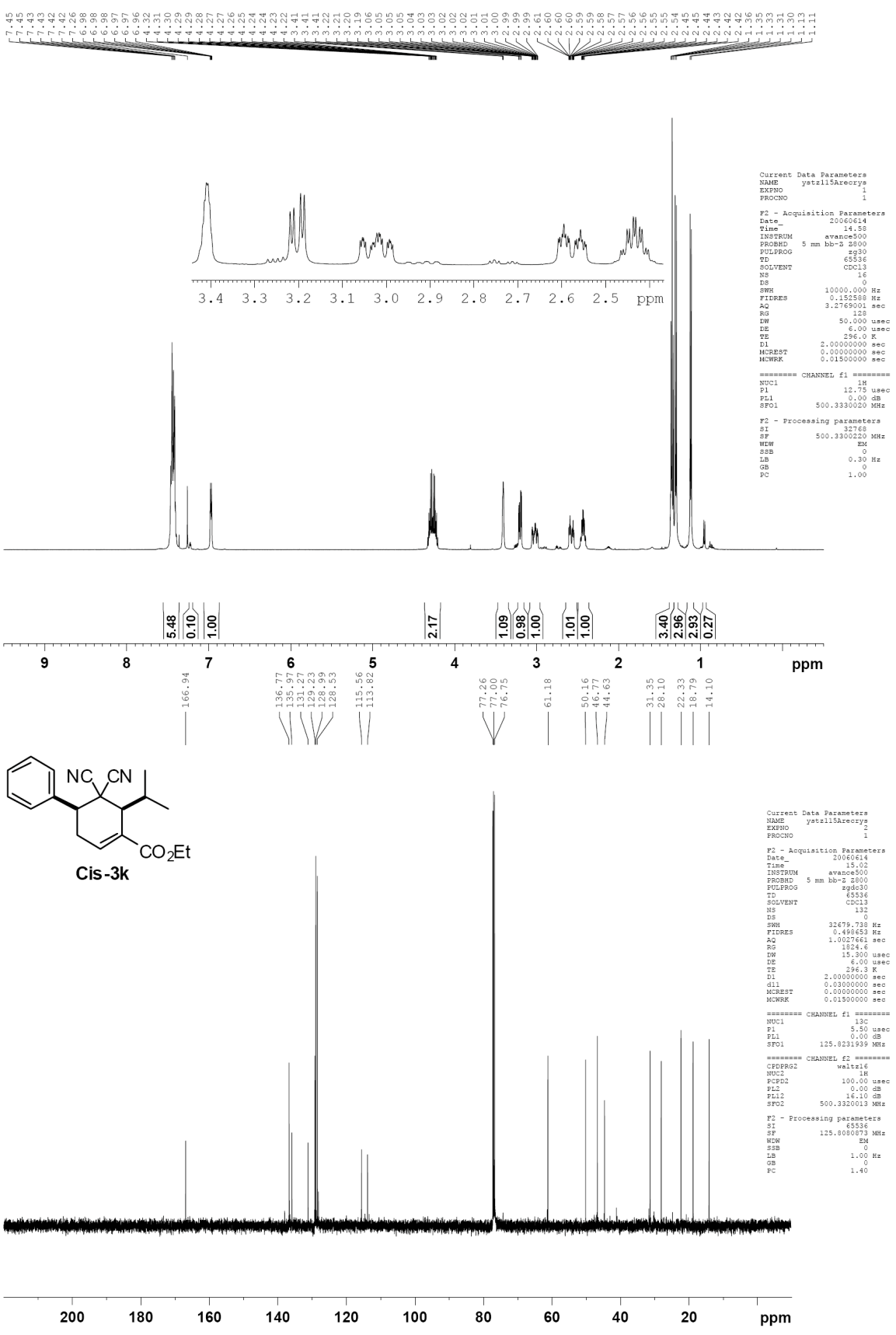
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

