Electronic Supplementary Material (ESI) for RSC Advances

Supporting Information

An Efficient Pyrroline Annulation of Glycine Imine with Enones

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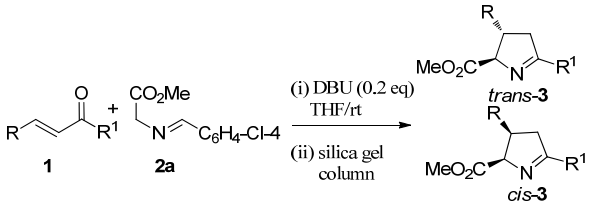
I. General information

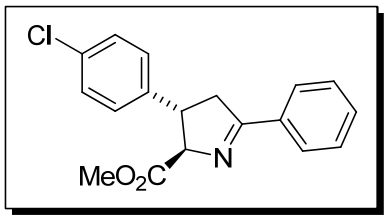
All reagents were purchased from commercial sources and used without further purification, unless otherwise indicated. All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F254). The products were purified by column chromatography on flash silica gel (300−400 mesh). Melting points were uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were determined on a Varian 500 MHz and 125 MHz, respectively, with TMS as the internal standard. All shifts are given in ppm. IR spectra (KBr) were recorded on a Magna-560 FTIR spectrophotometer in the range of 400~4000 cm^{-1}. High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI).

II. Synthetic procedures and analytical data of **3**, **4**, **7** and **9**

General procedure (**3a** as an example): To the mixture of enone **1a** (242 mg, 1.0 mmol) and azomethine ylide **2a** (253 mg, 1.2 mmol) in THF (5 mL) was added DBU (0.2 mmol) in one portion at room temperature. After **1a** was consumed as indicated by TLC, the resulting mixture was poured into ice brine (30 mL) under stirring. The precipitates were collected by filtration, washed with water (10 mL × 3), and dried in vacuo. The crude product was purified by flash column chromatography (silica gel, petroleum ether: EtOAc = 10 : 1, V/V) to give **3a** (289 mg, 92%). The *trans*/*cis* configuration of **3** was calculated on the integration of the signal at 4.1-5.4 ppm in ^{1}H NMR spectra of the diastereomeric mixture based on *cis*-**3d** (For details, see Part III).



3a, *trans*:*cis* = 1.5:1.0



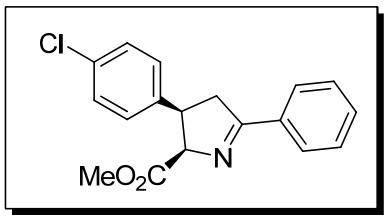
trans-**3a**: yellowish crystals, m.p. 107–109 °C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.10 (dd, *J* = 17.5, 7.0 Hz, 1H), 3.63 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.79 (s, 3H, OCH_3), 3.86 (m, 1H), 4.89 (d, *J* = 6.0 Hz, 1H), 7.16 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 7.40–7.48 (m, 3H), 7.90 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.4, 45.7, 52.4, 82.3, 128.1, 128.4, 128.5, 128.9, 131.3, 132.5, 133.4, 141.5, 172.3, 174.7.

IR (KBr, cm^{-1}): 3061, 2957, 2842, 1733, 1690, 1343.

HRMS (ESI-TOF) Calcd for $C_{18}H_{17}ClNO_2^+$ ($[M+H]^+$) 314.0942. Found 314.0916.



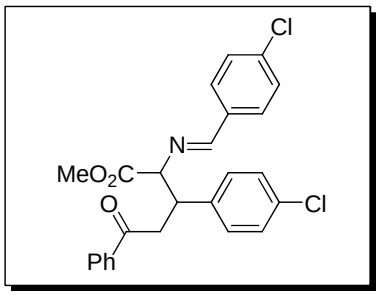
cis-**3a**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.34 (s, 3H, OCH_3), 3.40 (m, 2H), 3.99 (m, 1H), 5.23 (d, *J* = 8.5 Hz, 1H), 7.10 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.44–7.52 (m, 3H), 7.95 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 42.0, 45.8, 51.4, 79.1, 128.0, 128.3, 128.5, 128.9, 131.2, 132.7, 133.4, 138.3, 170.5, 176.0.

IR (KBr, cm^{-1}): 3061, 2951, 2845, 1739, 1684, 1344.

HRMS (ESI-TOF) Calcd for $C_{18}H_{17}ClNO_2^+$ ($[M+H]^+$) 314.0942. Found 314.0916.



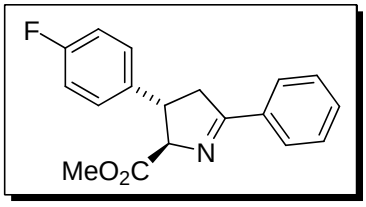
4a: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.45–3.50 (m, 1H), 3.64 (s, 3H, OCH_3), 4.23 (m, 2H), 7.17–7.22 (m, 4H), 7.39–7.44 (m, 4H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.69 (d, *J* = 8.5 Hz, 2H), 7.90 (d, *J* = 7.5 Hz, 2H), 8.07 (s, 1H).

IR (KBr, cm^{-1}): 3059, 3027, 2951, 1735, 1686, 1349.

HRMS (ESI-TOF) Calcd for $C_{25}H_{22}ClNO_3^+$ ($[M+H]^+$) 454.0971. Found 454.0977.

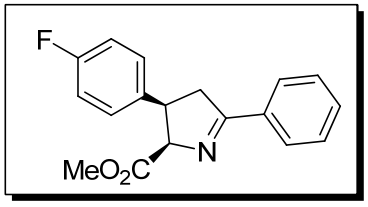
3b, *trans*:*cis* = 2.4:1.0



trans-**3b**: yellowish viscous oil

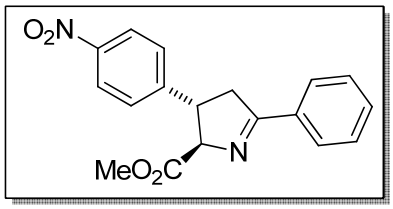
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.12 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.65 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.78 (s, 3H, OCH_3), 3.88 (m, 1H), 4.90 (d, *J* = 6.0 Hz, 1H), 7.00 (m, 2H), 7.19 (m, 2H), 7.41–7.49 (m, 3H), 7.91 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.7, 45.6, 52.4, 82.6, 115.7(d), 128.1, 128.4(d), 128.5, 131.3, 133.4, 138.8 (d), 161.7(d), 172.4, 174.9.
IR (KBr, cm^{-1}): 3060, 2952, 2846, 1740, 1684, 1345.
HRMS (ESI-TOF) Calcd for $C_{18}H_{17}FNO_2^+$ ($[M+H]^+$) 298.1238. Found 298.1247.

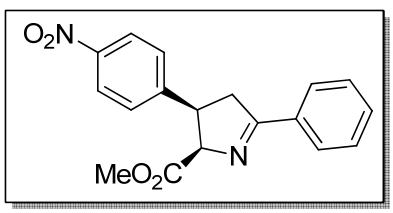


cis-**3b**: yellowish crystals, m.p. 85–87 °C
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.33 (s, 3H, OCH_3), 3.39 (m, 2H), 3.99 (m, 1H), 5.22 (d, *J* = 8.5 Hz, 1H), 6.95 (m, 2H), 7.13 (m, 2H), 7.44–7.50 (m, 3H), 7.96 (d, *J* = 8.0 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 42.2, 45.8, 51.4, 79.2, 115.1(d), 128.0, 128.5, 129.1(d), 131.3, 133.5, 135.5(d), 161.8(d), 170.7, 176.2.
IR (KBr, cm^{-1}): 3072, 2947, 2837, 1747, 1621, 1355.
HRMS (ESI-TOF) Calcd for $C_{18}H_{17}FNO_2^+$ ($[M+H]^+$) 298.1238. Found 298.1247.

3c, *trans*:*cis* = 1.3:1.0



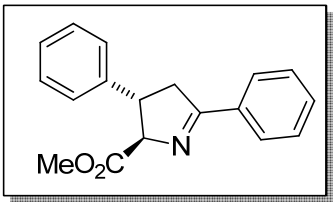
trans-**3c**: yellowish viscous oil
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.18 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.73 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.81 (s, 3H, OCH_3), 4.02–4.04 (m, 1H), 4.96 (d, *J* = 5.5 Hz, 1H), 7.41–7.47 (m, 4H), 7.50 (d, *J* = 7.0 Hz, 1H), 7.92 (d, *J* = 7.0 Hz, 2H), 8.19 (d, *J* = 8.5 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.5, 46.0, 52.7, 82.3, 124.2, 128.0, 128.1, 128.6, 131.5, 133.1, 147.0, 150.6, 171.9, 174.6.
IR (KBr, cm^{-1}): 3077, 2953, 2850, 1739, 1604, 1346.
HRMS (ESI-TOF) Calcd for $C_{18}H_{17}N_2O_4^+$ ($[M+H]^+$) 325.1183. Found 325.1185.



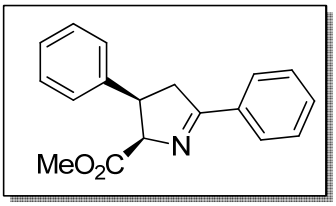
cis-**3c**: yellowish crystals, m.p. 118–120 °C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.34 (s, 3H, OCH_3), 3.45(dd, *J* = 17.5, 5.5 Hz, 1H), 3.50 (dd, *J* = 17.5, 8.5 Hz, 1H), 4.12 (m, 1H), 5.30 (d, *J* = 8.5 Hz, 1H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.51 (d, *J* = 7.0 Hz, 1H), 7.96 (d, *J* = 7.0 Hz, 2H), 8.13 (d, *J* = 9.0 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 42.1, 46.3, 51.7, 79.1, 123.6, 128.1, 128.5, 128.7, 131.6, 133.2, 147.0, 147.7, 170.3, 175.9.
IR (KBr, cm^{-1}): 3054, 2950, 2842, 1743, 1673, 1346.
HRMS (ESI-TOF) Calcd for $C_{18}H_{17}N_2O_4^+$ ($[M+H]^+$) 325.1183. Found 325.1185.

3d, *trans*:*cis* = 1.4:1.0

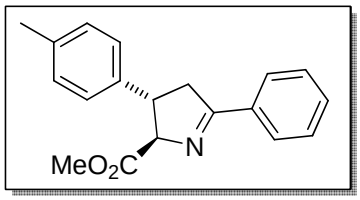


trans-**3d**: yellowish viscous oil
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.15 (dd, *J* = 17.5, 7.0 Hz, 1H), 3.65 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.77 (s, 3H, OCH_3), 3.87–3.90 (m, 1H), 4.96 (d, *J* = 6.0 Hz, 1H), 7.22–7.24 (m, 3H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.40–7.47 (m, 3H), 7.91 (d, *J* = 7.0 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.5, 46.1, 52.2 , 82.4, 126.7, 126.8, 128.0, 128.4, 128.7, 131.0, 133.4, 143.0, 172.4, 174.8.
IR (KBr, cm^{-1}): 3061, 2951, 2844, 1740, 1614, 1343.
HRMS (ESI-TOF) Calcd for $C_{18}H_{18}NO_2^+$ ($[M+H]^+$) 280.1332. Found 280.1319.



cis-**3d**: yellowish crystals, m.p. 106–108 °C
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.28 (s, 3H, OCH_3), 3.41–3.43 (m, 2H), 4.00 (m, 1H), 5.24 (d, *J* = 8.5 Hz, 1H), 7.18 (d, *J* = 7.0 Hz, 2H), 7.23 (d, *J* = 7.0 Hz, 1H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.46–7.50 (m, 3H), 7.96 (d, *J* = 7.0 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 41.9, 46.5, 51.3, 79.3, 127.0, 127.5, 128.0, 128.2, 128.5, 131.1, 133.6, 139.7, 170.7, 176.3.
IR (KBr, cm^{-1}): 3062, 2921, 2852, 1740, 1677, 1346.
HRMS (ESI-TOF)Calcd for $C_{18}H_{18}NO_2^+$ ($[M+H]^+$) 280.1332. Found 280.1319.

3e, *trans*:*cis* = 1.6:1.0



trans-**3e**: yellowish viscous oil

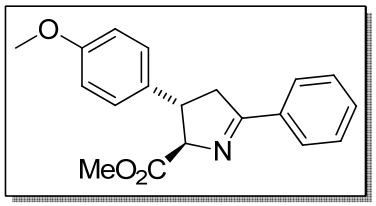
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.33 (s, 3H, CH_3), 3.13 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.63 (dd, *J* = 17.5, 10 Hz, 1H), 3.78 (s, 3H, OCH_3), 3.83–3.87 (m, 1H), 4.93 (d, *J* = 6.0 Hz, 1H), 7.13 (s, 4H), 7.42–7.48(m, 3H), 7.90 (d, *J* = 7.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.0, 44.7, 46.0, 52.4, 82.6, 126.9, 128.1, 128.5, 129.5, 133.2, 133.6, 136.5, 140.1, 172.7, 175.1.

IR (KBr, cm^{-1}): 3055, 2924, 2854, 1744, 1614, 1344.

HRMS (ESI-TOF) Calcd for $C_{19}H_{20}NO_2^+$ ($[M+H]^+$) 294.1489. Found 294.1490.

3f, *trans*:*cis* = 1.4:1.0



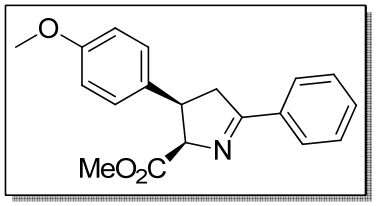
trans-**3f**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.10 (dd, *J* = 17.5, 7.5 Hz, 1H), 3.62 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.76 (s, 3H), 3.82–3.86 (m, 1H), 4.90 (d, *J* = 6.5 Hz, 1H), 6.84 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 7.40–7.46 (m, 3H), 7.89–7.91 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.6, 45.6, 52.4, 55.2, 82.6, 114.2, 128.0, 128.1, 128.5, 131.2, 133.6, 135.0, 158.5, 172.7, 175.1.

IR (KBr, cm^{-1}): 3001, 2952, 2838, 1740, 1613, 1345.

HRMS (ESI-TOF) Calcd for $C_{19}H_{20}NO_3^+$ ($[M+H]^+$) 310.1438. Found 310.1427.



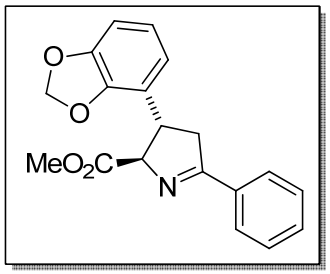
cis-**3f**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.33 (s, 3H), 3.39 (dd, *J* = 7.5, 1.5 Hz, 2H), 3.76 (s, 3H), 3.95–3.99 (m, 1H), 5.19 (d, *J* = 9.0 Hz, 1H), 6.79 (d, *J* = 8.5 Hz, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 7.44–7.49 (m, 3H), 7.95–7.97 (d, *J* = 9.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 42.2, 45.8, 51.5, 55.2, 79.3, 113.6, 128.1, 128.5, 128.6, 131.2, 131.7, 133.7, 158.5, 170.9, 176.4.

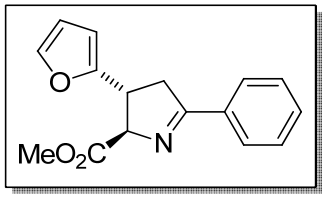
IR (KBr, cm^{-1}): 3059, 2951, 2837, 1739, 1681, 1435.
HRMS (ESI-TOF) Calcd for $C_{19}H_{20}NO_3^+$ ($[M+H]^+$) 310.1438. Found 310.1427.

3g, *trans*:*cis* = 1.3:1.0

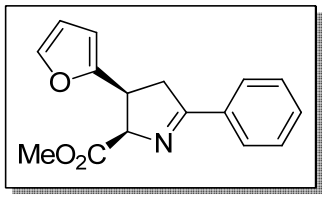


trans-**3g**: yellowish viscous oil
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.12 (dd, *J* = 17.0, 6.5 Hz, 1H), 3.60–3.66 (dd, *J* = 17.0, 9.5 Hz, 1H), 3.79 (s, 3H), 3.80–3.84 (m, 1H), 4.89 (d, *J* = 6.0 Hz, 1H), 5.93 (s, 2H), 6.69 (m, 2H), 6.75 (d, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.47 (d, *J* = 7.0 Hz, 1H), 7.90 (d, *J* = 7.5 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.7, 46.1, 52.4, 82.6, 101.0, 107.1, 108.4, 120.1, 128.0, 128.5, 131.2, 133.5, 136.9, 146.4, 148.0, 172.5, 175.0.
IR (KBr, cm^{-1}): 3060, 2952, 2900, 1733, 1615, 1341.
HRMS (ESI-TOF) Calcd for $C_{19}H_{18}NO_4^+$ ($[M+H]^+$) 324.1230. Found 324.1237.

3h, *trans*:*cis* = 1.2:1.0



trans-**3h**: yellowish viscous oil
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.33 (dd, *J* = 16.5, 9.0 Hz, 1H), 3.42 (dd, *J* = 16.5, 8.0 Hz, 1H), 3.49 (s, 3H), 4.05 (m, 1H), 5.19 (d, *J* = 8.5 Hz, 1H), 6.13 (d, *J* = 3.0 Hz, 1H), 6.29 (dd, *J* = 3.0, 2.0 Hz, 1H), 7.31 (d, *J* = 2.0 Hz, 1H), 7.48 (m, 3H), 7.92 (d, *J* = 8.0 Hz, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz) δ 39.5, 41.3, 52.3, 79.2, 105.4, 110.2, 127.9, 128.3, 131.0, 133.3, 141.7, 154.6, 172.1, 174.5.
IR (KBr, cm^{-1}): 3064, 2958, 2888, 1750, 1674, 1355.
HRMS (ESI-TOF) Calcd for $C_{16}H_{16}NO_3^+$ ($[M+H]^+$) 270.1125. Found 270.1136.



cis-**3h**: yellowish crystals, m.p. 101–103 °C

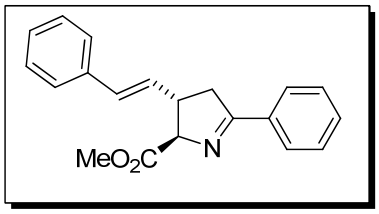
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.25 (dd, *J* = 17.0, 7.0 Hz, 1H), 3.54 (dd, *J* = 17.0, 9.5 Hz, 1H), 3.80 (s, 3H), 3.99 (m, 1H), 5.00 (d, *J* = 6.5 Hz ,1H), 6.14 (d, *J* = 3.0 Hz, 1H), 6.30 (dd, *J* = 3.0, 2.0 Hz, 1H), 7.34 (d, *J* = 2.0 Hz, 1H), 7.40–7.46 (m, 3H), 7.88 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 39.8, 40.3, 51.9, 77.4, 106.3, 110.2, 128.0, 128.5, 131.2, 133.5, 141.7, 152.8, 170.5, 176.1.

IR (KBr, cm^{-1}): 3063, 2956, 2873, 1740, 1615, 1351.

HRMS (ESI-TOF) Calcd for $C_{16}H_{16}NO_3^+$ ($[M+H]^+$) 270.1125. Found 270.1136.

3i, *trans*:*cis* = 1.1:1.0



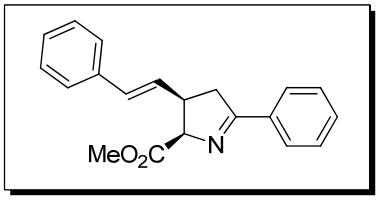
trans-**3i**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.93 (dd, *J* = 16.5, 7.0 Hz, 1H), 3.43 (dd, *J* = 16.5, 9.0 Hz, 1H), 3.48 (m, 1H), 3.79 (s, 3H), 4.71 (d, *J* = 6.0 Hz, 1H), 6.24 (dd, *J* = 15.5, 7.5 Hz, 1H), 6.52 (d, *J* = 15.5 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.39–7.46 (m, 3H), 7.88 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 42.3, 45.0, 52.4, 79.9, 126.2, 127.6, 128.0, 128.5, 128.6, 130.1, 131.2, 131.4, 133.6, 136.7, 172.5, 175.2.