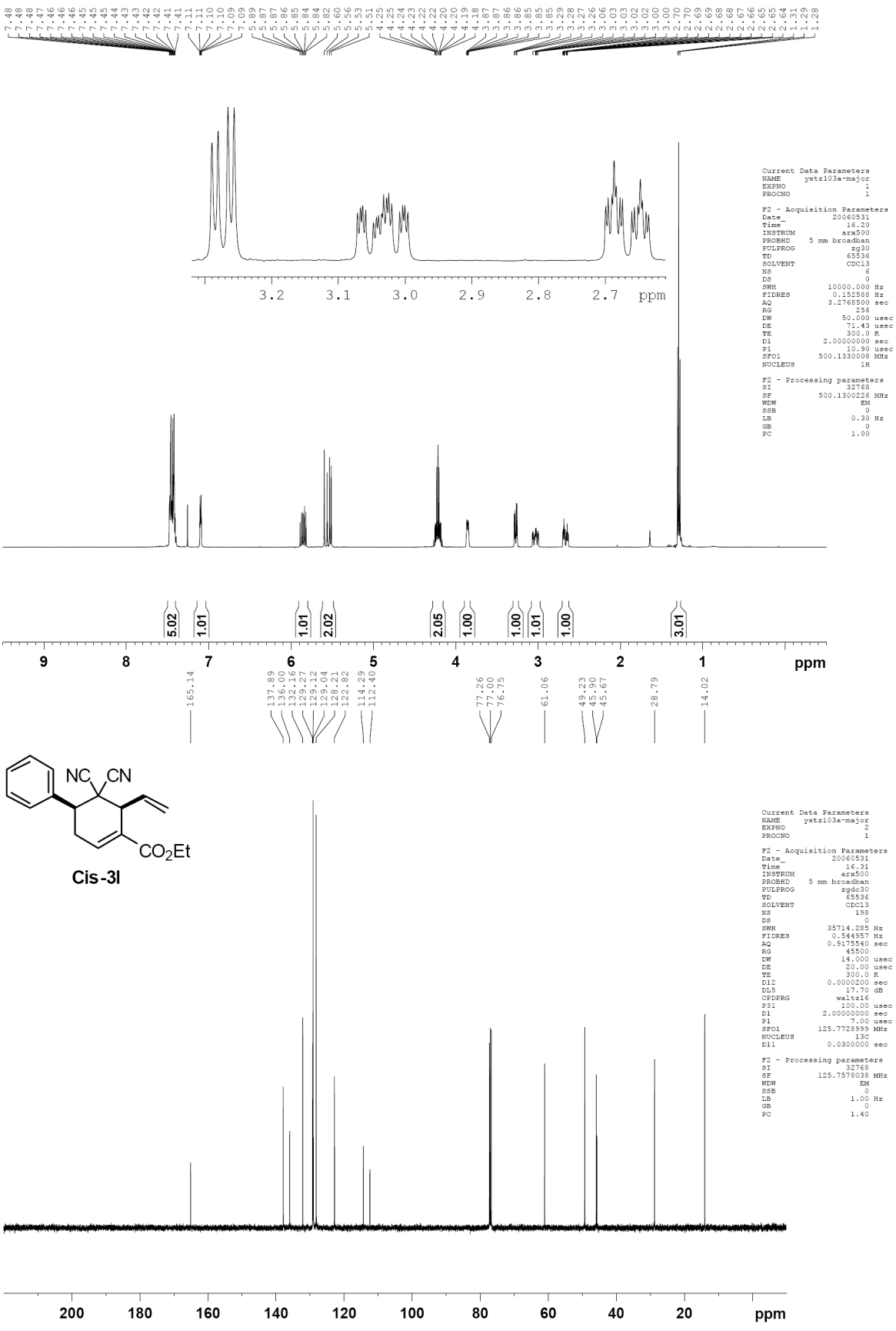
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