IR (KBr, cm^{-1}): 3058, 2924, 2854, 1743, 1650, 1344.

HRMS (ESI-TOF) Calcd for $C_{20}H_{20}NO_2^+$ ($[M+H]^+$) 306.1489. Found 306.1480.



cis-**3i**: yellowish viscous oil

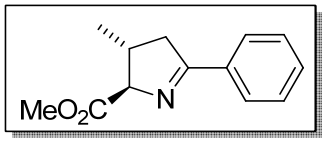
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.14 (dd, *J* = 16.5, 6.5 Hz, 1H), 3.25 (dd, *J* = 16.5, 8.5 Hz, 1H), 3.54–3.57 (m, 1H), 3.66 (s, 3H), 5.07 (d, *J* = 8.0 Hz, 1H), 6.12 (dd, *J* = 15.5, 9.0 Hz, 1H), 6.52 (d, *J* = 15.5 Hz, 1H), 7.23 (t, *J* = 7.0 Hz, 1H), 7.29–7.34 (m, 4H), 7.42–7.49 (m, 3H), 7.92 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 41.4, 44.5, 51.9, 78.0, 126.2, 127.5, 128.0, 128.4, 128.5, 130.9, 131.2, 132.3, 133.5, 136.7, 171.0, 176.3.

IR (KBr, cm^{-1}): 3058, 2951, 2844, 1739, 1652, 1345.

HRMS (ESI-TOF) Calcd for $C_{20}H_{20}NO_2^+$ ($[M+H]^+$) 306.1489. Found 306.1480.

3j, *trans*:*cis* = 1.1:1.0



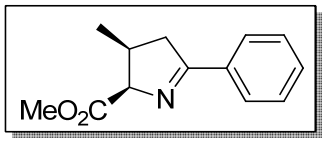
trans-**3j:** yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.25 (d, *J* = 7.0 Hz, 3H), 2.61 (dd, *J* = 16.5, 6.5 Hz, 1H), 2.67–2.73 (m, 1H), 3.32 (dd, *J* = 16.5, 8.5 Hz, 1H), 3.77 (s, 3H), 4.46 (d, *J* = 6.0 Hz, 1H), 7.38–7.46(m, 3H), 7.86 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.8, 36.0, 43.4, 52.0, 81.1, 127.8, 128.3, 130.8, 133.7, 173.0, 175.4.

IR (KBr, cm^{-1}): 3056, 2952, 2843, 1738, 1652, 1344.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}NO_2^+$ ($[M+H]^+$) 218.1176. Found 218.1167.



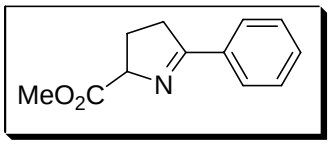
cis-**3j**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.04 (d, *J* = 7.0 Hz, 3H), 2.61 (dd, *J* = 16.5, 6.0 Hz, 1H), 2.87–2.89 (m, 1H), 2.61 (dd, *J* = 16.5, 8.5 Hz, 1H), 3.76 (s, 3H), 4.91 (d, *J* = 8.0 Hz, 1H), 7.40–7.46(m, 3H), 7.86 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 15.8, 34.9, 43.0, 51.7, 77.4, 127.8, 128.4, 130.9, 133.9, 171.7, 176.2.

IR (KBr, cm^{-1}): 3057, 2951, 2845, 1740, 1650, 1346.

HRMS (ESI-TOF) Calcd for $C_{13}H_{16}NO_2^+$ ($[M+H]^+$) 218.1176. Found 218.1167.



3k: yellowish viscous oil

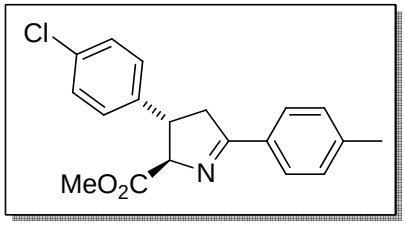
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.26 (m, 1H), 2.35 (m, 1H), 3.00 (m, 1H), 3.15 (m, 1H), 3.78 (s, 3H), 4.92 (dd, *J* = 8.5, 7.0 Hz, 1H), 7.42 (m, 3H), 7.87 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 26.3, 35.4, 52.3, 74.5, 128.0, 128.4, 131.0, 133.7, 173.4, 176.1.

IR (KBr, cm^{-1}): 3060, 2952, 2844, 1732, 1615, 1345.

HRMS (ESI-TOF) Calcd for $C_{12}H_{14}NO_2^+$ ($[M+H]^+$) 204.1019. Found 204.1020.

3l, *trans*:*cis* = 1.6:1.0



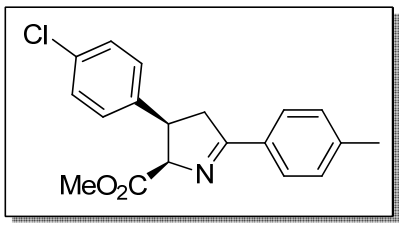
trans-**3l**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.40 (s, 3H), 3.10 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.65 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.79 (s, 3H), 3.86 (m, 1H), 4.88 (d, *J* = 6.0 Hz, 1H), 7.17 (d, *J* = 9.0 Hz, 2H), 7.24 (d, *J* = 9.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.5, 44.5, 45.7, 52.4, 82.4, 128.1, 128.4, 129.0, 129.3, 130.7, 132.7, 141.6, 141.7, 172.4, 174.7.

IR (KBr, cm^{-1}): 3087, 2999, 2923, 1733, 1660, 1410.

HRMS (ESI-TOF) Calcd for $C_{19}H_{19}ClNO_2^+$ ($[M+H]^+$) 328.1099. Found 328.1101.



cis-**3l**: yellowish viscous oil

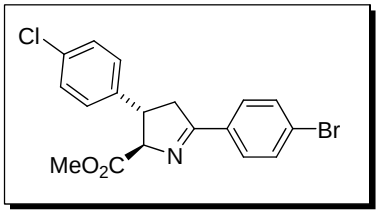
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.42 (s, 3H), 3.33 (s, 3H), 3.34–3.40 (m, 2H), 3.97 (m, 1H), 5.21 (d, *J* = 8.5 Hz, 1H), 7.10 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.84 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.5, 42.1, 45.9, 51.5, 79.1, 128.1, 128.4, 128.8, 129.0, 129.3, 130.8, 132.8, 138.5, 141.8, 170.7, 176.0.

IR (KBr, cm^{-1}): 3066, 2951, 2848, 1733, 1615, 1340.

HRMS (ESI-TOF) Calcd for $C_{19}H_{19}ClNO_2^+$ ($[M+H]^+$) 328.1099. Found 328.1101.

3m, *trans*:*cis* = 1.4:1.0



trans-**3m**: yellowish viscous oil

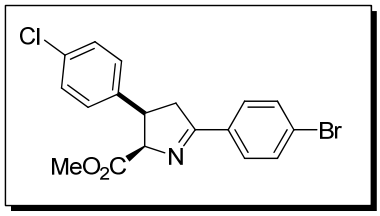
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.08 (dd, *J* = 17.0, 7.0 Hz, 1H), 3.65 (dd, *J* = 17.0, 10.0 Hz, 1H), 3.78 (s, 3H), 3.85–3.89 (m, 1H), 4.88 (d, *J* = 6.5 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 2H), 7.56 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.4, 45.7, 45.8, 52.5, 82.4, 126.0, 128.1, 129.0, 129.6, 131.8, 132.2,

132.8, 144.2, 172.1, 173.9.

IR (KBr, cm^{-1}): 3064, 2950, 2840, 1735, 1616, 1349.

HRMS (ESI-TOF) Calcd for $C_{18}H_{16}ClBrNO_2^+$ ($[M+H]^+$) 392.0047. Found 392.0048.



cis-**3m**: yellowish viscous oil

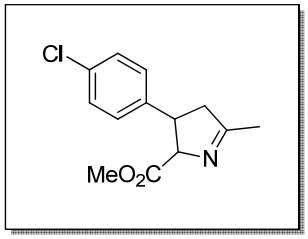
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.34 (s, 3H), 3.38 (m, 2H), 4.00 (m, 1H), 5.22 (d, *J* = 8.5 Hz, 1H), 7.09 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.83 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.5, 45.7, 52.4, 82.4, 128.1, 128.4, 129.0, 129.3, 130.7, 132.7, 141.6, 141.7, 172.5, 174.7.

IR (KBr, cm^{-1}): 3065, 2949, 2842, 1734, 1615, 1348.

HRMS (ESI-TOF) Calcd for $C_{18}H_{16}ClBrNO_2^+$ ($[M+H]^+$) 392.0047. Found 392.0048.

3n, *trans*:*cis*= 1.3:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

trans-**3n**, δ 2.16 (s, 3H), 2.68 (dd, *J* = 18.0, 7.0 Hz, 1H), 3.16 (dd, *J* =18.0, 9.5Hz, 1H), 3.71–3.75 (m, 4H), 4.65 (d, *J* = 8.5Hz, 1H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.5 Hz, 2H).

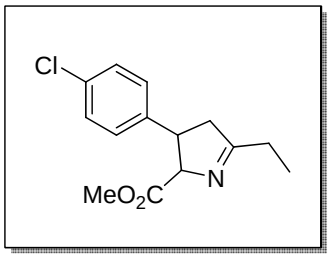
cis-**3n**, δ 2.23 (s, 3H), 2.86 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.96 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.31 (s, 3H), 3.83 (m, 1H), 4.98 (d, *J* = 8.5 Hz, 1H), 7.03 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.8, 20.1, 45.9, 46.0, 46.1, 48.2, 51.5, 52.4, 78.8, 82.0, 128.2, 128.3, 128.8, 128.9, 132.5, 132.7, 138.5, 141.3, 170.8, 172.4, 177.3, 178.7.

IR (KBr, cm^{-1}): 3060, 3030, 2955, 2874, 2842, 1739, 1682, 1613, 1347.

HRMS (ESI-TOF) Calcd for $C_{13}H_{15}ClNO_2^+$ ($[M+H]^+$) 252.0786. Found 252.0790.

3o, *trans*:*cis*= 1.7:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

trans-**3o**, δ 1.22 (t, *J* = 7.5 Hz, 3H), 2.46 (q, *J* = 7.5 Hz, 2H), 2.67 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.16 (dd, *J* = 17.5, 10.0 Hz, 1H), 3.68–3.72 (m, 1H), 3.75 (s, 3H), 4.66 (d, *J* = 6.0 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H) , 7.28 (d, *J* = 8.0 Hz, 2H).

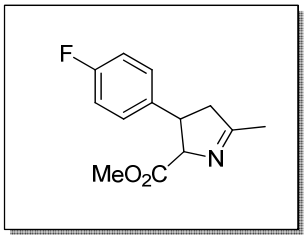
cis-**3o**, δ 1.26 (t, *J* = 7.5 Hz, 3H), 2.54 (q, *J* = 7.5 Hz, 2H), 2.90 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.97 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.33 (s, 3H), 3.77–3.86 (m, 1H), 4.99 (d, *J* = 8.5 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 2H) , 7.22 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 10.5, 10.6, 27.0, 27.2, 44.0, 45.7, 45.9, 46.4, 51.4, 52.3, 78.6, 81.8, 128.3, 128.4, 128.8, 128.9, 132.5, 132.7, 138.5, 141.5, 170.8, 172.6, 181.7, 183.1.

IR (KBr, cm^{-1}): 3061, 3029, 2956, 2875, 2843, 2359, 1739, 1735, 1662, 1615, 1344.

HRMS (ESI-TOF) Calcd for $C_{14}H_{17}ClNO_2^+$ ($[M+H]^+$) 266.0942. Found 266.0945.

3p, *trans*:*cis*= 1.4:1.0



Yellowish viscous oil

^{1}H NMR($CDCl_3$, 500 MHz)

trans-**3p,** δ 2.15 (s, 3H), 2.68 (dd, *J* = 17.5, 7.0 Hz, 1H), 3.16 (dd, *J* = 17.5, 10.0 Hz, 1H), 3.73 (m, 1H), 3.75 (s, 3H), 4.65 (d, *J* = 6.5 Hz, 1H), 6.99 (t, *J* =8.5 Hz, 2H), 7.16 (dd, *J* =8.5, 5.0 Hz , 2H).

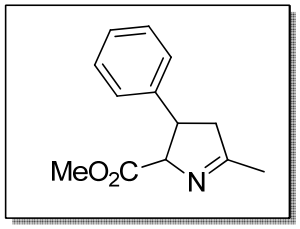
cis-**3p**, δ 2.23 (s, 3H), 2.87 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.99 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.29 (s, 3H), 3.86 (m, 1H), 4.97 (d, *J* = 8.5 Hz, 1H), 6.93 (t, *J* = 8.5 Hz, 2H), 7.07 (dd, *J* = 8.5, 5.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.8, 20.1, 45.8, 46.0, 46.1, 48.4, 51.4, 52.3, 78.9, 82.1, 115.1 (d), 115.7 (d), 128.4 (d), 129.0 (d), 135.6 (d), 138.6 (d), 160.7 (d), 162.7 (d), 170.9, 172.6, 177.4, 178.9.

IR (KBr, cm^{-1}): 3044, 2998, 2953, 2846, 1732, 1716, 1682, 1651, 1381, 1361.

HRMS (ESI-TOF) Calcd for $C_{13}H_{14}FNO_2^+$ ($[M+H]^+$) 236.1081. Found 236.1078.

3q, *trans*:*cis*= 1.5:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

trans-**3t,** δ 2.15 (s, 3H), 2.71 (dd, *J* = 18.0, 10.0 Hz, 1H), 3.15 (dd, *J* = 18.0, 7.0 Hz, 1H), 3.74 (m, 4H), 4.70 (d, *J* = 9.0 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 2H), 7.33 (m, *J* = 7.5 Hz, 3H).

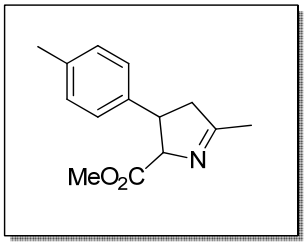
cis-**3t,** δ 2.22 (s, 3H), 2.95 (m, 2H), 3.24 (s, 3H), 3.85 (m, 1H), 4.99 (d, *J* = 9.0 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 2H), 7.20 (m, *J* = 7.5 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.6, 19.9, 45.7, 46.4, 46.6, 48.2, 51.1, 52.1, 78.8, 81.9, 126.7, 126.8, 126.9, 127.3, 128.1, 128.6, 139.7, 142.8, 170.8, 172.5, 177.5, 179.0.

IR (KBr, cm^{-1}): 3062, 3029, 3001, 2951, 2846, 1732, 1647, 1603, 1584, 1380, 1315.

HRMS (ESI-TOF) Calcd for $C_{13}H_{15}NO_2^+$ ($[M+H]^+$) 218.1176. Found 218.1181.

3r, *trans*:*cis*= 1.5:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

trans-**3r**, δ 2.14 (s, 3H), 2.31 (s, 3H), 2.68 (dd, *J* = 17.5, 7.5 Hz, 1H), 3.12 (dd, *J* = 17.5 , 7.5Hz, 1H), 3.66–3.72 (m, 4H), 4.66 (d, *J* = 6.5 Hz, 1H), 7.04 (d, *J* = 8.5 Hz, 2H)， 7.07 (d, *J* = 8.5 Hz, 2H).

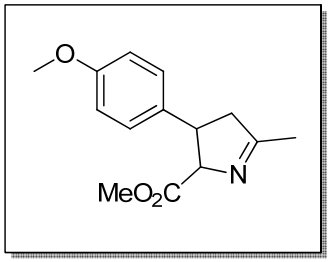
cis-**3r**, δ 2.21 (s, 3H), 2.28 (s, 3H), 2.87 (dd, *J* = 17.0, 8.5 Hz, 1H), 2.94 (dd, *J* = 17.0, 8.5 Hz, 1H), 3.26 (s, 3H), 3.82 (m, 1H), 4.95 (d, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.1, 19.8, 20.1, 20.9, 46.0, 46.3, 46.4, 48.4, 51.3, 52.2, 78.9, 82.1, 126.8, 127.3, 128.9, 129.4, 136.4, 136.5, 136.8, 139.9, 171.0, 172.8, 177.6, 179.1.

IR (KBr, cm^{-1}): 3022, 2951, 2922, 1733, 1683, 1646, 1578, 1380, 1356.

HRMS (ESI-TOF) Calcd for $C_{14}H_{18}NO_2^+$ ($[M+H]^+$) 232.1332. Found 232.1330.

3s, *trans*:*cis*= 1.5:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

trans-**3s**, δ 2.15 (s, 3H), 2.68 (dd, *J* = 17.5, 7.0 Hz, 1H), 3.13 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.70 (m, 1H), 3.74 (s, 3H), 3.76 (s, 3H), 4.65 (d, *J* = 6.5 Hz, 1H), 6.85 (d, *J* = 8.5 Hz, 2H), 7.11 (d, *J* = 8.5 Hz, 2H).

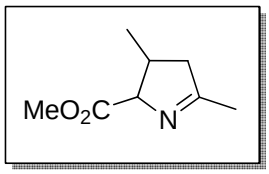
cis-**3s**, δ 2.23 (s, 3H), 2.87 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.96 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.29 (s, 3H), 3.78 (s, 3H), 3.81 (m, 1H), 4.95 (d, *J* = 8.5Hz, 1H), 6.79 (d, *J* = 9.0 Hz, 2H), 7.02 (d, *J* = 9.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.8, 20.1, 45.9, 46.0, 46.1, 48.4, 51.4, 52.3, 55.1, 55.2, 79.0, 82.1, 113.6, 114.1, 127.9, 128.5, 131.9, 134.9, 158.4, 158.5, 171.1, 172.8, 177.6, 179.0.

IR (KBr, cm^{-1}): 2998, 2952, 2837, 1739, 1683, 1669, 1646, 1379, 1356.

HRMS (ESI-TOF) Calcd for $C_{14}H_{18}NO_3^+$ ($[M+H]^+$) 248.1281. Found 248.1297.

3t, *trans*:*cis*= 1.4:1.0



Yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz)

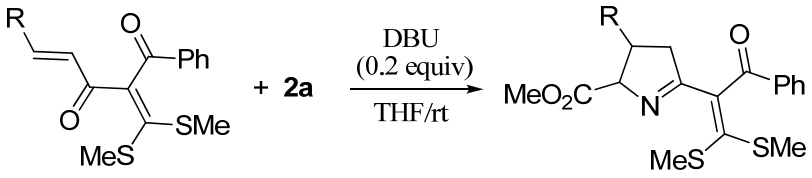
trans-**3t**, δ 1.18 (d, 3H), 2.05 (s, 3H), 2.18 (dd, *J* = 17.0, 7.0 Hz, 1H), 2.53–2.58 (m, 1H), 3.13 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.76 (s, 3H), 4.21 (d, *J* = 6.0 Hz, 1H).

cis-**3t**, δ 0.93 (d, 3H), 2.08 (s, 3H), 2.28 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.67–2.76 (m, 2H), 3.75 (s, 3H), 4.65 (d, *J* = 7.0 Hz, 1H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 15.8, 19.7, 20.0, 20.2, 35.1, 36.4, 46.9, 47.3, 51.6, 52.1, 80.8, 172.0, 173.3, 178.1, 178.6.

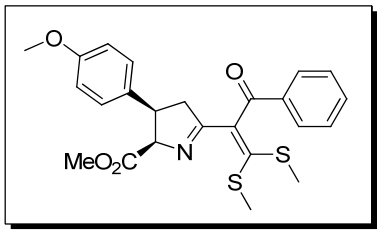
IR (KBr, cm^{-1}): 2956, 2875, 2845, 1740, 1645, 1380, 1355.