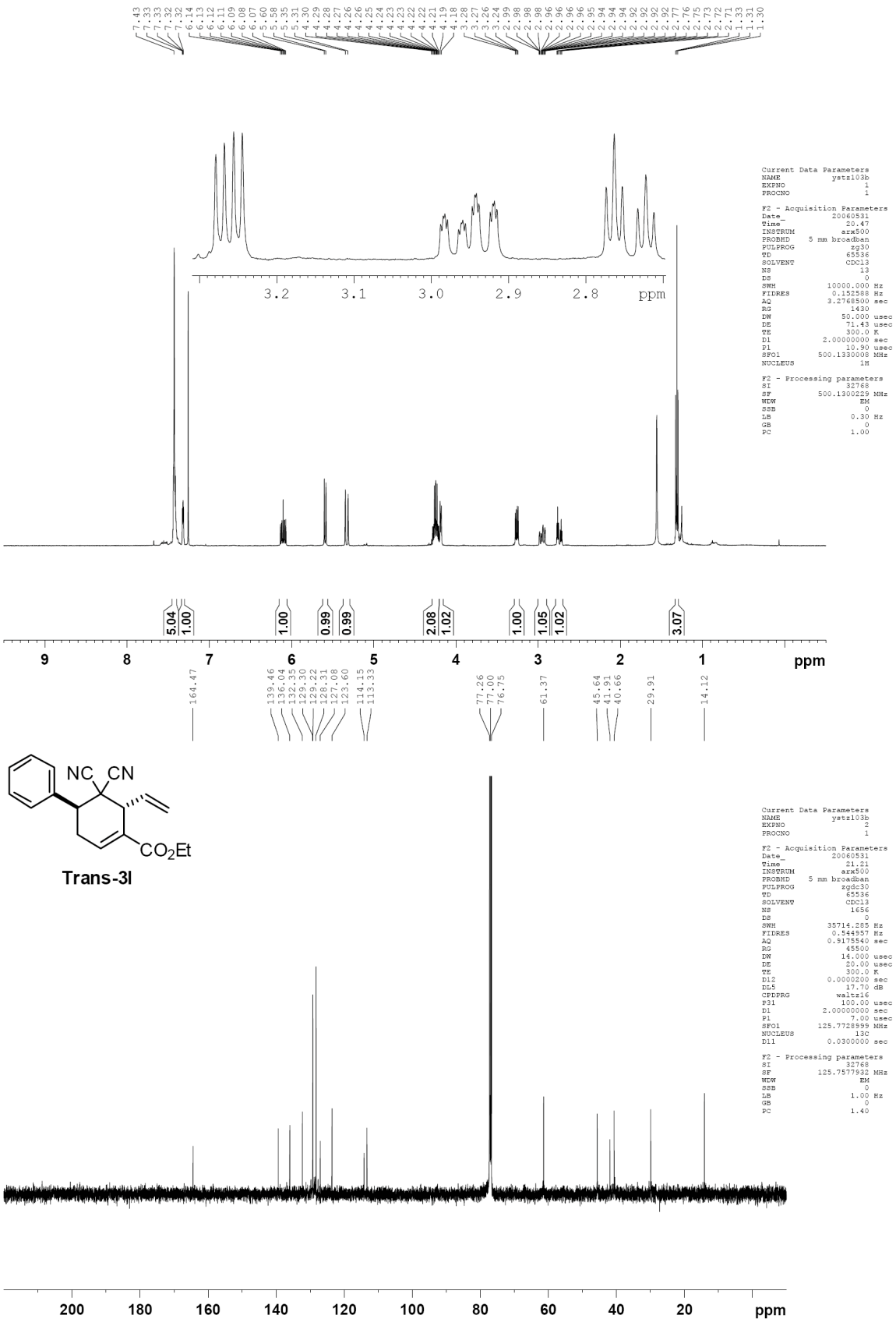
F2 - Processing parameters
SI 65536
SF 125.8081123 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