HRMS (ESI-TOF) Calcd for $C_8H_{14}NO_2^+$ ($[M+H]^+$) 156.1019. Found 156.1027.



6a-6c **7a-7c**

7a, *trans*:*cis*= 1.7:1.0



cis-**7a**: yellowish viscous oil

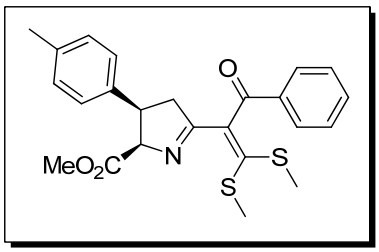
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.09 (s, 3H), 2.41 (s, 3H), 3.18 (s, 3H), 3.24 (d, *J* = 8.5 Hz, 2H), 3.69 (s, 3H), 3.73 (m, 1H), 5.01 (d, *J* = 8.5 Hz, 1H), 6.69 (d, *J* = 8.5 Hz, 2H), 6.98 (d, *J* = 8.5 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.92 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 29.7, 30.5, 44.4, 46.0, 51.4, 55.2, 78.7, 113.6, 128.6, 128.7, 129.4, 130.8, 133.2, 137.0, 139.5, 146.0, 158.5, 170.3, 174.6, 193.9.

IR (KBr, cm^{-1}): 3061, 2955, 2924, 1734, 1669, 1307.

HRMS (ESI-TOF) Calcd for $C_{24}H_{26}NO_4S_2^+$ ($[M+H]^+$) 456.1289. Found 456.1290.

7b, *trans*:*cis*= 1.4:1.0



cis-**7b**: yellowish crystals, m.p. 190–192 °C

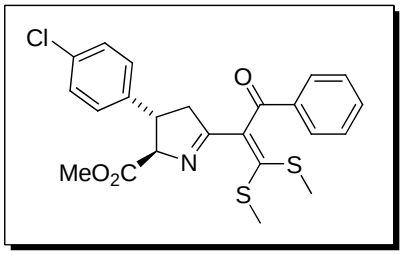
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.16 (s, 3H), 2.28 (s, 3H), 2.47 (s, 3H), 3.23 (s, 3H), 3.33 (m, 2H), 3.82 (m, 1H), 5.10 (d, *J* = 8.5 Hz, 1H), 7.02 (m, 4H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.55(t, *J* = 7.5 Hz, 1H), 7.99 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 16.5, 16.6, 30.5, 34.1, 43.1, 46.2, 51.4, 77.9, 128.3, 128.7, 128.8, 129.0, 129.1, 129.3, 129.4, 130.9, 132.8, 133.1, 137.0, 137.2, 170.3, 180.8, 197.4.

IR (KBr, cm^{-1}): 3055, 2950, 2924, 1740, 1662, 1312.

HRMS (ESI-TOF) Calcd for $C_{24}H_{26}NO_3S_2^+$ ($[M+H]^+$) 440.1349. Found 440.1356.

7c, *trans*:*cis*= 1.3:1.0



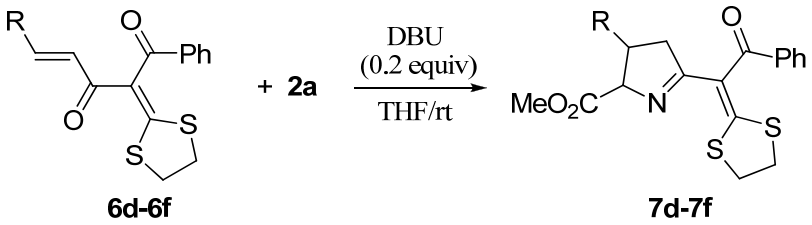
trans-**7c**: yellowish crystals, m.p. 150–152 ^{o}C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.16 (s, 3H), 2.46 (s, 3H), 3.01 (dd, *J* = 17.5, 6.0 Hz, 1H), 3.56 (dd, *J* = 17.5, 9.5 Hz, 1H), 3.67 (m, 1H), 3.70 (s, 3H), 4.80 (d, *J* = 5.5 Hz, 1H), 7.08 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.5 Hz, 2H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.95 (d, *J* = 7.5 Hz, 2H)

^{13}C NMR ($CDCl_3$, 125 MHz) δ 44.4, 45.7, 46.0, 47.2, 51.5, 78.6, 128.3, 128.4, 128.7, 129.0, 129.1, 129.4, 132.8, 133.3, 137.0, 137.5, 139.2, 146.6, 170.1, 174.4, 193.9.

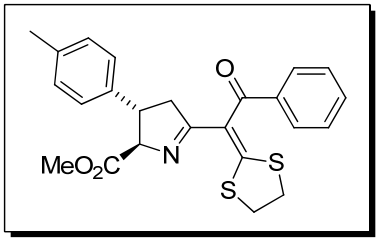
IR (KBr, cm^{-1}): 3066, 2952, 1736, 1615, 1345.

HRMS (ESI-TOF) Calcd for $C_{23}H_{23}ClNO_3S_2^+$ ($[M+H]^+$) 460.0802. Found 460.0822.



6d-6f **7d-7f**

7d, *trans*:*cis*= 1.5:1.0



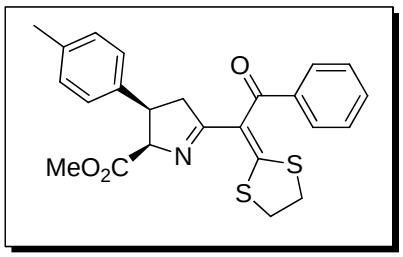
trans-**7d**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.28 (s, 3H), 2.43 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.77 (dd, *J* = 17.5, 8.0 Hz, 1H), 3.39 (m, 2H), 3.44 (m, 2H), 3.54 (m, 1H), 3.79 (s, 3H), 4.87 (d, *J* = 6.0 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 2H), 7.04 (d, *J* = 8.5 Hz, 2H), 7.42 (t, *J* = 8.5 Hz, 2H), 7.51 (t, *J* = 8.5 Hz, 1H), 7.77 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 36.7, 38.1, 46.3, 47.4, 52.2, 81.2, 120.3, 126.7, 128.4, 128.6, 129.3, 132.1, 136.3, 139.1, 139.2, 170.5, 172.3, 175.6, 191.0.

IR (KBr, cm^{-1}): 3005, 2950, 1744, 1681, 1310.

HRMS (ESI-TOF) Calcd for $C_{24}H_{24}NO_3S_2^+$ ($[M+H]^+$) 424.1036. Found 424.1035.



cis-**7d**: yellowish crystals, m.p. 176–178 °C

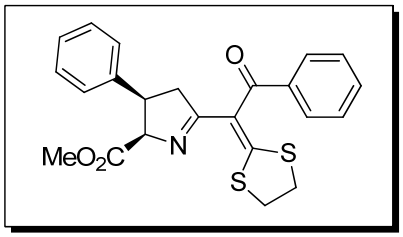
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.53 (s, 3H), 2.53 (dd, *J* = 17.0, 9.0 Hz, 1H), 2.68 (dd, *J* = 17.0, 9.0 Hz, 1H), 3.31 (s, 3H), 3.37 (m, 2H), 3.42 (m, 2H), 3.67 (m, 1H), 5.18 (d, *J* = 9.0 Hz, 1H), 6.85 (d, *J* = 9.0 Hz, 2H), 6.99 (d, *J* = 9.0 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.9, 36.9, 37.9, 44.4, 46.7, 51.2, 78.1, 120.7, 127.3, 128.4, 128.6, 128.7, 132.2, 135.5, 136.4, 139.2, 170.4, 170.5, 176.5, 191.7.

IR (KBr, cm^{-1}): 3011, 2920, 1736, 1626, 1319.

HRMS (ESI-TOF) Calcd for $C_{23}H_{22}NO_3S_2^+$ ($[M+H]^+$) 424.1036. Found 424.1035.

7e, *trans:cis*= 1.4:1.0



cis-**7e**: yellowish crystals, m.p. 150–152 °C

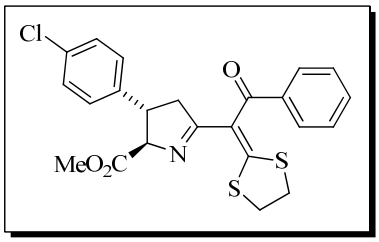
^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.45 (dd, *J* = 17.5, 6.5 Hz, 1H),2.80 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.36–3.41 (m, 2H), 3.42–3.44 (m, 2H), 3.57 (m, 1H), 3.79 (s, 3H), 4.91 (d, *J* = 6.0 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 2H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 1H),7.78 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 36.8, 38.1, 46.6, 47.5, 52.3, 81.3, 120.4, 126.8, 126.9, 128.5, 128.7, 132.2, 139.3, 142.4, 170.7, 172.4, 175.8, 191.2.

IR (KBr, cm^{-1}): 3027, 2953, 1731, 1618, 1350.

HRMS (ESI-TOF) Calcd for $C_{24}H_{24}NO_3S_2^+$ ($[M+H]^+$) 438.1192. Found 438.1197.

7f, *trans*:*cis*= 1.3:1.0



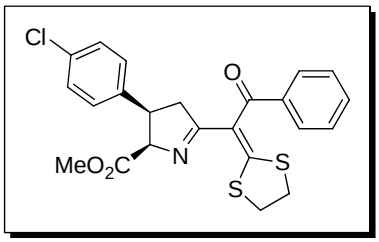
trans-**7f**: yellowish crystals, m.p. 153–155 °C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.32 (dd, *J* = 17.0, 6.5 Hz, 1H), 2.73 (dd, *J*=17.5, 9.5 Hz, 1H), 3.30 (m, 2H), 3.35 (m, 2H), 3.47 (m, 1H), 3.72 (s, 3H), 4.78 (d, *J* = 6.0 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 2H), 7.12 (d, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.69 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 36.8, 38.1, 45.9, 47.3, 52.3, 81.1, 120.2, 128.2, 128.5, 128.6, 128.7, 132.2, 132.5, 139.2, 140.9, 170.7, 172.1, 175.4, 191.1.

IR (KBr, cm^{-1}): 3060, 2952, 2928, 1736, 1630, 1352.

HRMS (ESI-TOF) Calcd for $C_{23}H_{21}ClNO_3S_2^+$ ($[M+H]^+$) 458.0646. Found 458.0663.



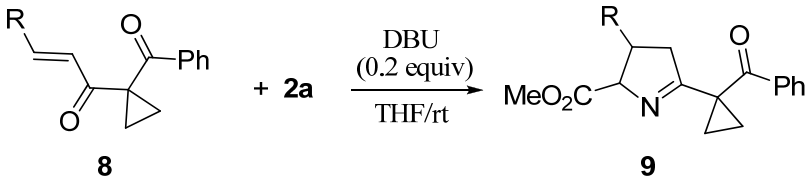
cis-**7f**: yellowish crystals, m.p. 172–174 °C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.58 (dd, *J* = 17.0, 8.5 Hz, 1H), 2.65 (dd, *J*= 17.0, 7.5 Hz, 1H), 3.33 (s, 3H), 3.39 (m, 2H), 3.43 (m, 2H), 3.68 (m, 1H), 5.19 (d, *J* = 8.5 Hz, 1H), 6.90 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.80 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 37.1, 38.0, 44.6, 46.4, 51.5, 78.1, 120.6, 128.3, 128.6, 128.8, 128.9, 132.4, 132.7, 137.5, 139.3, 170.4, 170.6, 176.1, 191.9.

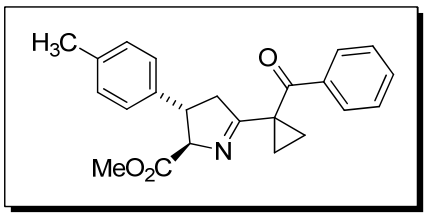
IR (KBr, cm^{-1}): 3061, 2949, 2924, 1736, 1625, 1345.

HRMS (ESI-TOF) Calcd for $C_{23}H_{21}ClNO_3S_2^+$ ($[M+H]^+$) 458.0646. Found 458.0663.



8 **9**

9a, *trans*:*cis*= 5.8:1.0



trans-**9a**: yellowish crystals, m.p. 89–91 °C

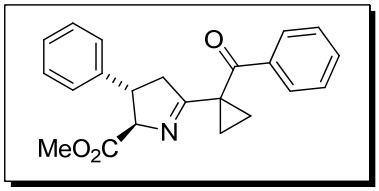
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.63 (s, 2H), 1.64 (s, 2H), 2.28 (s, 3H), 2.48 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.96 (dd, *J* = 17.5, 10.0 Hz, 1H), 3.53 (m, 1H), 3.72 (s, 3H), 4.67 (d, *J* = 5.0 Hz, 1H), 6.82 (d, *J* = 8.5 Hz, 2H), 7.02 (d, *J* = 8.5 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.99 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 16.1, 16.5, 20.8, 33.7, 45.9, 46.0, 52.1, 81.3, 126.3, 128.4, 128.8, 129.3, 132.8, 136.3, 136.9, 139.7, 172.2, 179.4, 197.2.

IR (KBr, cm^{-1}): 3060, 2952, 2924, 1739, 1668, 1438.

HRMS (ESI-TOF) Calcd for $C_{23}H_{24}NO_3^+$ ($[M+H]^+$)362.1751. Found 362.1750.

9b, *trans*:*cis*= 2.0:1.0



trans-**9b**: yellowish viscous oil

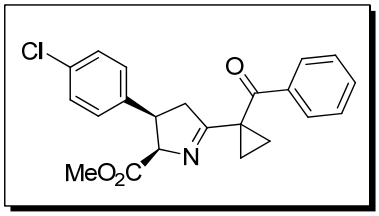
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.64 (s, 4H), 2.50 (dd, *J* = 17.5, 5.0 Hz, 1H), 2.99 (dd, *J* = 17.5, 5.0 Hz, 1H), 3.58 (m, 1H), 3.73 (s, 3H), 4.71 (d, *J* = 3.5 Hz, 1H), 6.93 (d, *J* = 7.0 Hz, 2H), 7.17–7.21 (m, 3H), 7.46 (t, *J* = 7.0 Hz, 2H), 7.57 (t, *J* = 7.0 Hz, 1H), 7.99 (d, *J* = 7.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 16.3, 16.7, 33.9, 46.1, 46.3, 52.3, 81.4, 126.6, 126.9, 128.6, 128.8, 128.9, 133.0, 137.0, 142.9, 172.3, 179.6, 197.5.

IR (KBr, cm^{-1}): 3063, 2952, 2927, 1738, 1670, 1373.

HRMS (ESI-TOF) Calcd for $C_{22}H_{22}NO_3^+$ ($[M+H]^+$) 348.1594. Found 348.1589.

9c, *trans*:*cis*= 1.2:1.0



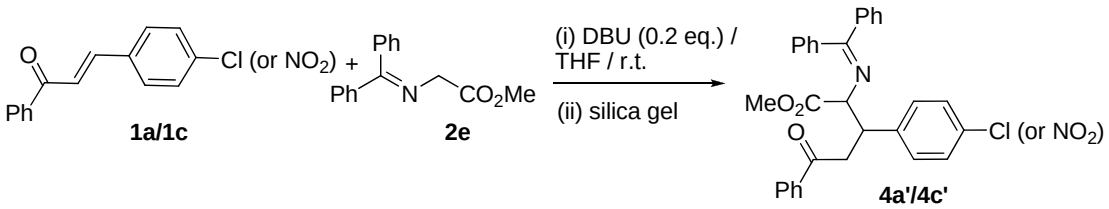
cis-**9c**: yellowish viscous oil

^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.65 (m, 2H), 1.72 (m, 2H), 2.75 (m, 2H), 3.27 (s, 3H), 3.69 (m, 1H), 4.97 (d, *J* = 9.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.60 (t, *J* = 7.0 Hz, 1H), 8.04 (d, *J* = 7.0 Hz, 2H).

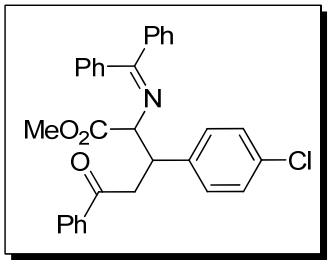
^{13}C NMR ($CDCl_3$, 125 MHz) δ 17.2, 18.1, 21.0, 44.3, 46.4, 51.3, 78.7, 127.5, 128.6, 128.9, 129.4, 133.2, 135.7, 136.6, 137.0, 139.5, 146.0, 170.2, 174.6, 193.9.

IR (KBr, cm^{-1}): 3069, 2890, 2924, 1745, 1676, 1381.

HRMS (ESI-TOF) Calcd for $C_{22}H_{21}ClNO_3^+$ ($[M+H]^+$) 382.1204. Found 382.1215.



1a/1c **2e** **4a'/4c'**



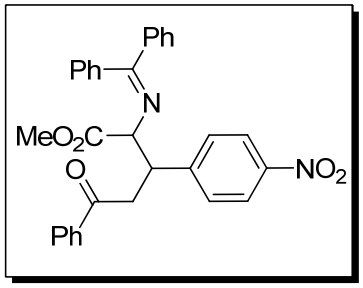
4a': colorless crystals, m.p. 147–149 ^{o}C

^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.64 (s, 3H), 3.71 (m, 1H), 3.79 (dd, *J* = 17.5, 10.0 Hz, 1H), 4.21 (m, 1H), 4.29 (d, *J* = 4.5 Hz, 1H), 6.66 (d, *J* = 4.5 Hz, 2H), 7.07 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 7.29–7.37 (m, 4H), 7.39 –7.46 (m, 4H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 7.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 39.2, 43..7, 52.1, 69.8, 127.2, 128.0, 128.1, 128.2, 128.3, 128.4, 128.5, 128.8, 129.6, 130.6, 132.3, 133.0, 135.6, 136.8, 138.9, 139.7, 171.2, 171.9, 198.1.

IR (KBr, cm^{-1}): 3060, 3025, 2951, 1727, 1686, 1358.

HRMS (ESI-TOF) Calcd for $C_{31}H_{27}ClNO_3^+$ ($[M+H]^+$) 496.1674. Found 496.1679.



4c': yellowish viscous oil

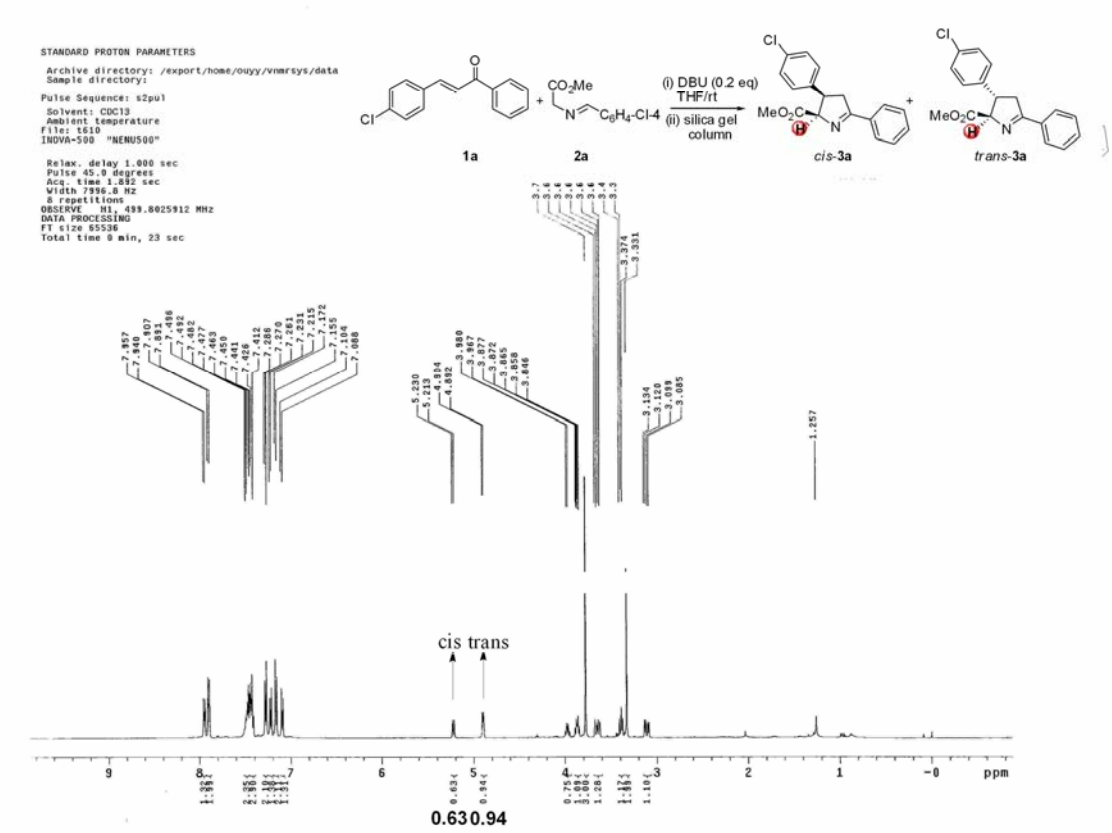
^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.67 (s, 3H), 3.77 (m, 1H), 3.93 (dd, *J* = 17.5, 10.0 Hz, 1H), 4.32 (m, 2H), 6.66 (d, *J* = 4.5 Hz, 2H), 7.29–7.35 (m, 7H), 7.37–7.48 (m, 3H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.97 (d, *J* = 8.0 Hz, 2H), 8.05 (d, *J* = 10.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 39.0, 44.1, 52.4, 69.2, 123.4, 127.1, 128.0, 128.2, 128.4, 128.6, 128.7, 128.8, 129.2, 130.9, 133.3, 135.5, 136.5, 138.7, 146.6, 149.3, 170.8, 172.5, 197.7.

IR (KBr, cm^{-1}): 3060, 3027, 2952, 1738, 1685, 1346.

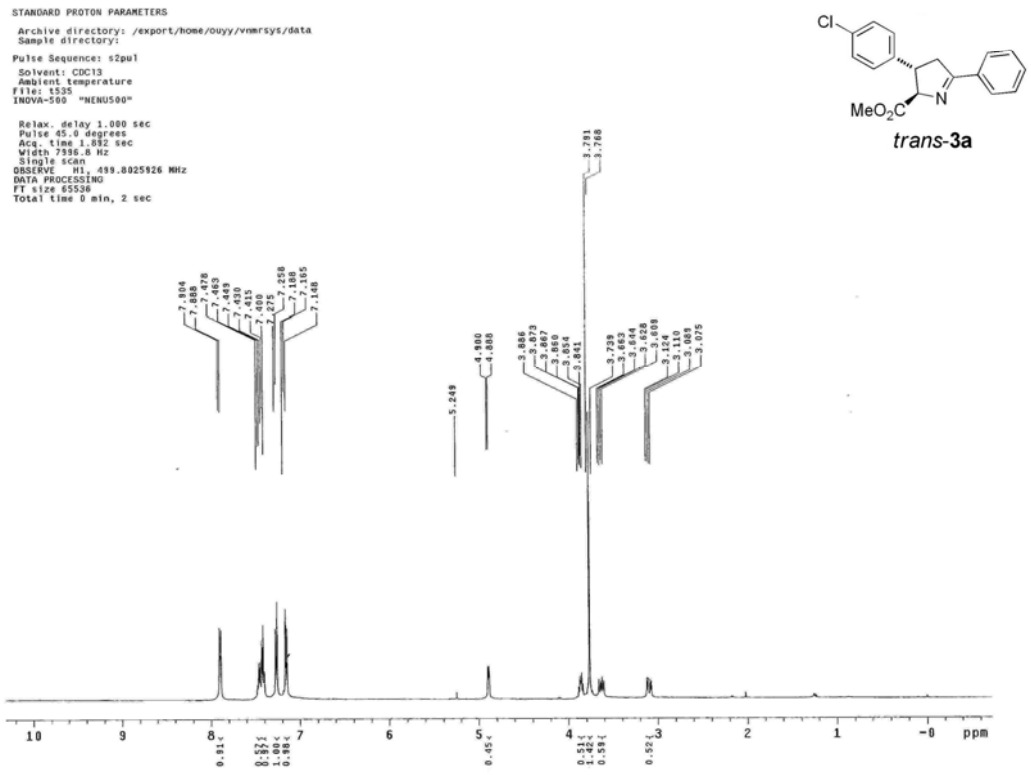
HRMS (ESI-TOF) Calcd for $C_{31}H_{27}N_2O_5^+$ ($[M+H]^+$) 507.1914. Found 507.1923.

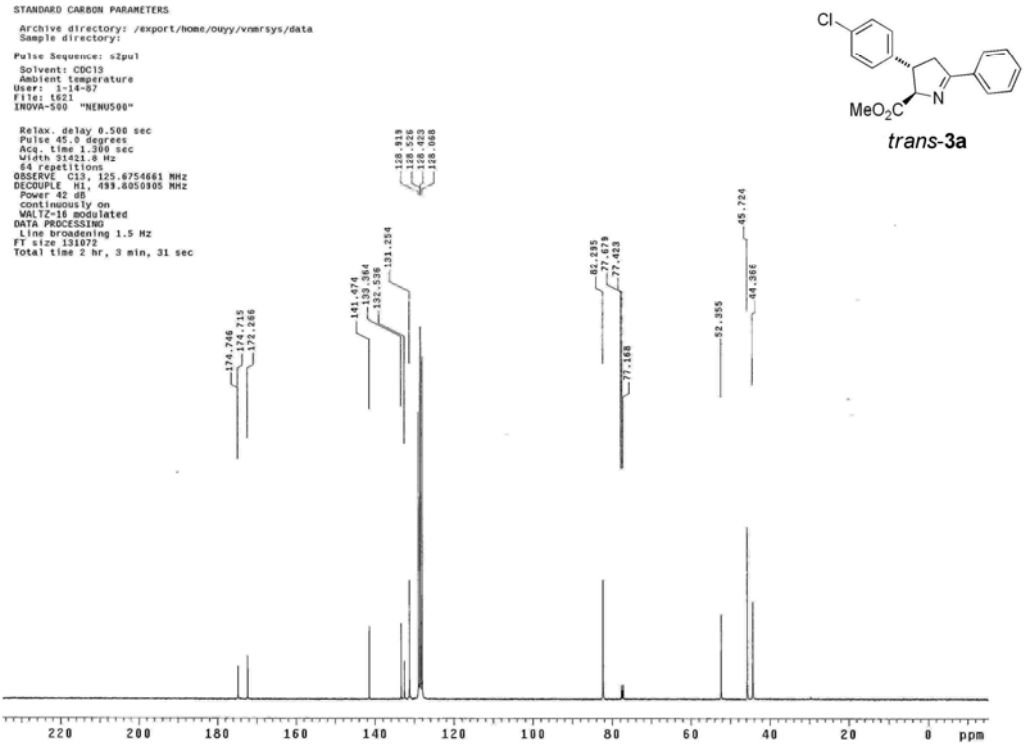
III. Determination on the ratio of *trans*/*cis* configuration

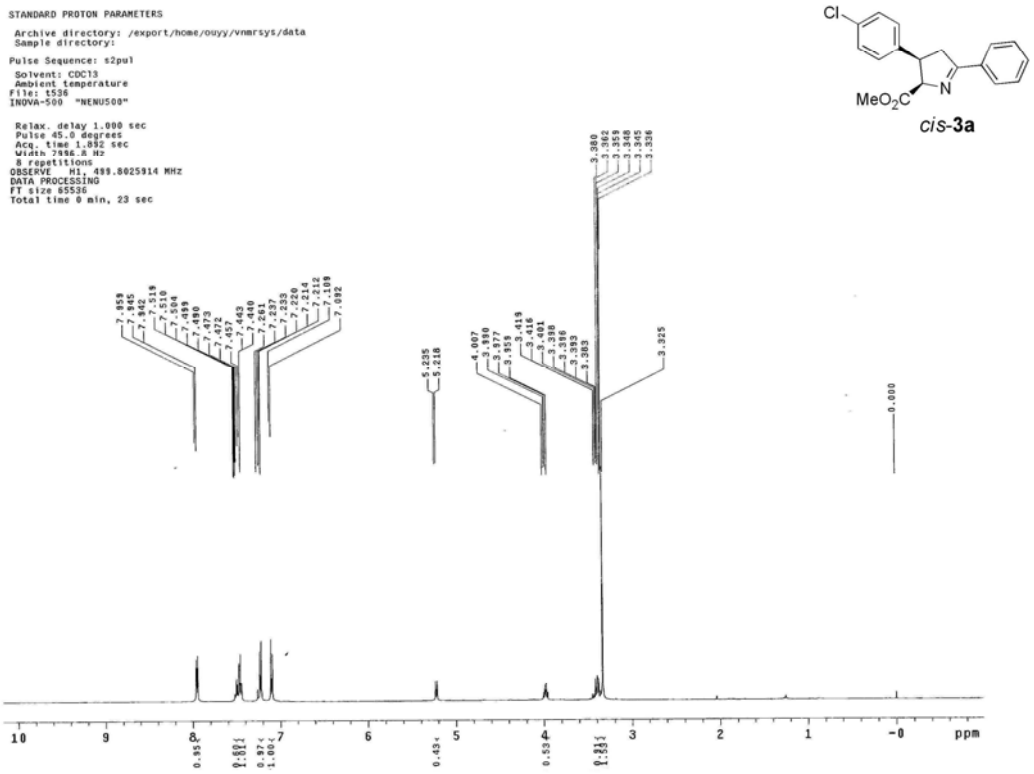


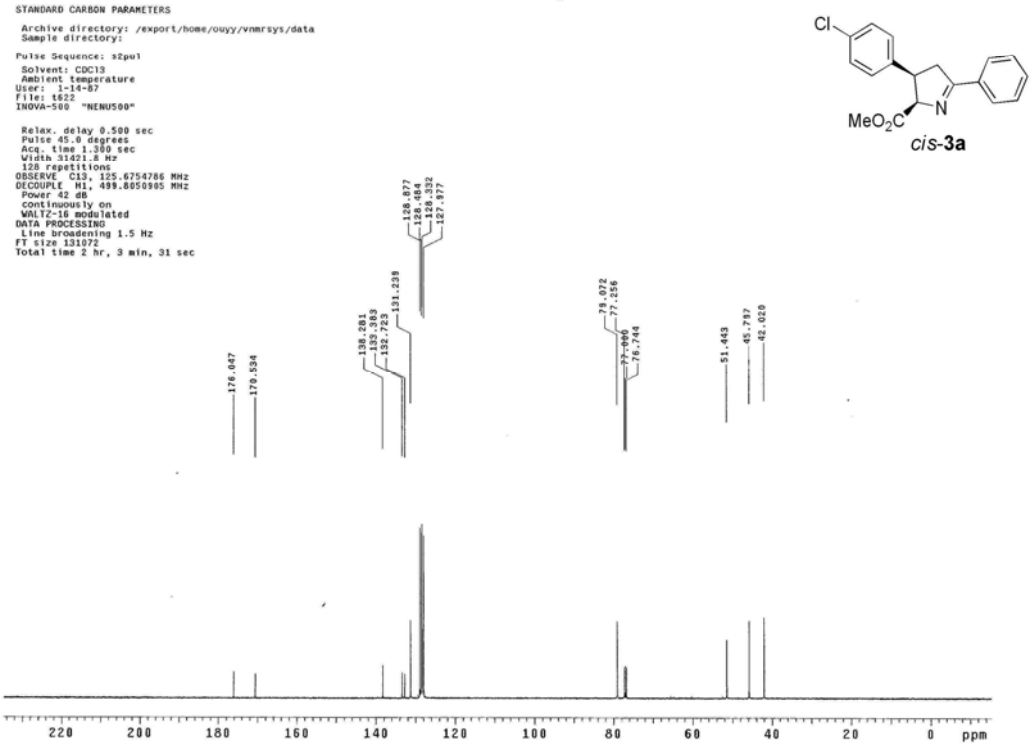
The *trans*/*cis* configuration of **3** was calculated on the integration of the methylene proton connected to CO_2Me group at 4.1-5.4 ppm in ^1H NMR spectra of the diastereomeric mixture, in which the signal for the *cis*-diastereoisomer was further determined by the crystallographic data of *cis*-**3d** (see supplementary crystallographic data in part V). For example, in the ^1H NMR spectrum of diastereomeric mixture **3a**, the peak at 5.22 ppm is attributed to the *cis*-form while the peak at 4.90 ppm is attributed to the *trans*-form with an integration ratio of 0.94/0.63 = 1.5/1.0. Thus, the ratio of *trans*/*cis* configuration of **3a** is determined as 1.5/1.0.

IV. Copies of ^{1}H NMR and ^{13}C NMR spectra

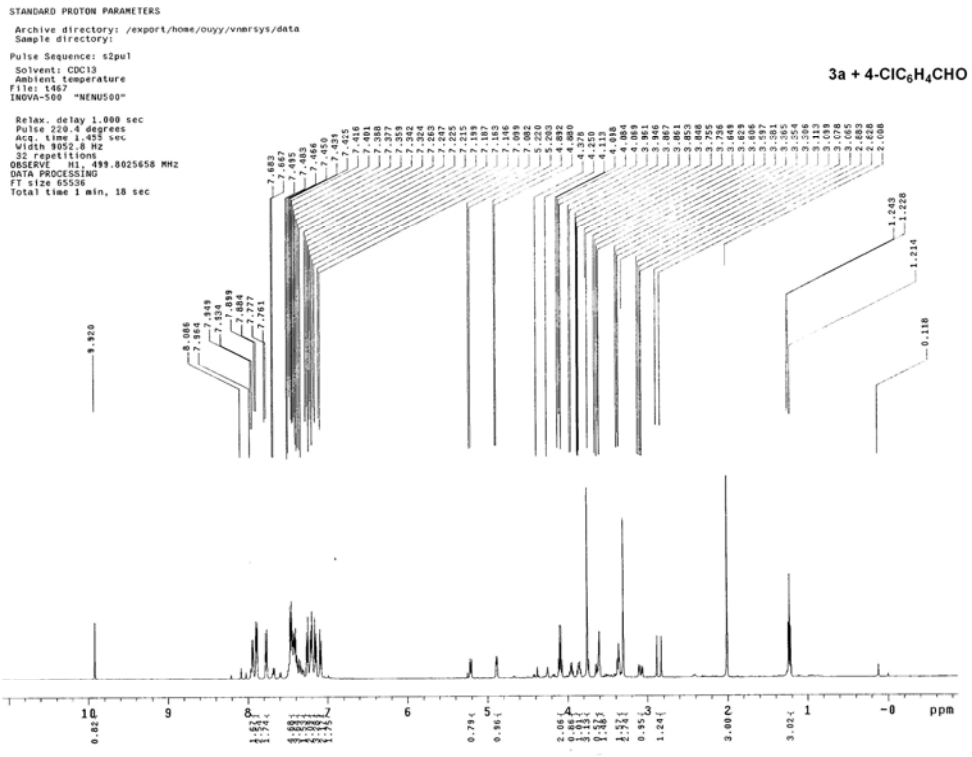


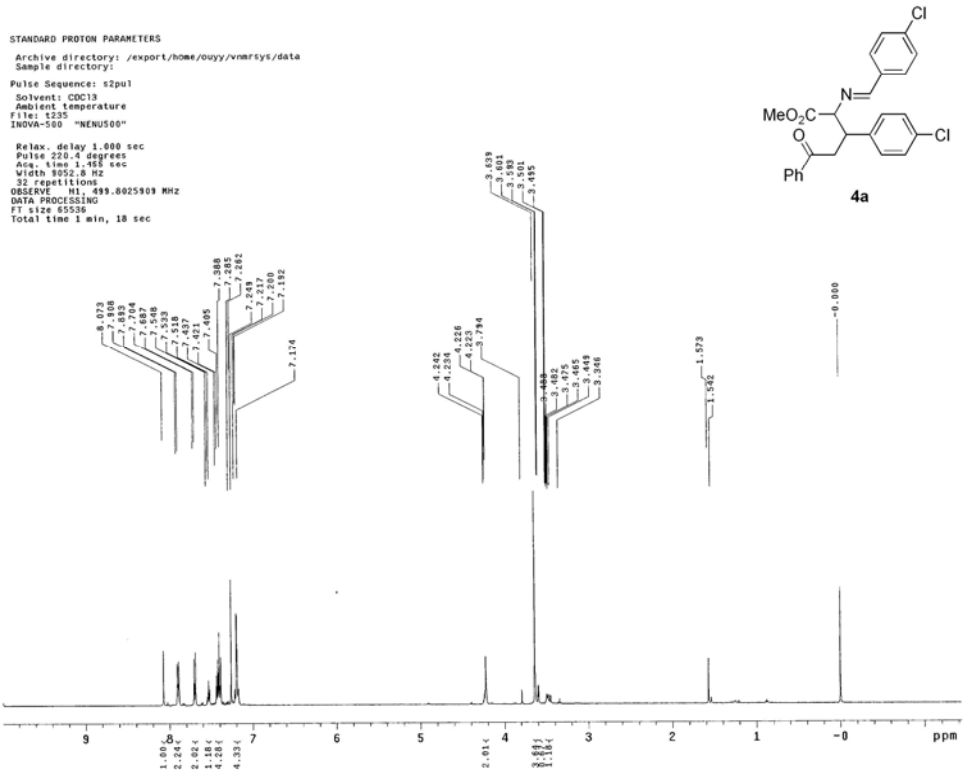


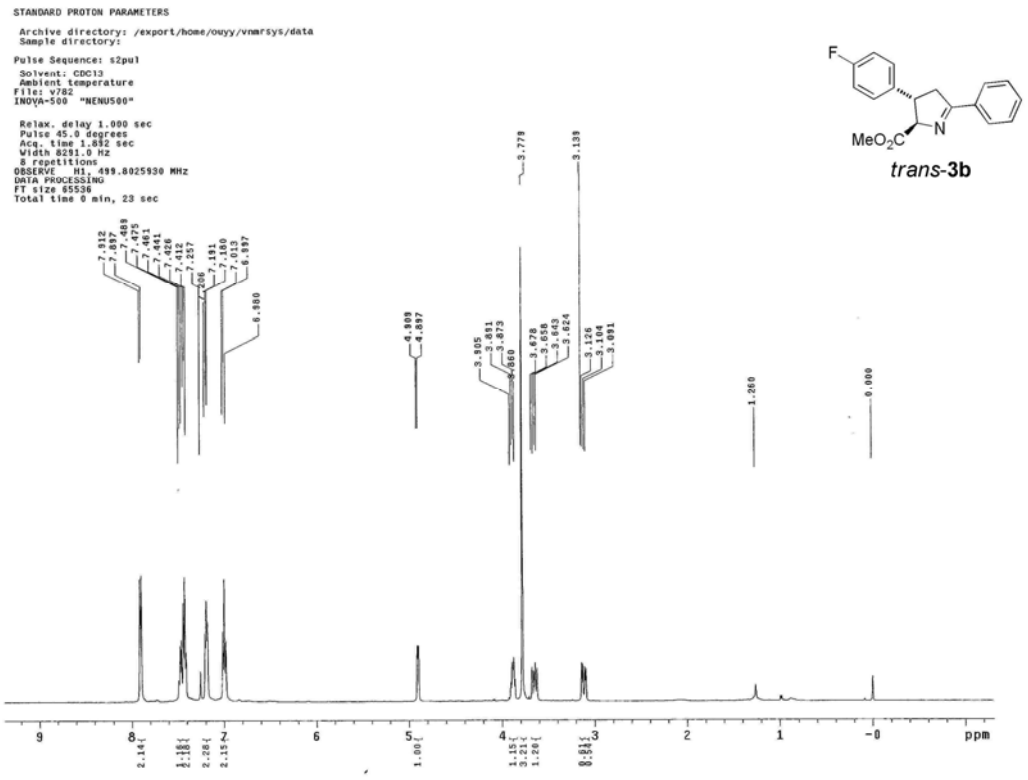

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t536
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
MeO2C
N
cis-3a
ppm