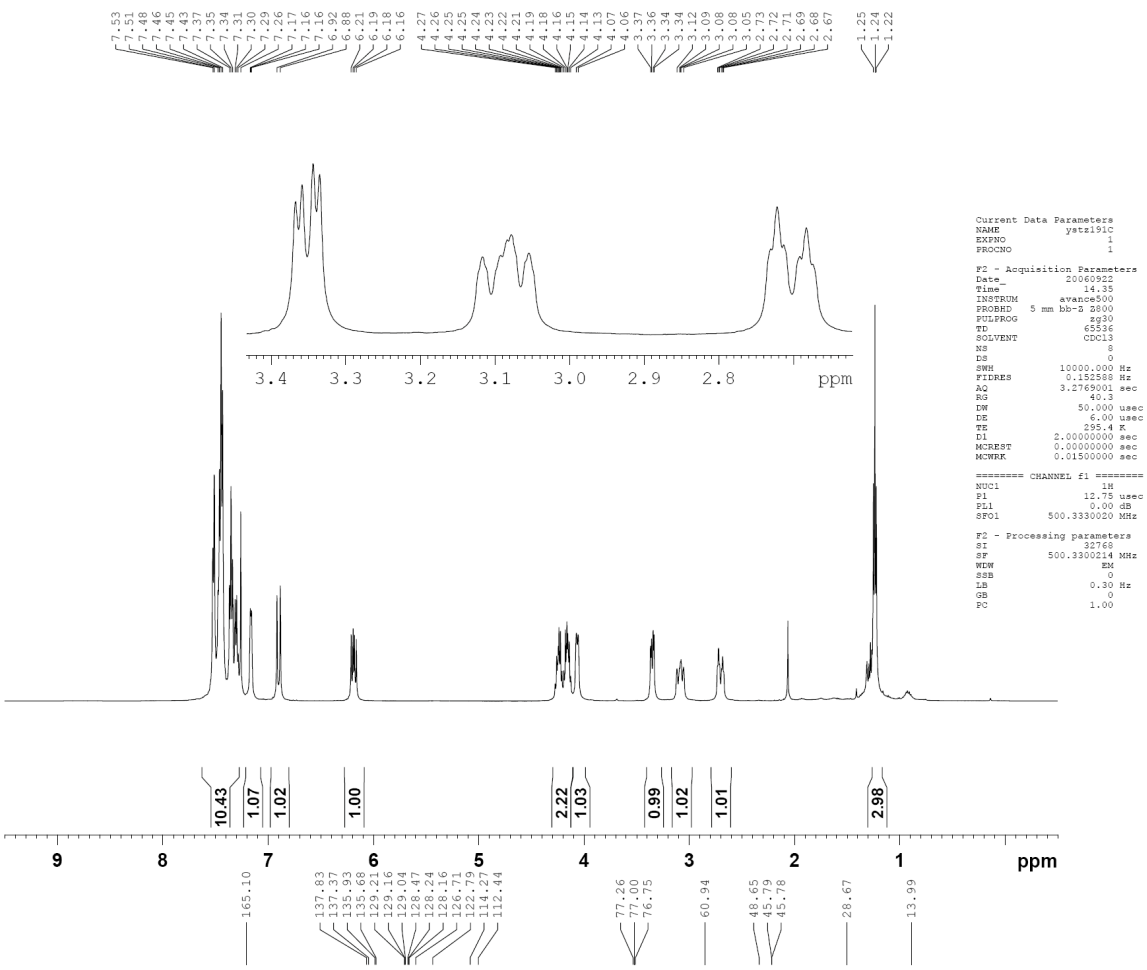

NC
CN
CO2Et
Cis-3i
ppm

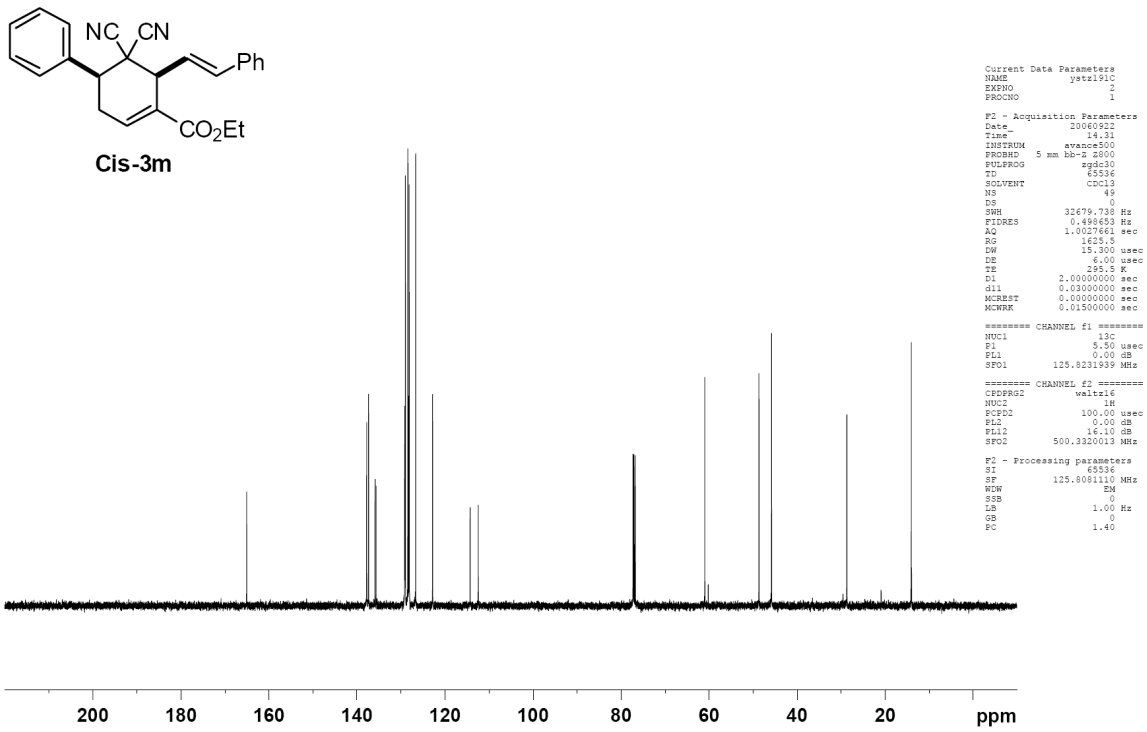

7.45 7.44 7.26 7.01 7.00 6.99 6.99 6.98 4.30 4.28 4.25 4.23 3.22 3.21 3.18 3.17 3.06 3.05 3.05 3.04 3.02 3.01 3.01 3.00 3.00 2.99 2.98 2.98 2.96 2.95 2.94 2.94 2.64 2.63 2.62 2.62 2.61 2.60 2.58 2.57 2.57 2.56 2.55 2.54 2.54 2.18 2.17 2.16 2.14 2.13 2.12 2.11 2.09 2.08 2.07 2.06 1.99 1.96 1.94 1.93 1.91 1.91 1.89 1.88 1.86 1.83 1.37 1.36 1.35 1.34 1.33 1.31 1.30 1.27 1.25 1.22
3.2 3.0 2.8 2.6 2.4 2.2 2.0 ppm
Current Data Parameters
NAME ystz58A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060414
Time 14.54
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 456.1
DW 83.400 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 65536
SF 300.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
5.43 0.09 1.00 2.16 1.02 1.00 1.03 1.00 1.08 1.07 3.48 3.89
9 8 7 6 5 4 3 2 1 ppm
165.94 136.69 136.02 131.52 129.27 129.05 128.52 115.48 112.99 77.25 77.00 76.75 61.16 46.85 46.16 43.99 28.34 25.27 14.15 12.33
NC CN
CO2Et
Cis-3j
Current Data Parameters
NAME ystb304aac
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070408
Time 20.30
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1216
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1149.4
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080863 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

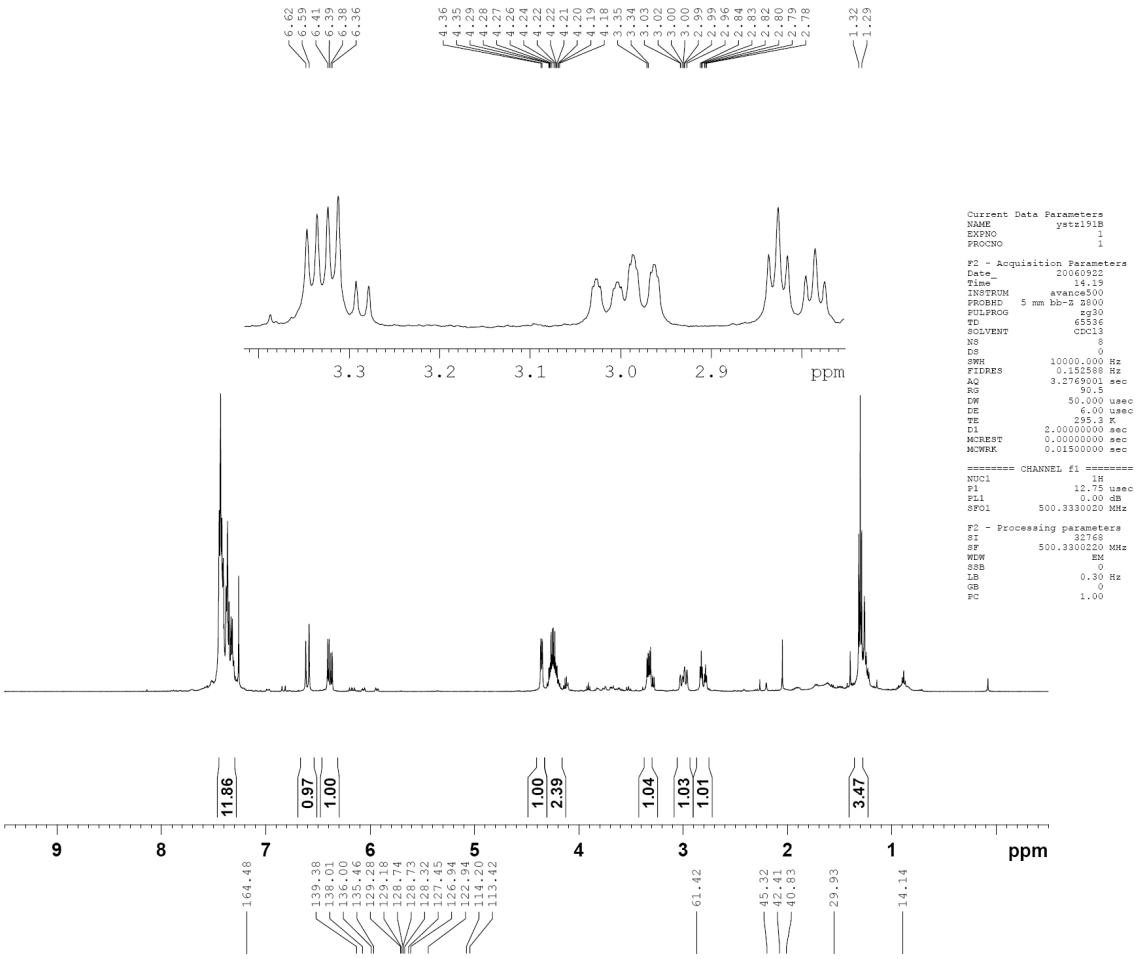

NC
CN
CO2Et
Cis-3k
ppm

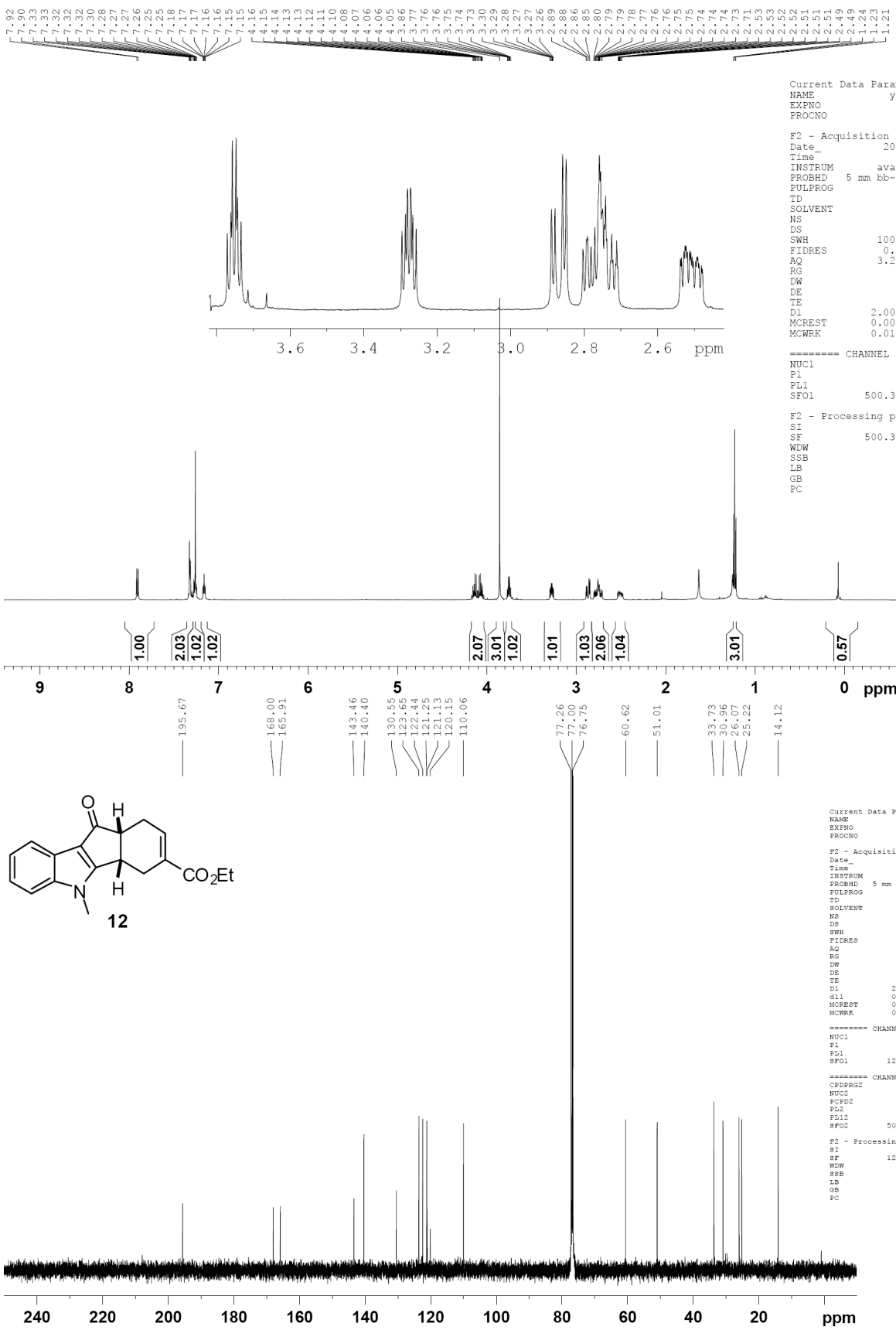

NC
CN
CO2Et
Cis-3l
Current Data Parameters
NAME ystz103a-major
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060531
Time 16.20
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 256
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 10.90 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 500.1300226 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Current Data Parameters
NAME ystz103a-major
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060531
Time 16.31
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 198
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.00002000 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 7.00 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 - Processing parameters
SI 32768
SF 125.7578038 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.43 7.33 7.33 7.32 7.32 6.14 6.13 6.12 6.11 6.09 6.08 6.07 5.60 5.58 5.35 5.31 4.30 4.29 4.28 4.27 4.26 4.26 4.25 4.24 4.23 4.23 4.22 4.22 4.21 4.19 4.18 3.28 3.27 3.26 3.24 3.09 2.98 2.98 2.96 2.96 2.96 2.96 2.95 2.94 2.94 2.94 2.92 2.92 2.92 2.92 2.77 2.76 2.75 2.73 2.72 2.71 1.33 1.31 1.30
3.2 3.1 3.0 2.9 2.8 ppm
Current Data Parameters
NAME ystz103b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060531
Time 20.47
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1430
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 10.90 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 500.1300229 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.04 1.00 1.00 0.99 0.99 2.08 1.02 1.00 1.05 1.02 3.07
9 8 7 6 5 4 3 2 1 ppm
164.47 139.46 136.04 132.35 129.30 129.22 128.31 127.08 123.60 114.15 113.33 77.26 77.00 76.75 61.37 45.64 41.91 40.66 29.91 14.12
NC CN
CO2Et
Trans-3l
Current Data Parameters
NAME ystz103b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060531
Time 21.21
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1656
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.00000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 7.00 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.03000000 sec
F2 - Processing parameters
SI 32768
SF 125.7577832 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