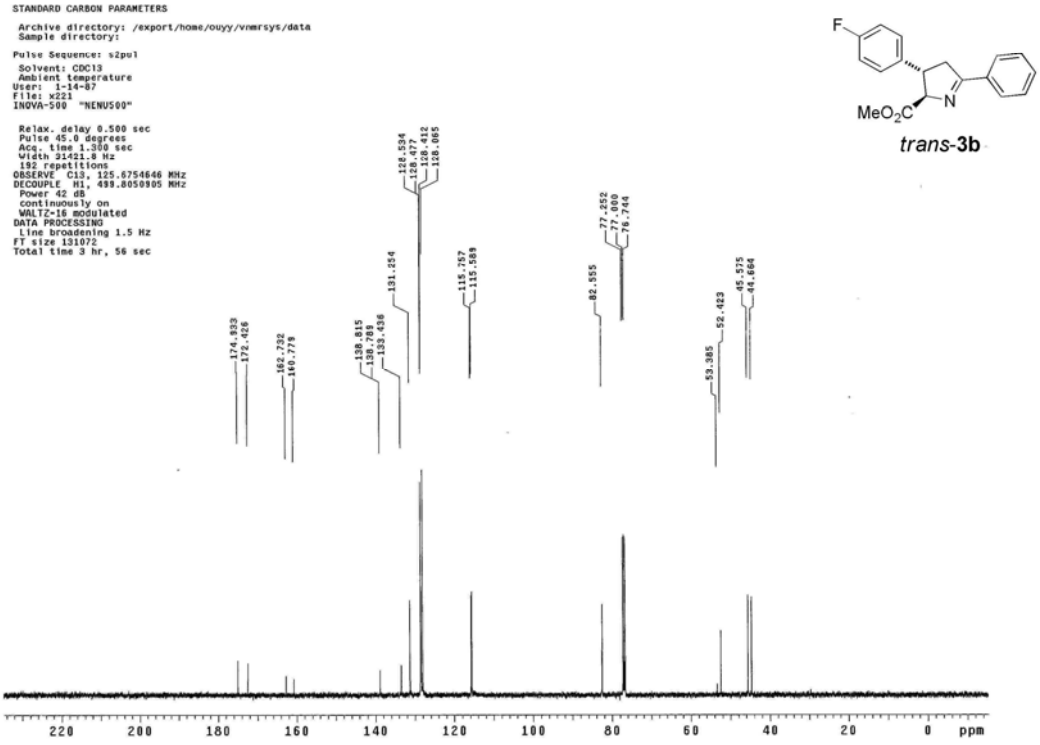

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t622
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.360 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754786 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Cl
MeO2C
N
cis-3a
ppm

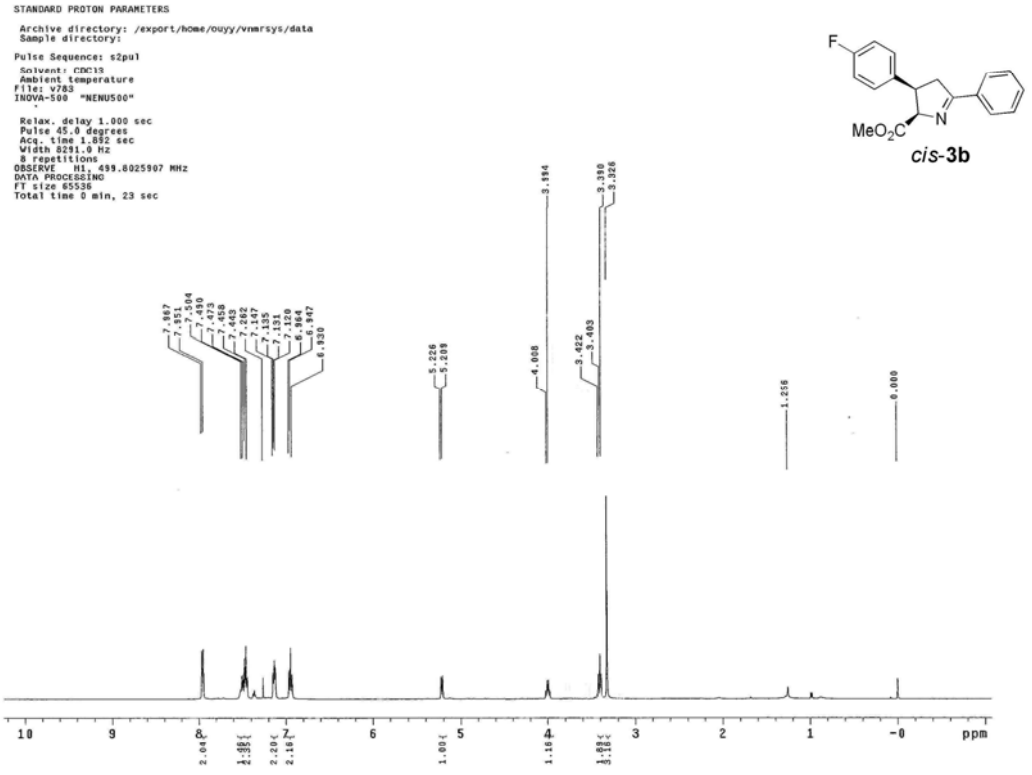
3a + 4-ClC_6H_4CHO (from the crude product of **1a** with **2a**)

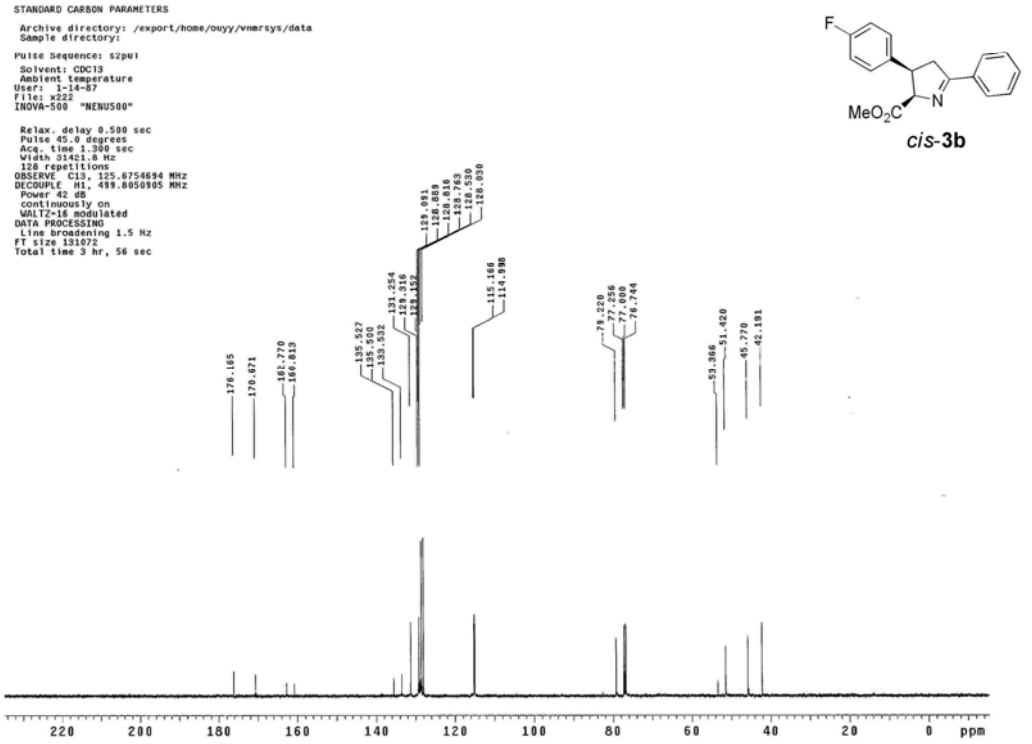


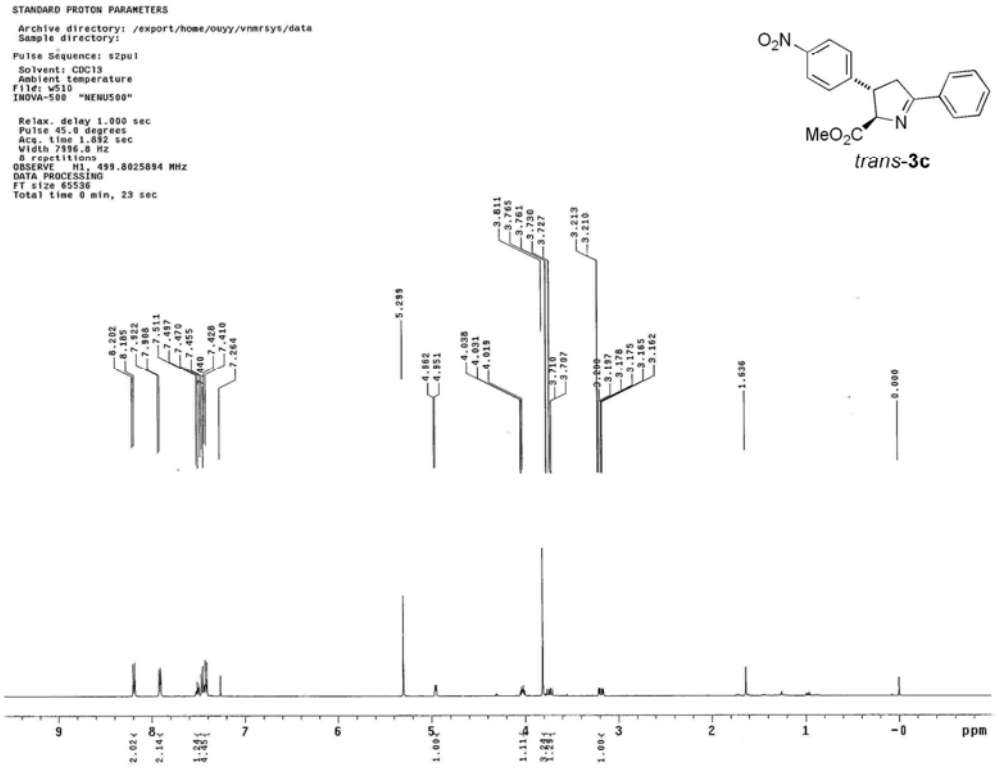


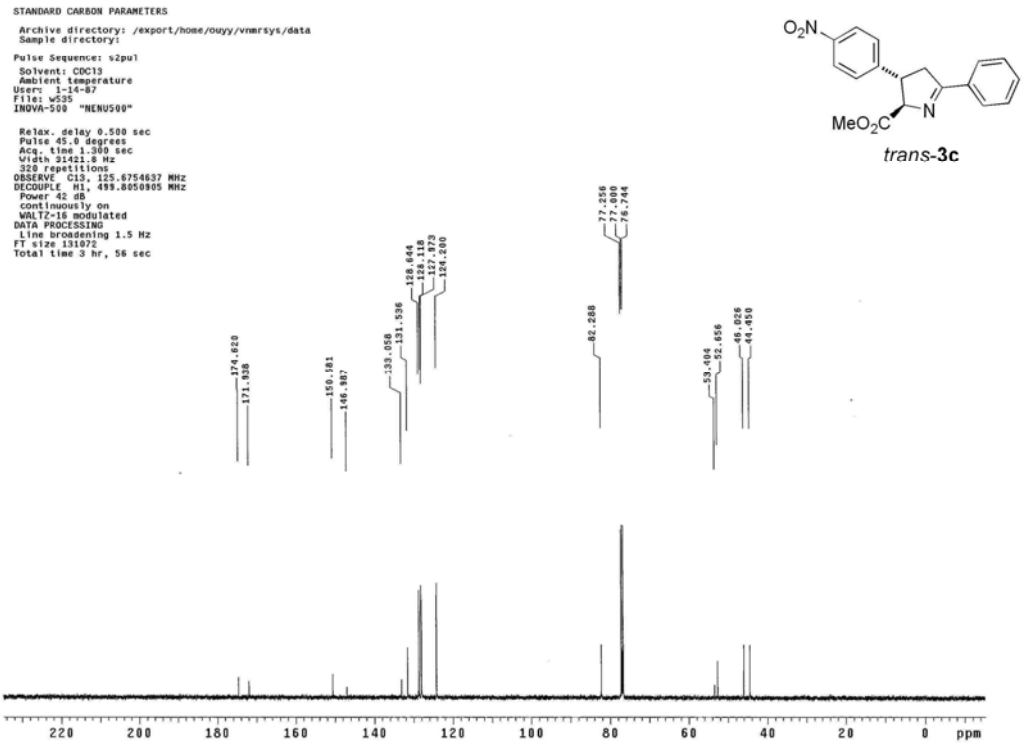

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v782
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025930 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
F
MeO2C
N
trans-3b
ppm

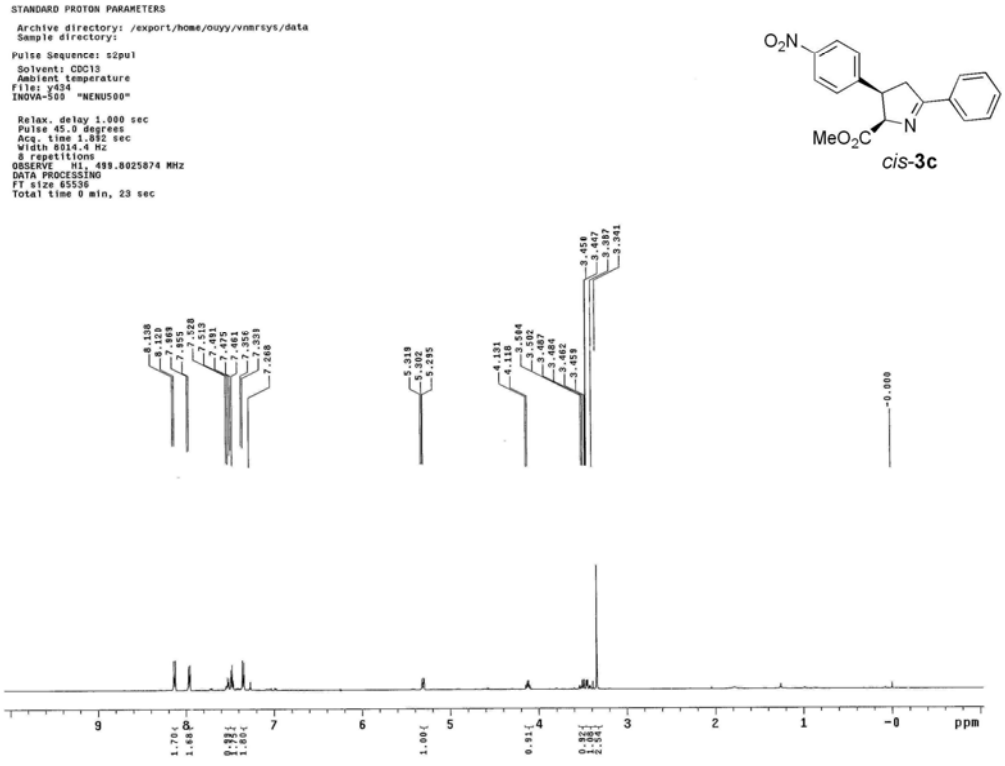

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: x221
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
F
MeO2C
N
trans-3b
ppm

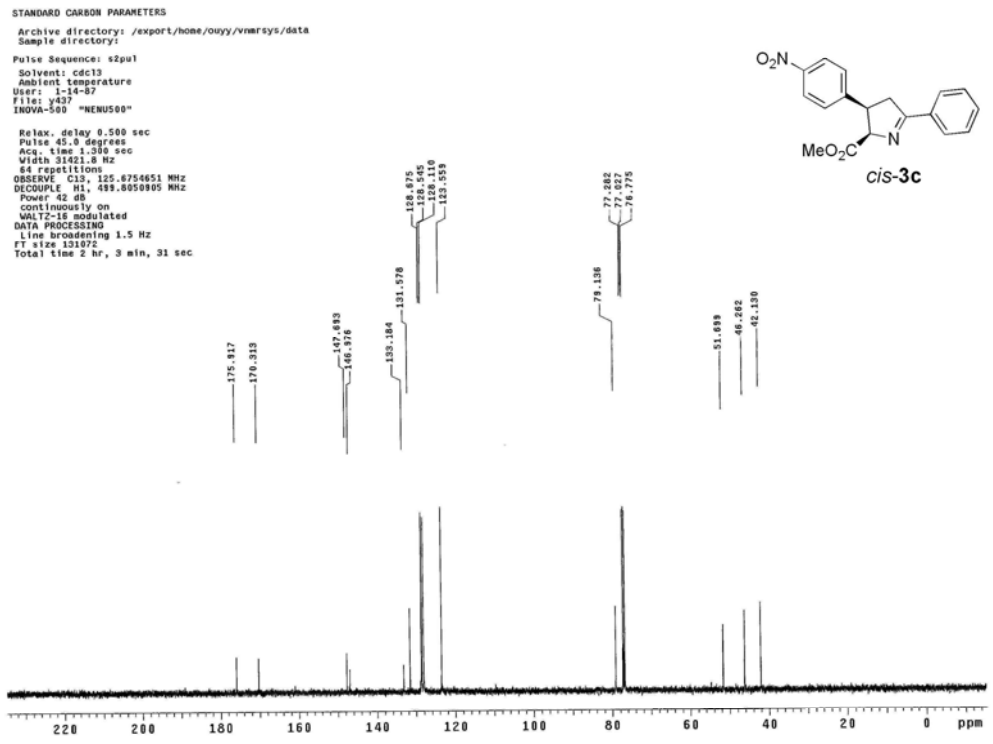

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v783
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025907 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
F
MeO2C
N
cis-3b
7.887
7.871
7.504
7.490
7.473
7.458
7.450
7.262
7.147
7.135
7.132
7.120
6.964
6.947
6.930
5.226
5.209
4.008
3.994
3.422
3.403
3.390
3.326
1.256
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm

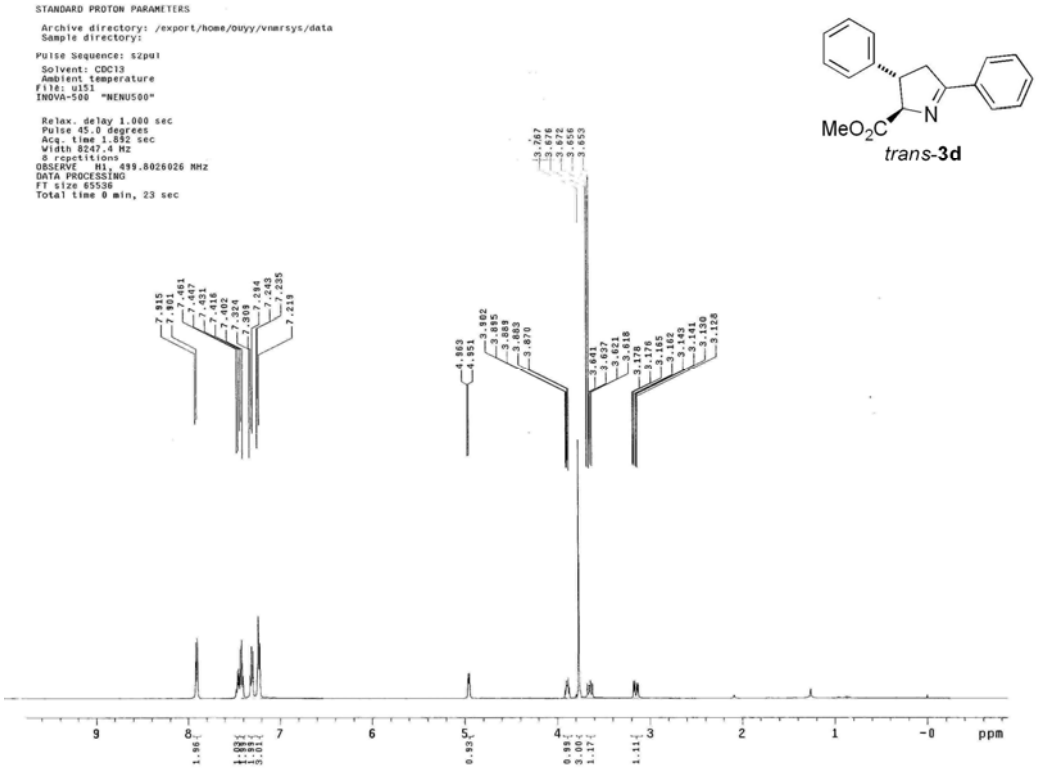

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: x222
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.6 Hz
128 repetitions
OBSERVE C13, 125.6754694 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
F
MeO2C
N
cis-3b
176.185
170.671
161.770
160.813
135.527
135.500
133.532
131.254
129.316
129.192
129.091
128.889
128.818
128.738
128.530
128.030
115.166
114.998
79.220
77.256
77.000
76.744
53.366
51.420
45.770
42.191
220
200
180
160
140
120
100
80
60
40
20
0
ppm