7.53 7.51 7.48 7.46 7.45 7.43 7.37 7.35 7.34 7.31 7.30 7.29 7.26 7.17 7.16 7.16 6.92 6.88 6.21 6.19 6.18 6.16
4.27 4.26 4.25 4.25 4.24 4.23 4.22 4.21 4.19 4.18 4.16 4.15 4.14 4.13 4.07 4.06 3.37 3.36 3.34 3.34 3.12 3.09 3.08 3.08 3.05 2.73 2.72 2.71 2.69 2.68 2.67
1.25 1.24 1.22
3.4 3.3 3.2 3.1 3.0 2.9 2.8 ppm
Current Data Parameters
NAME ystz191C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060922
Time 14.35
INSTRUM avance500
PROBHD 5 mm bb-Z 3800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 40.3
DW 50.000 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300214 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.43 1.07 1.02 1.00 2.22 1.03 0.99 1.02 1.01 2.98
9 8 7 6 5 4 3 2 1 ppm


165.10
137.83 137.37 135.93 135.68 129.21 129.16 129.04 128.47 128.24 128.16 126.71 122.79 114.27 112.44
77.26 77.00 76.75
60.94
48.65 45.79 45.78
28.67
13.99
NC CN
Ph
CO2Et
Cis-3m
Current Data Parameters
NAME ystz191C
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060922
Time 14.31
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 49
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1625.5
DW 15.300 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8081110 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

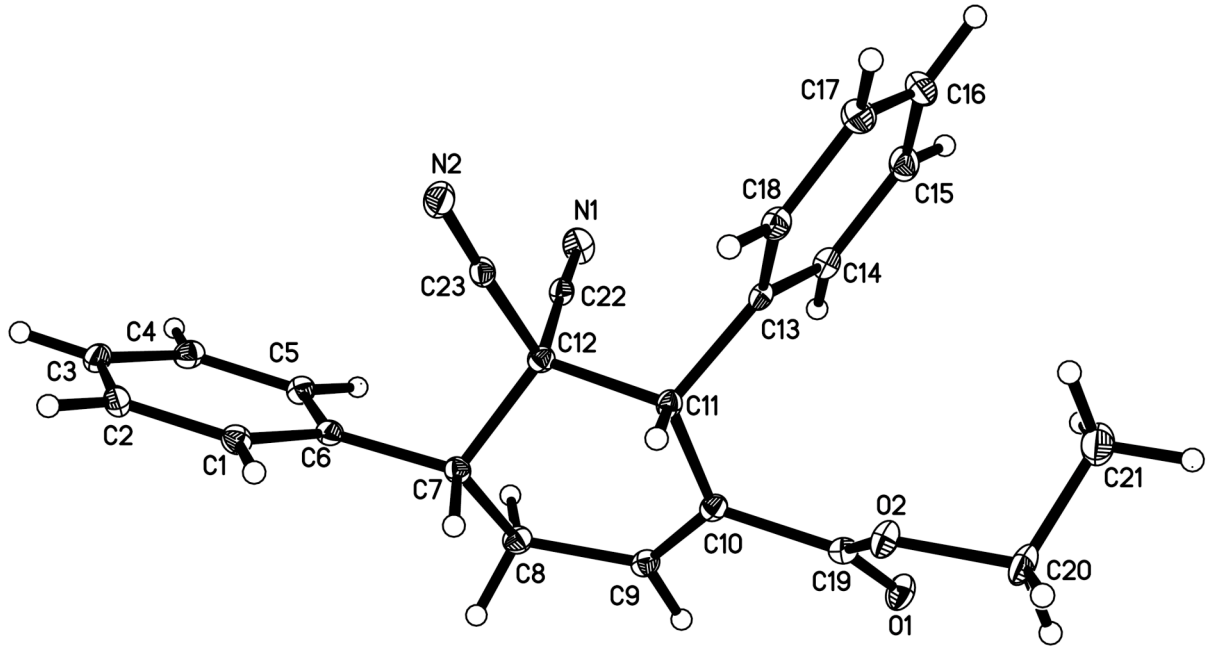

6.62
6.59
6.41
6.38
6.36
4.36
4.35
4.29
4.28
4.27
4.26
4.24
4.22
4.22
4.21
4.20
4.19
4.18
3.35
3.34
3.03
3.02
3.00
3.00
2.99
2.99
2.96
2.84
2.83
2.82
2.80
2.79
2.78
1.32
1.29
3.3
3.2
3.1
3.0
2.9
ppm
Current Data Parameters
NAME ystz191B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060922
Time 14.19
INSTRUM avance500
PROBHD 5 mm bb-2 Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.75 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11.86
0.97
1.00
1.00
2.39
1.04
1.03
1.01
3.47
9
8
7
6
5
4
3
2
1
ppm
164.48
139.38
138.01
136.00
135.46
129.28
129.18
128.74
128.73
128.32
127.45
126.94
122.94
114.20
113.42
61.42
45.32
42.41
42.03
29.93
14.14
NC
CN
Ph
CO2Et
Trans-3m
Current Data Parameters
NAME ystz191B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060922
Time 14.22
INSTRUM avance500
PROBHD 5 mm bb-2 Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 177
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2298.8
DW 15.300 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080877 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm