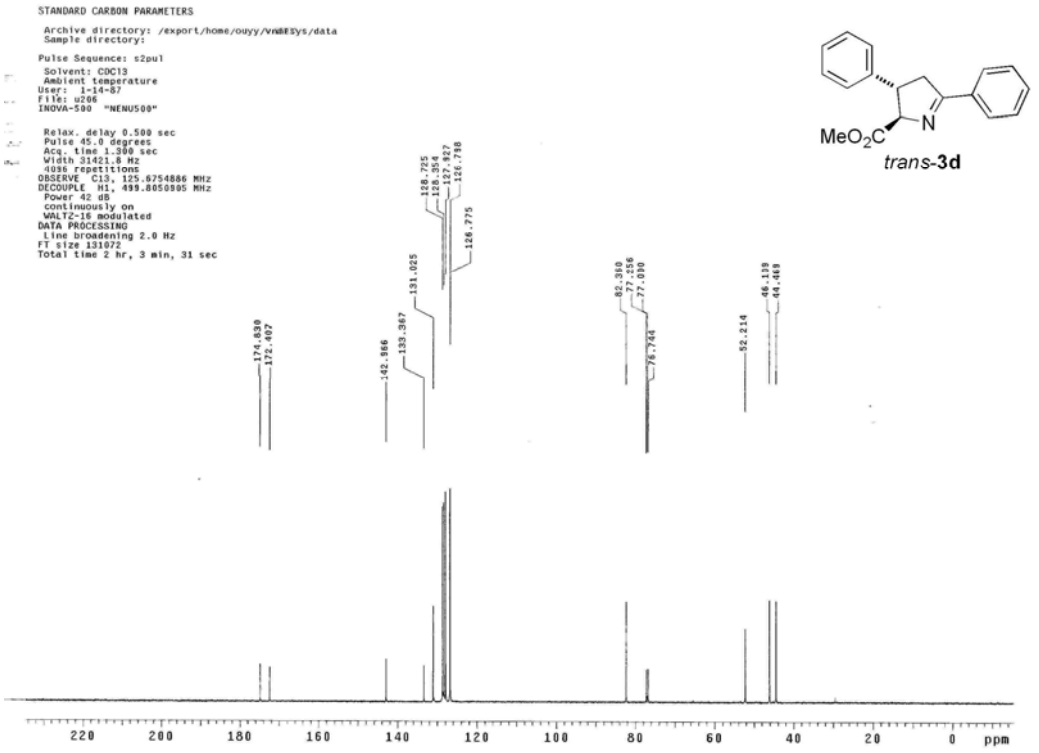

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w510
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
O2N
MeO2C
N
trans-3c
ppm

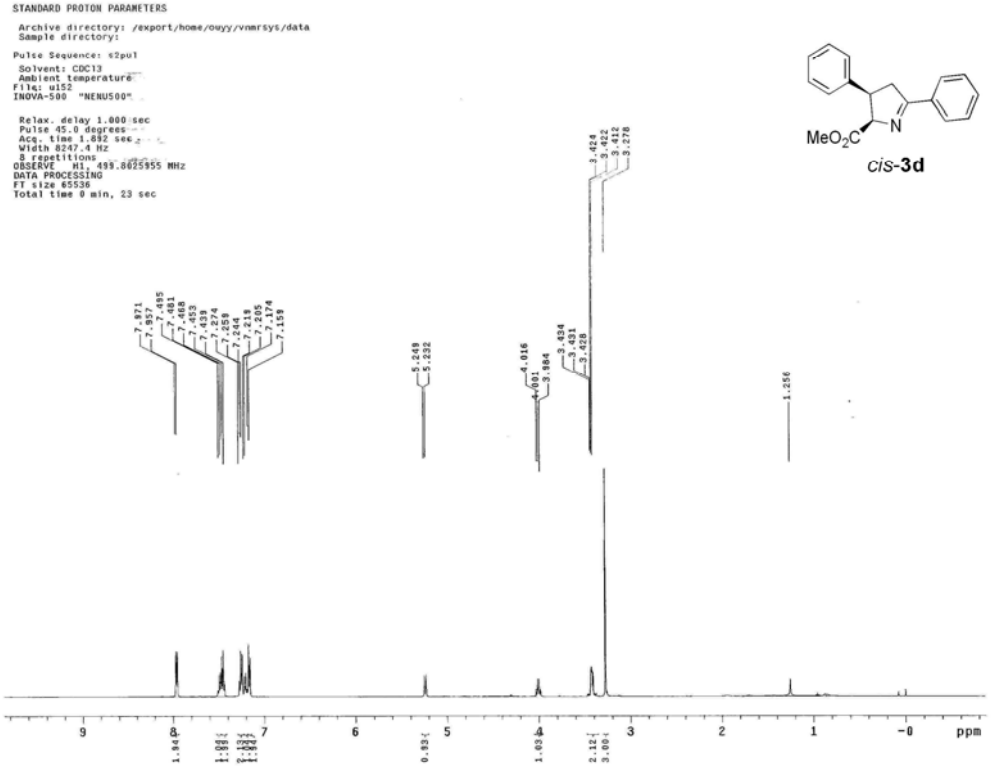

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: w535
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754637 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
O2N
MeO2C
N
trans-3c
ppm

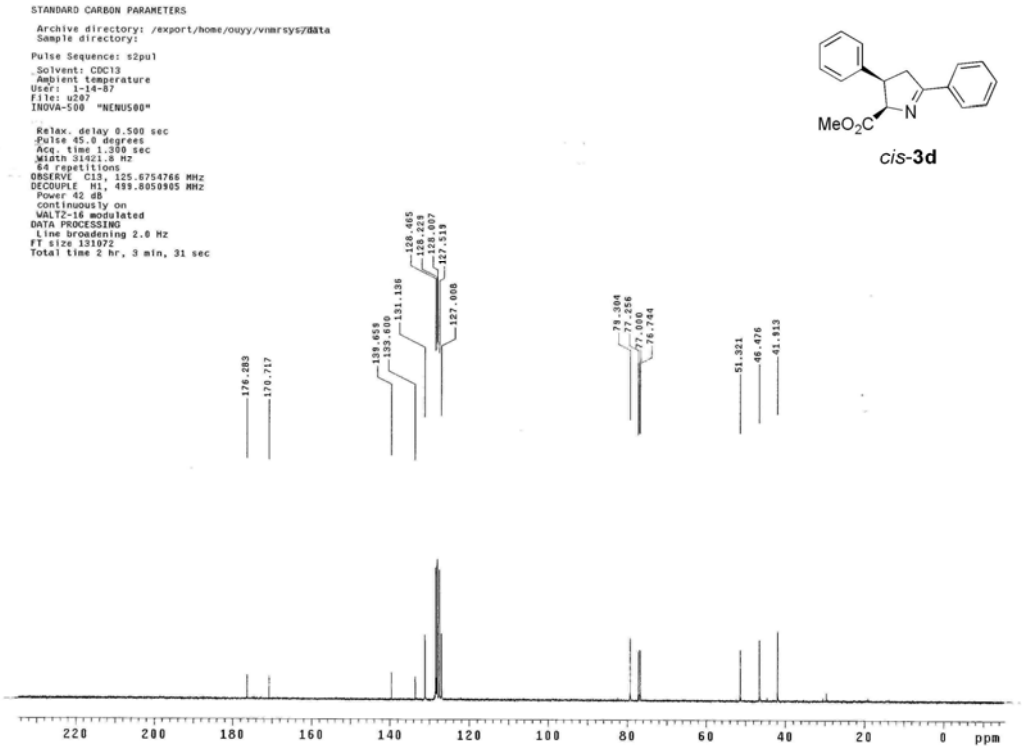

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y434
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8014.4 Hz
8 repetitions
OBSERVE H1, 499.8025874 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
O2N
MeO2C
N
cis-3c
ppm

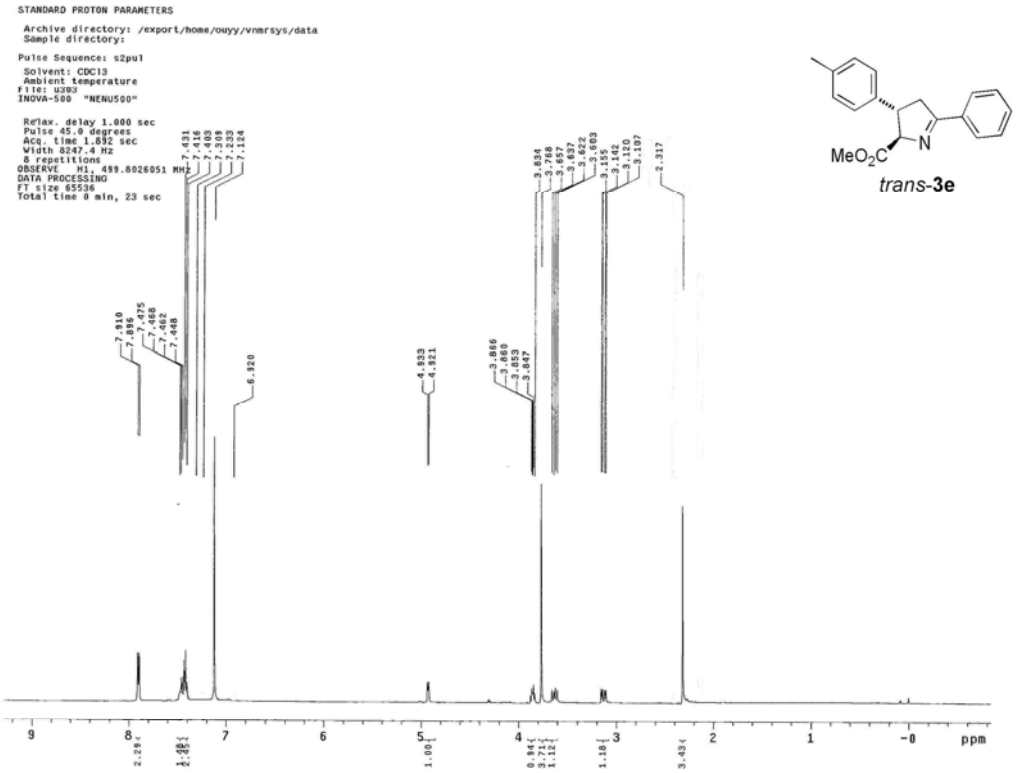

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y437
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
O2N
MeO2C
N
cis-3c
ppm

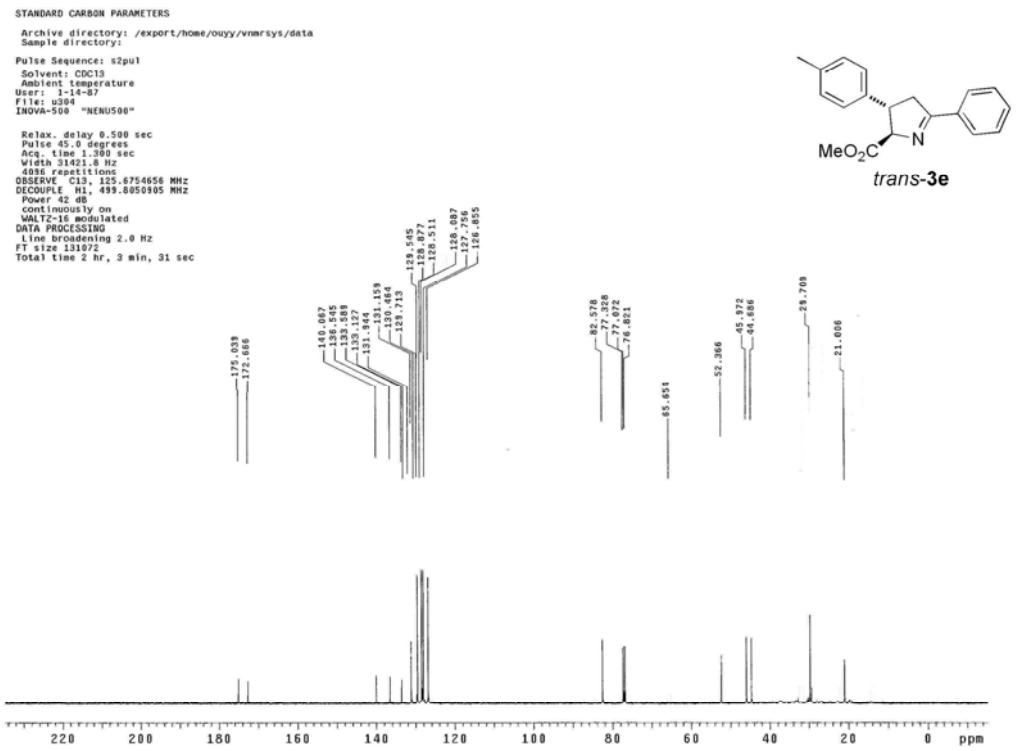

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u151
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8026026 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
trans-3d
ppm

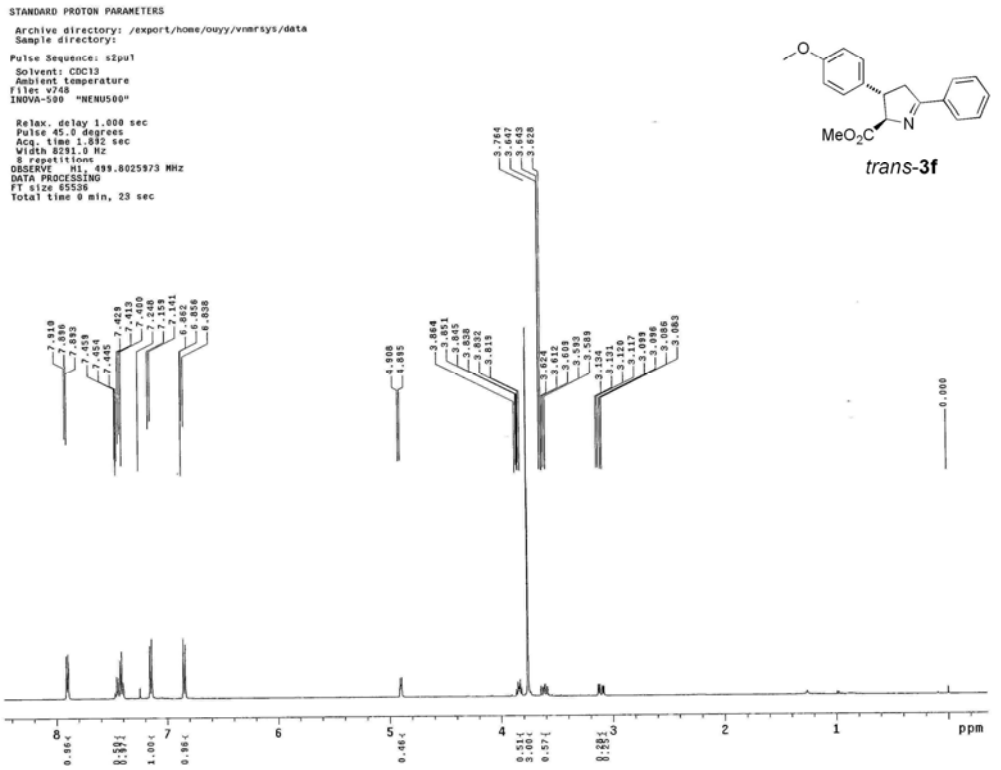

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: u206
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754886 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
trans-3d
ppm

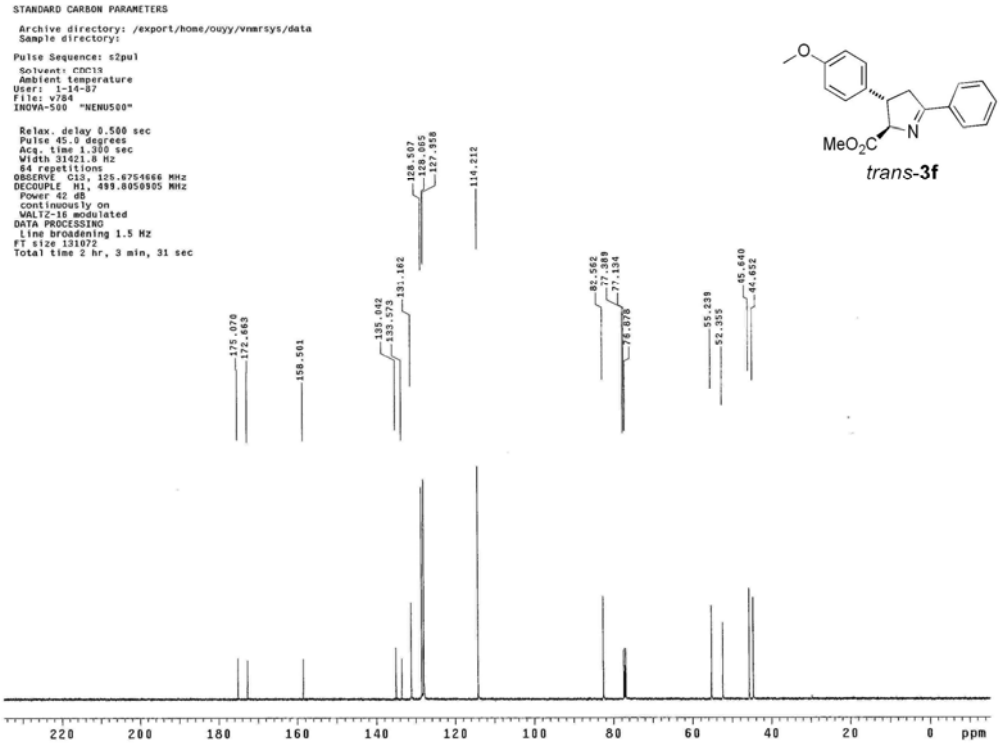

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u152
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.692 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8625955 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
cis-3d
ppm

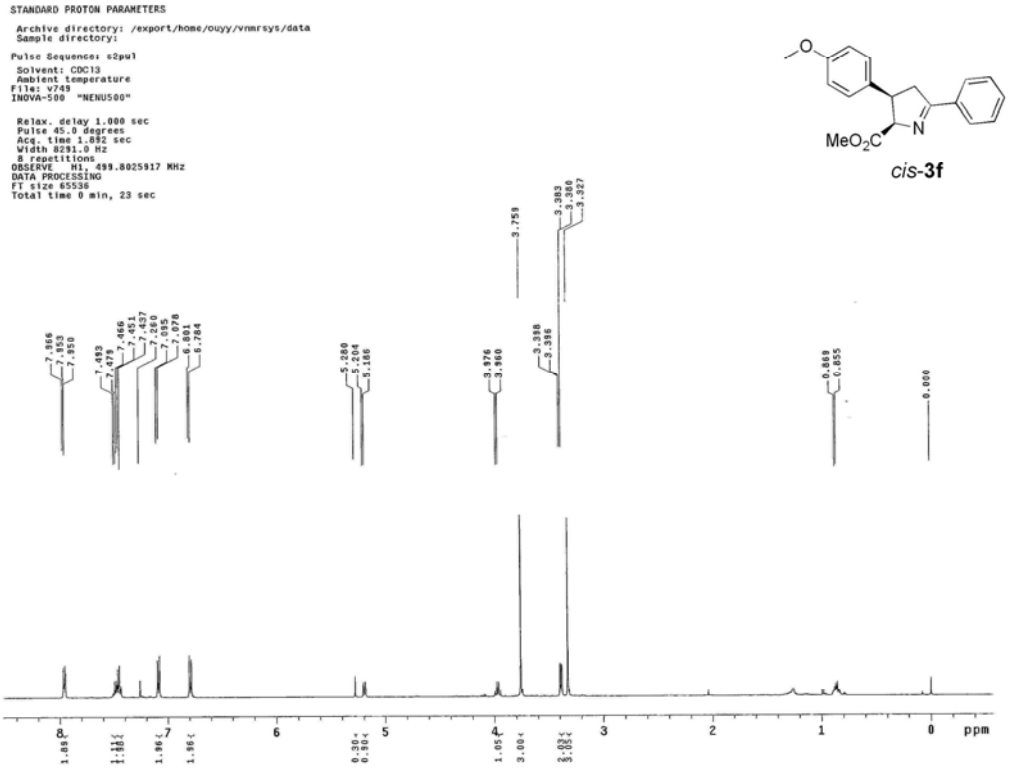

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: u207
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754766 MHz
DECOUPLE H1, 499.8050985 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
cis-3d
ppm

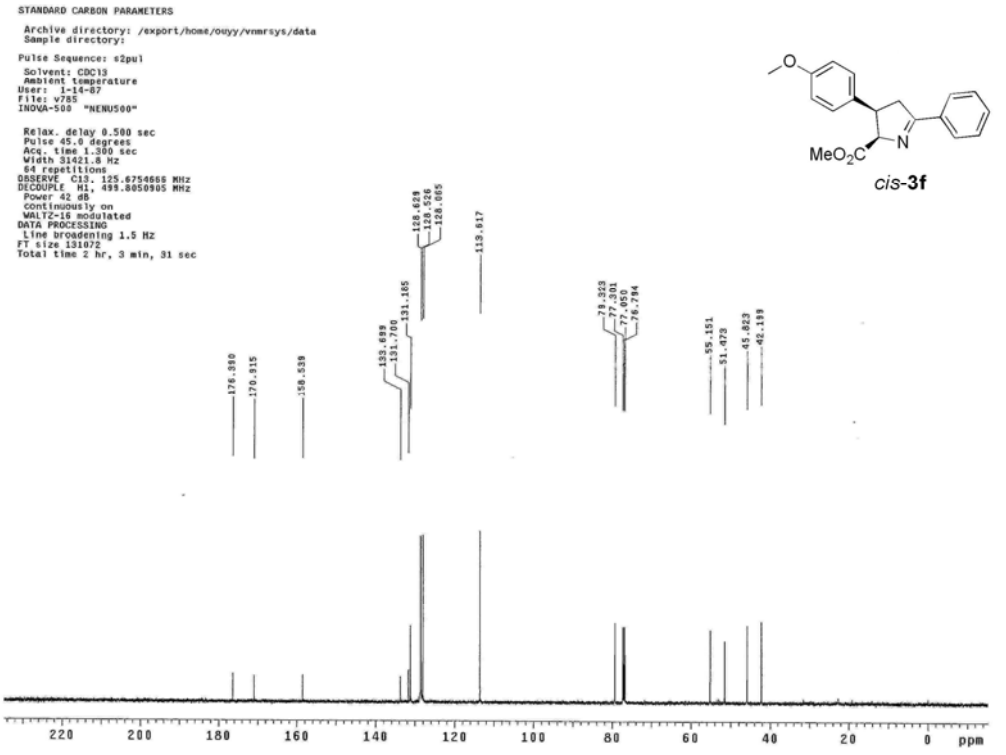

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u303
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8026051 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
trans-3e
ppm

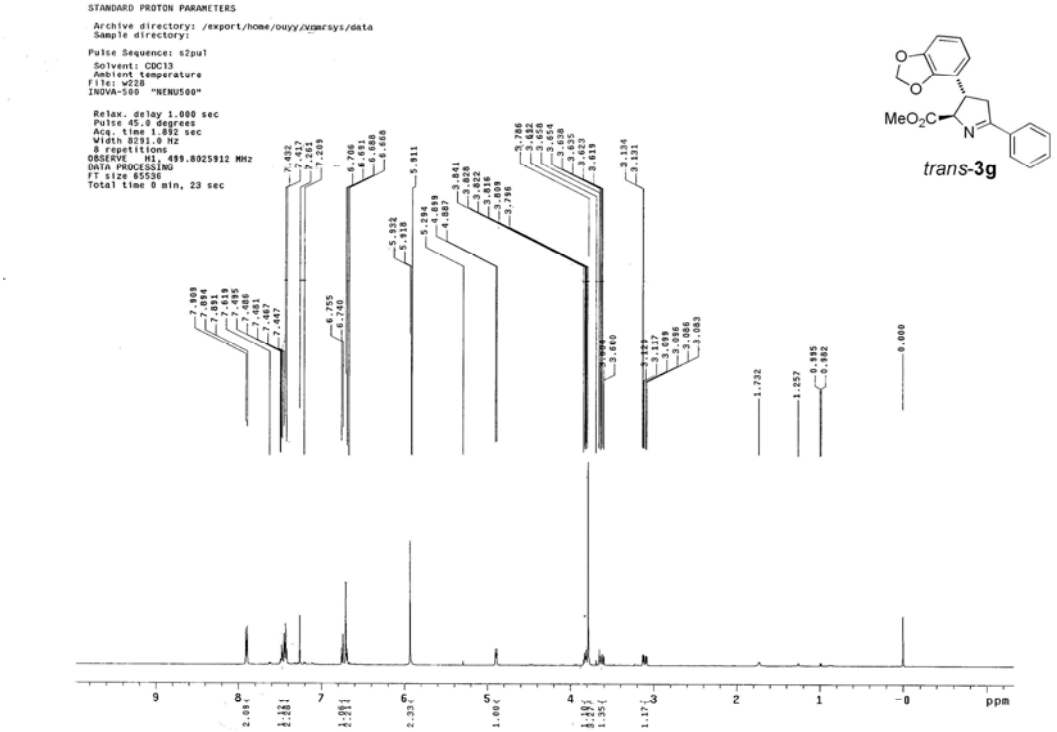

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: u304
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
trans-3e
ppm

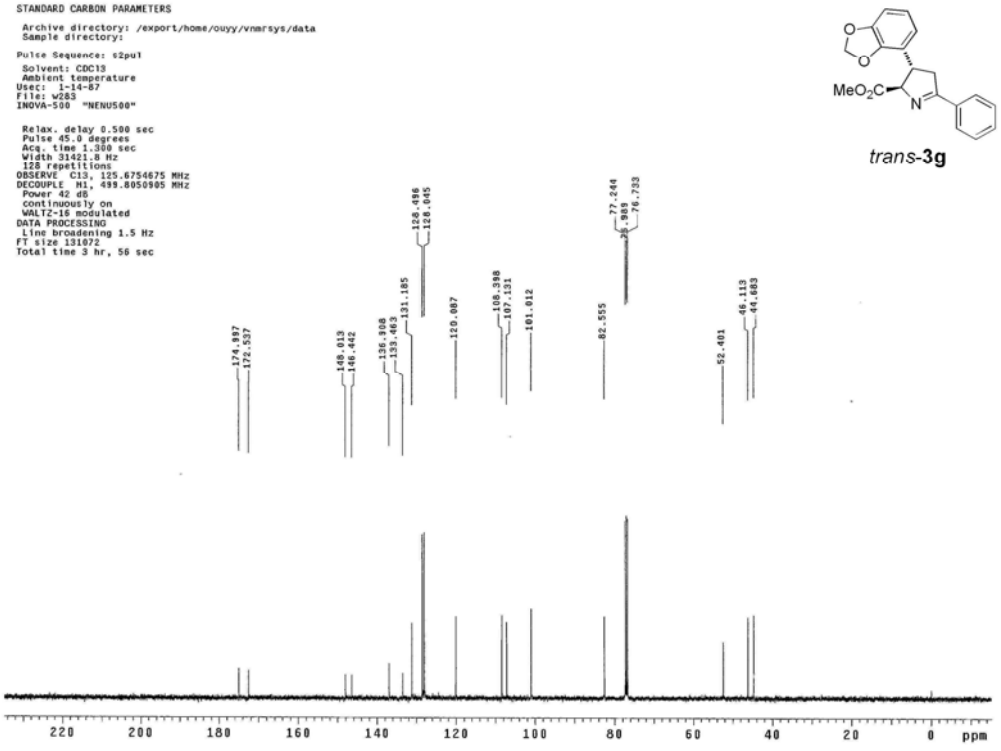

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w748
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025973 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
trans-3f
ppm

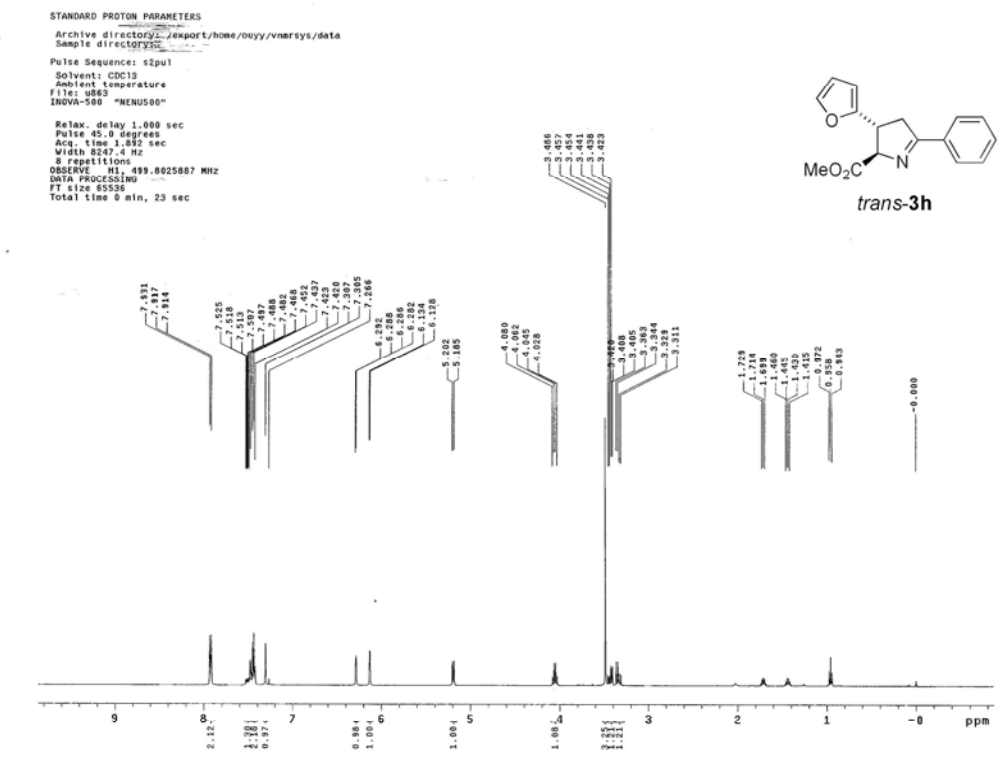

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v784
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.0 Hz
64 repetitions
OBSERVE C13, 125.6754666 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
trans-3f
ppm

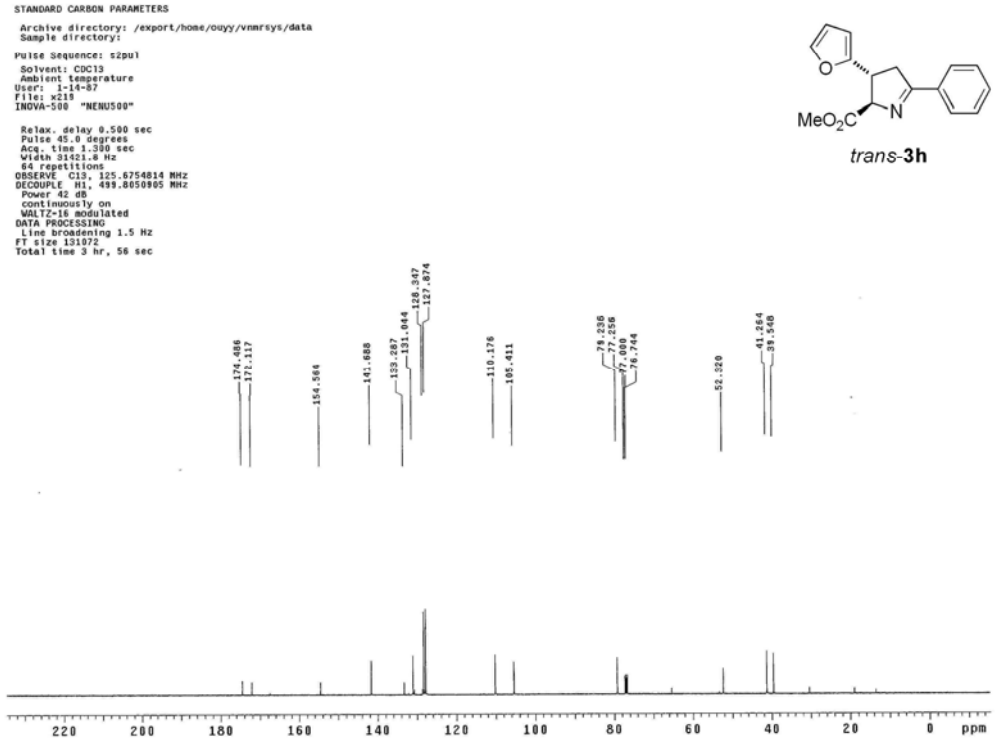

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v749
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025917 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
cis-3f
ppm

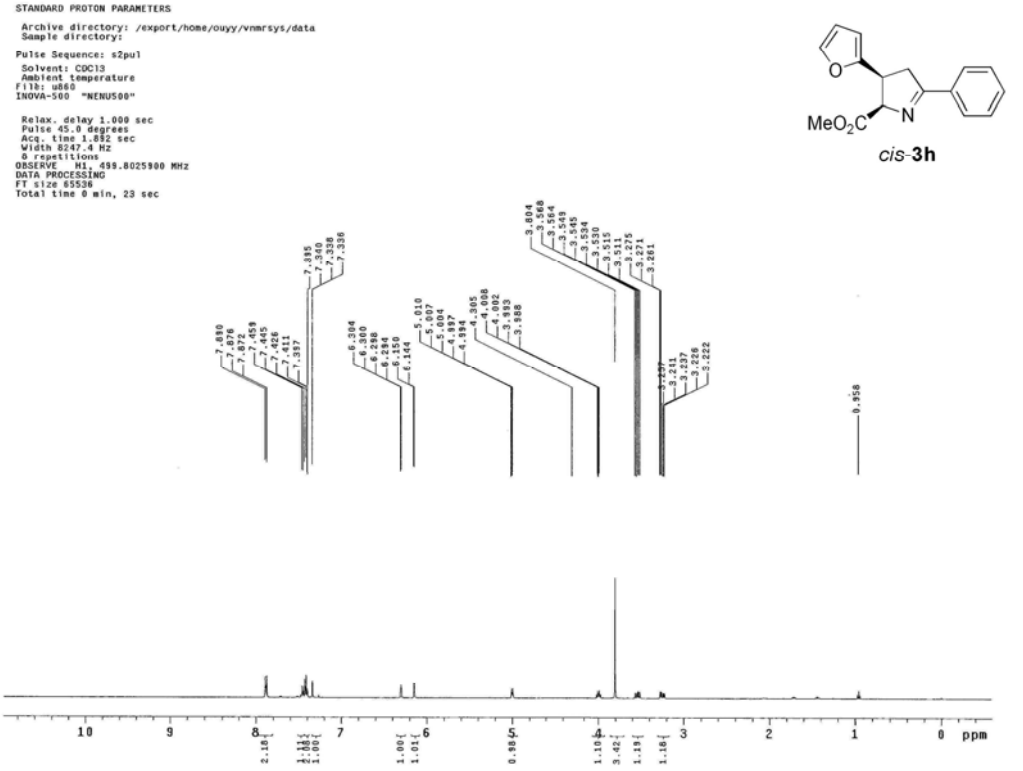

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: v785
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754666 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
cis-3f
ppm

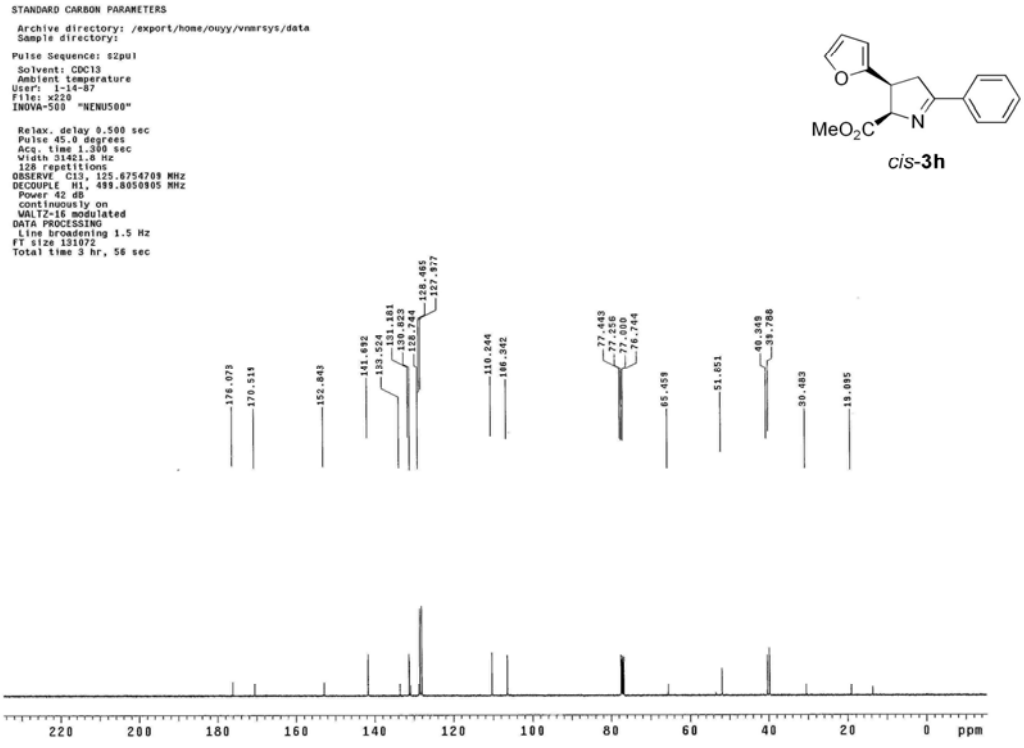

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w228
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025912 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
trans-3g
ppm