7.92 7.90 7.33 7.33 7.32 7.32 7.32 7.30 7.28 7.27 7.27 7.26 7.25 7.25 7.18 7.17 7.17 7.16 7.16 7.15 7.15 4.16 4.15 4.14 4.13 4.12 4.11 4.10 4.08 4.07 4.06 4.06 4.05 3.86 3.77 3.76 3.76 3.75 3.74 3.73 3.30 3.29 3.28 3.27 3.27 3.26 2.89 2.88 2.86 2.85 2.80 2.79 2.78 2.77 2.76 2.76 2.75 2.74 2.74 2.74 2.73 2.71 2.53 2.53 2.52 2.52 2.51 2.51 2.51 2.49 2.49 1.24 1.23 1.21
3.6 3.4 3.2 3.0 2.8 2.6 ppm
Current Data Parameters
NAME ystb404
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070627
Time_ 19.30
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 use
DE 6.00 use
TE 295.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =======
NUC1 1H
P1 12.75 use
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00 2.03 1.02 1.02 2.07 3.01 1.02 1.01 1.03 2.06 1.04 3.01 0.57
9 8 7 6 5 4 3 2 1 0 ppm
195.67 168.00 165.91 143.46 140.40 130.55 123.65 122.44 121.25 121.13 120.15 110.06 77.26 77.00 76.75 60.62 51.01 33.73 30.96 26.07 25.22 14.12
O H N H CO2Et
12
Current Data Parameters
NAME ystb404
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070627
Time_ 19.49
INSTRUM avance500
PROBHD 5 mm bb-Z 2800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 553
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2896.3
DW 15.300 use
DE 6.00 use
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =======
NUC1 13C
P1 5.50 use
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080855 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
240 220 200 180 160 140 120 100 80 60 40 20 ppm

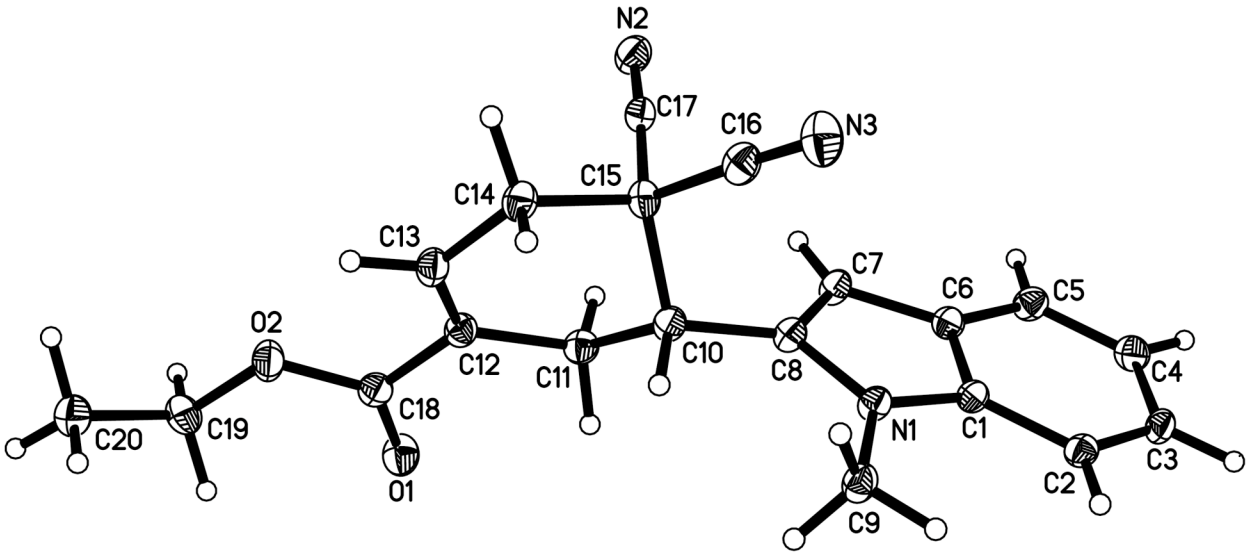
8. ORTEP Representations of the Solid State Structures of Compounds 3d, 4f, and 12

Crystallographic data for **3d**, **4f**, and **12** have been deposited with the Cambridge Crystallographic Data Centre as supplementary numbers CCDC-653533, -653534, and -653535. These data can be obtained online free of charge [or from the Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk].

A. Cyclohexene 3d



B. Cyclohexene 4f



C. Tetracycle 12