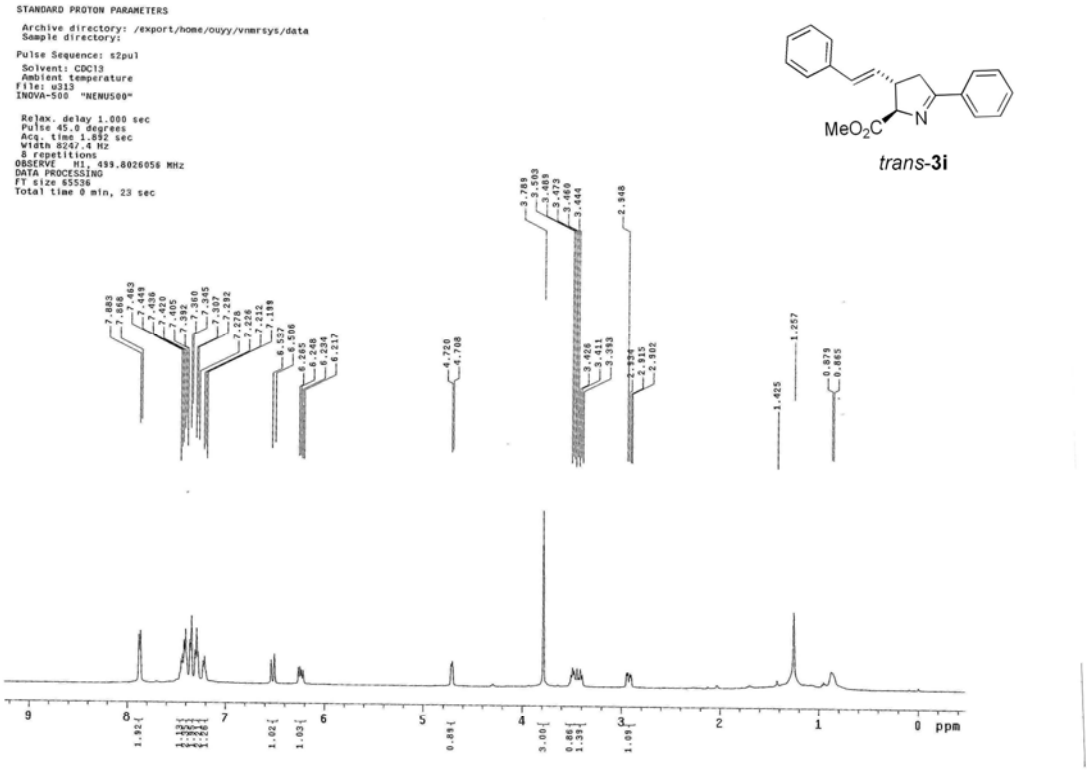

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Uxec: 1-14-87
File: w283
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
trans-3g
ppm

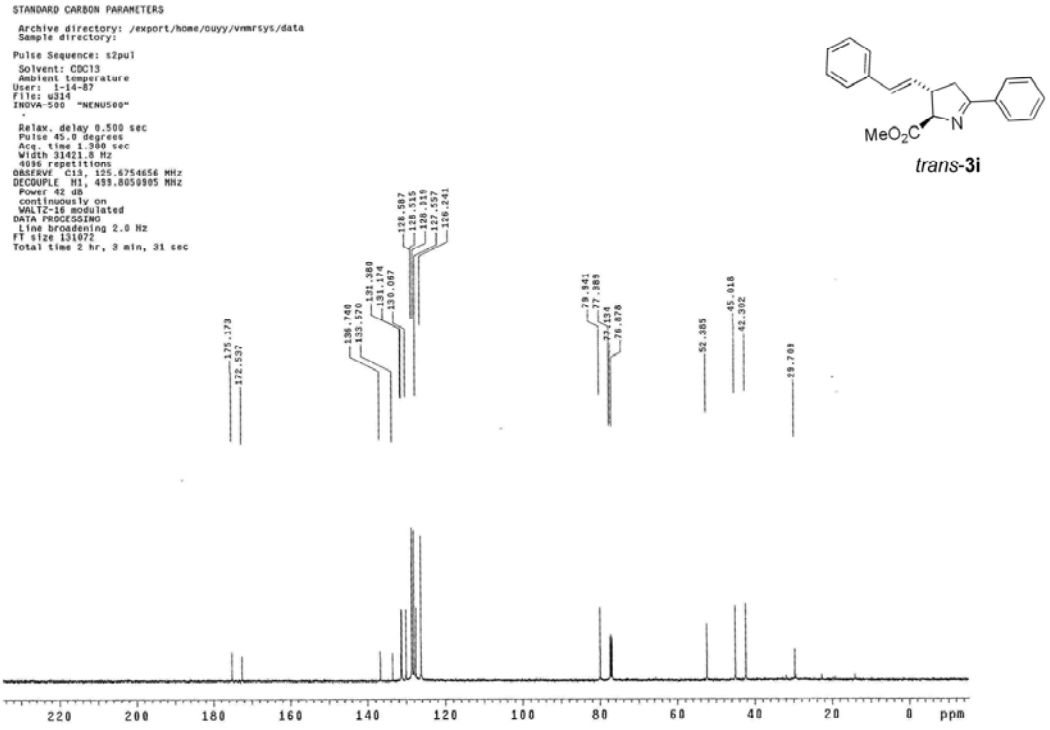

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u863
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8025887 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
trans-3h
ppm

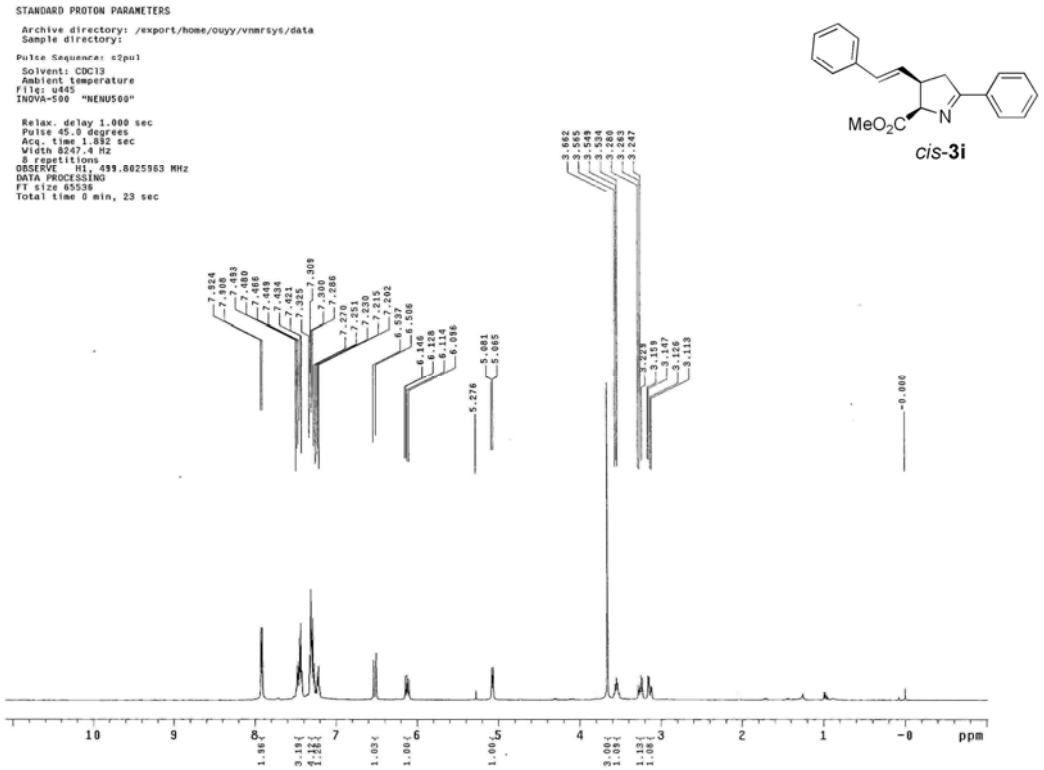

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: x219
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754814 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
trans-3h
ppm

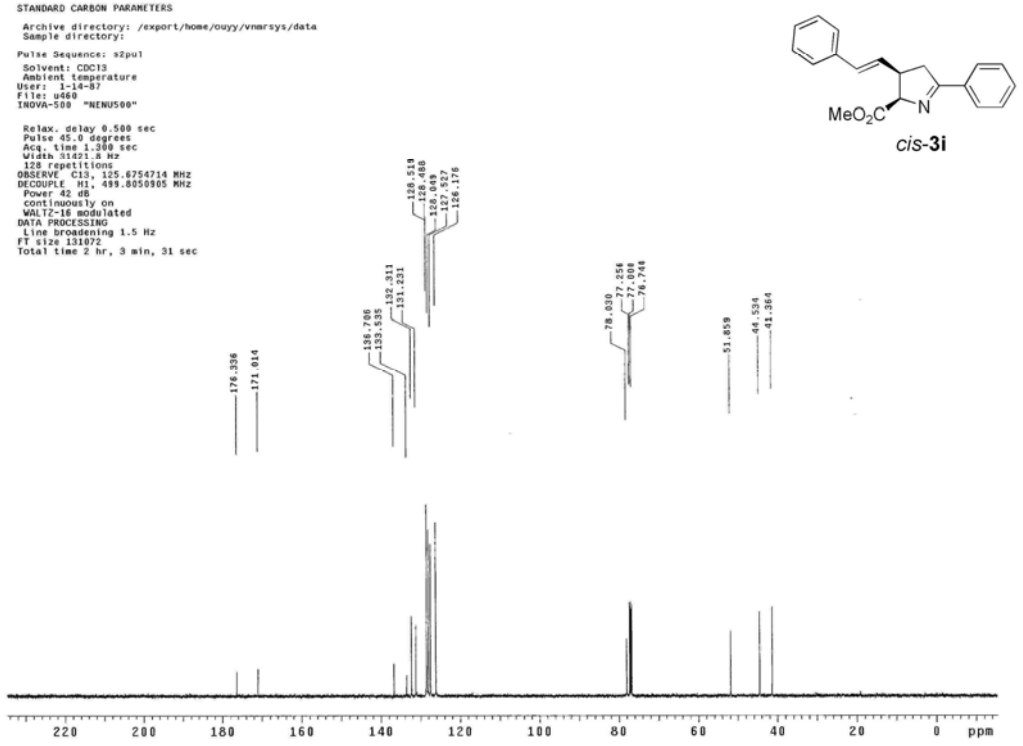

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u660
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
cis-3h
ppm

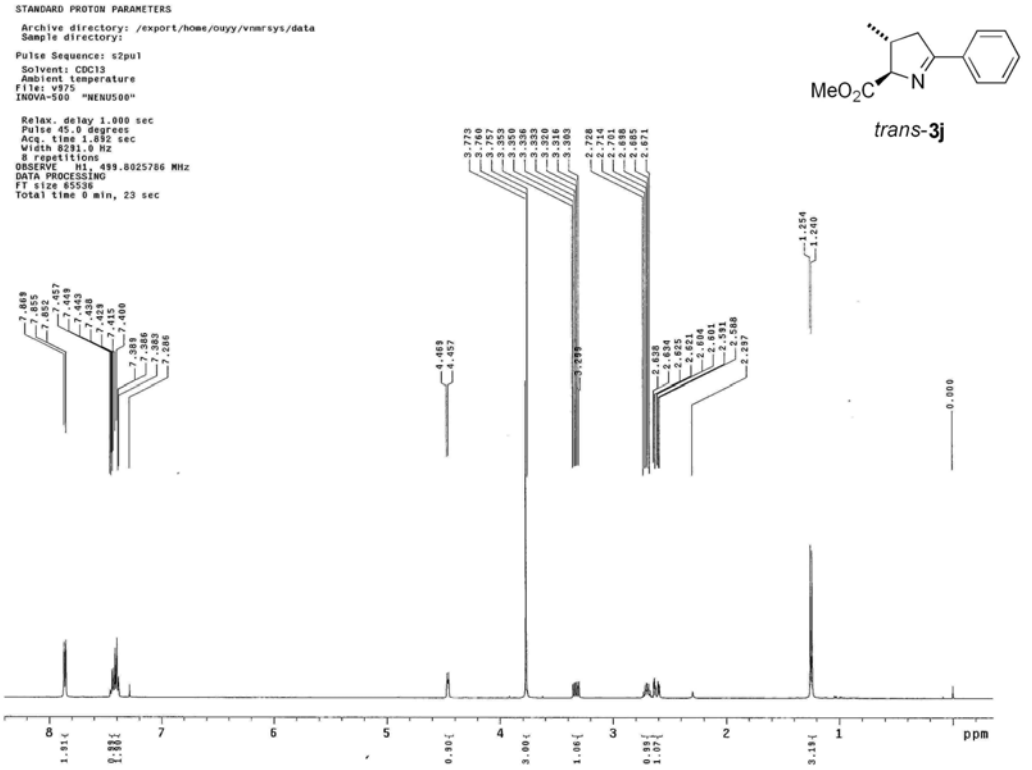

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: x220
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
cis-3h
ppm

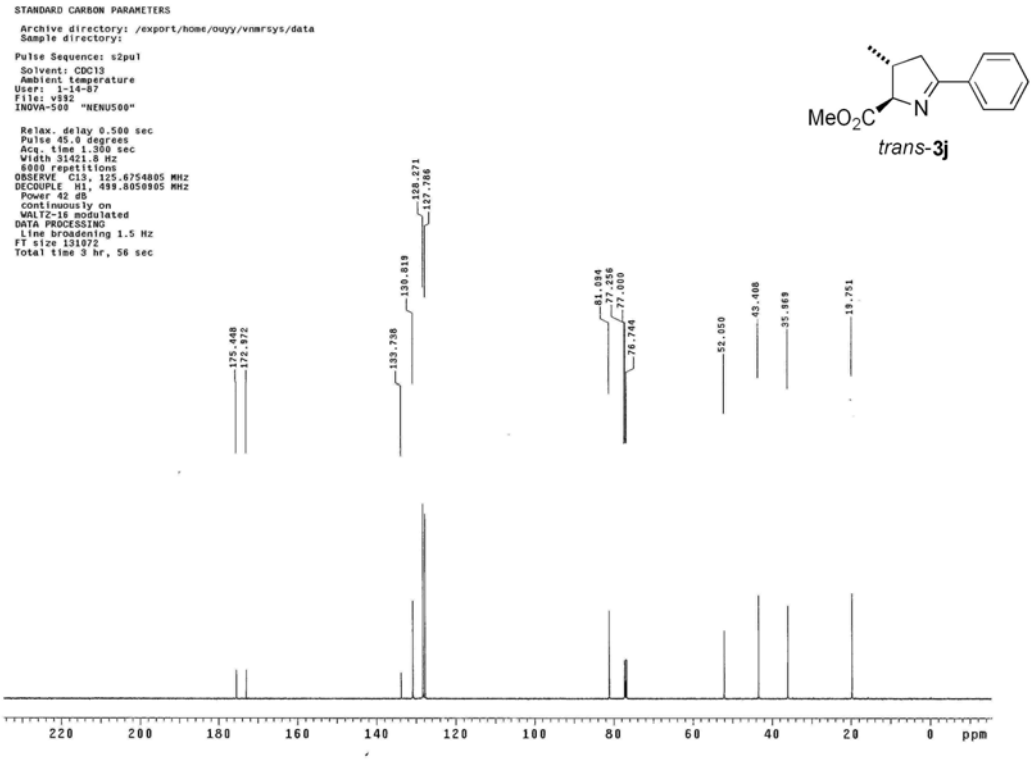

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w313
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8026056 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
trans-3i
ppm

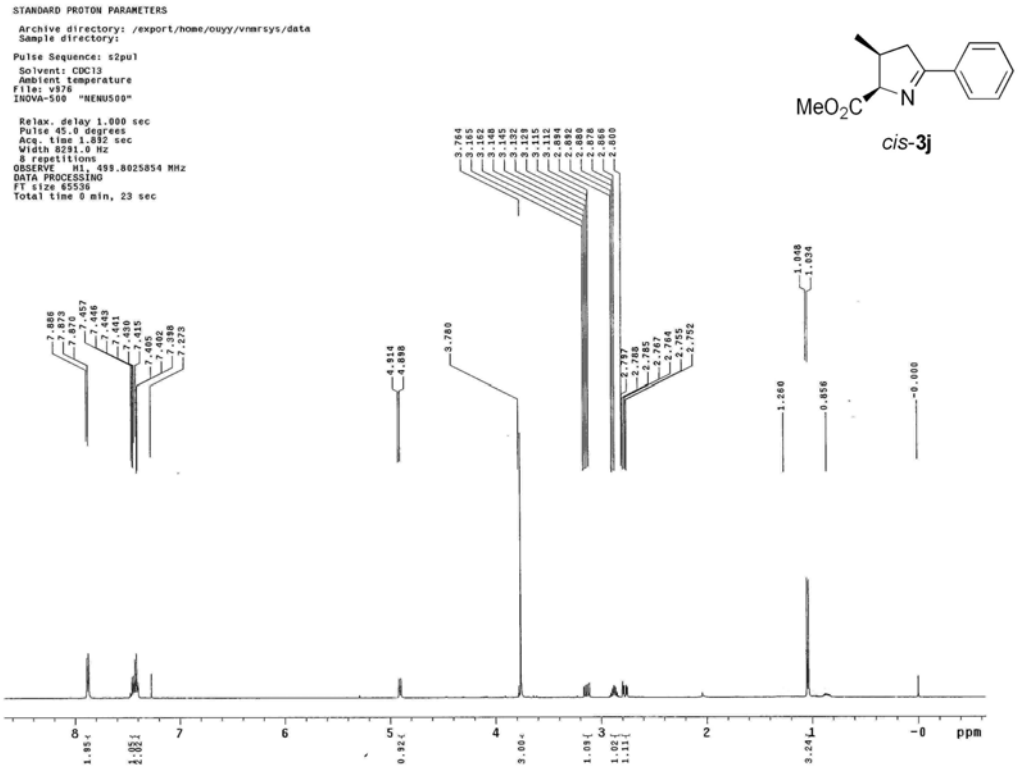

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: w314
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
trans-3i
ppm

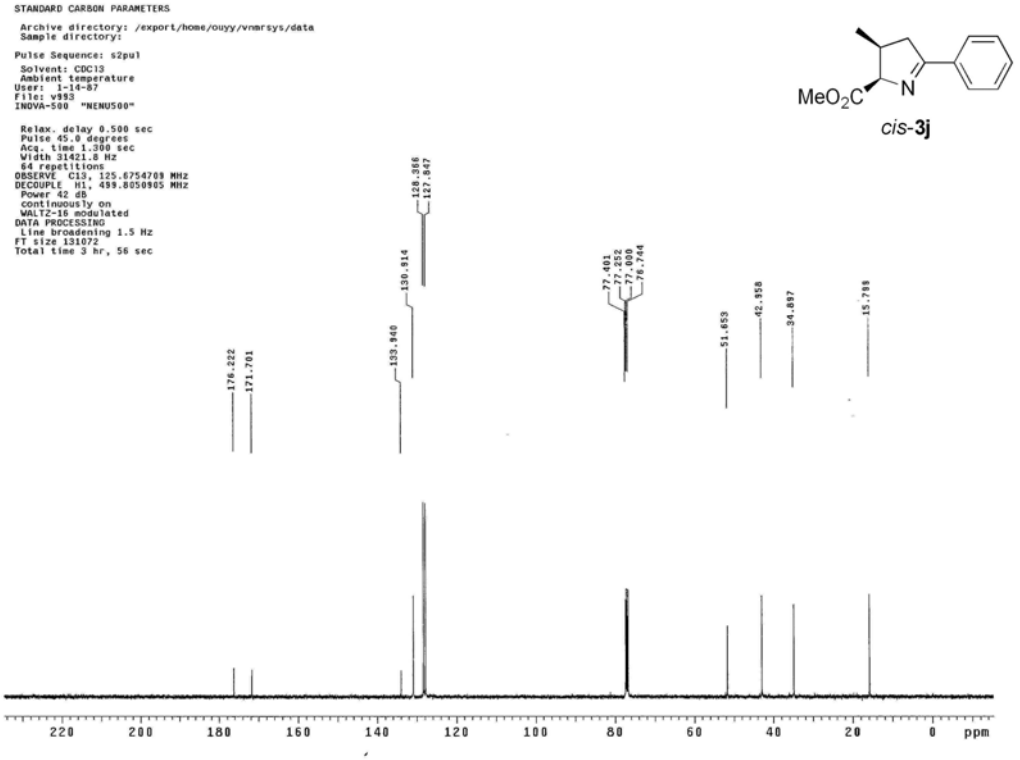

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u445
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8025963 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
cis-3i
ppm

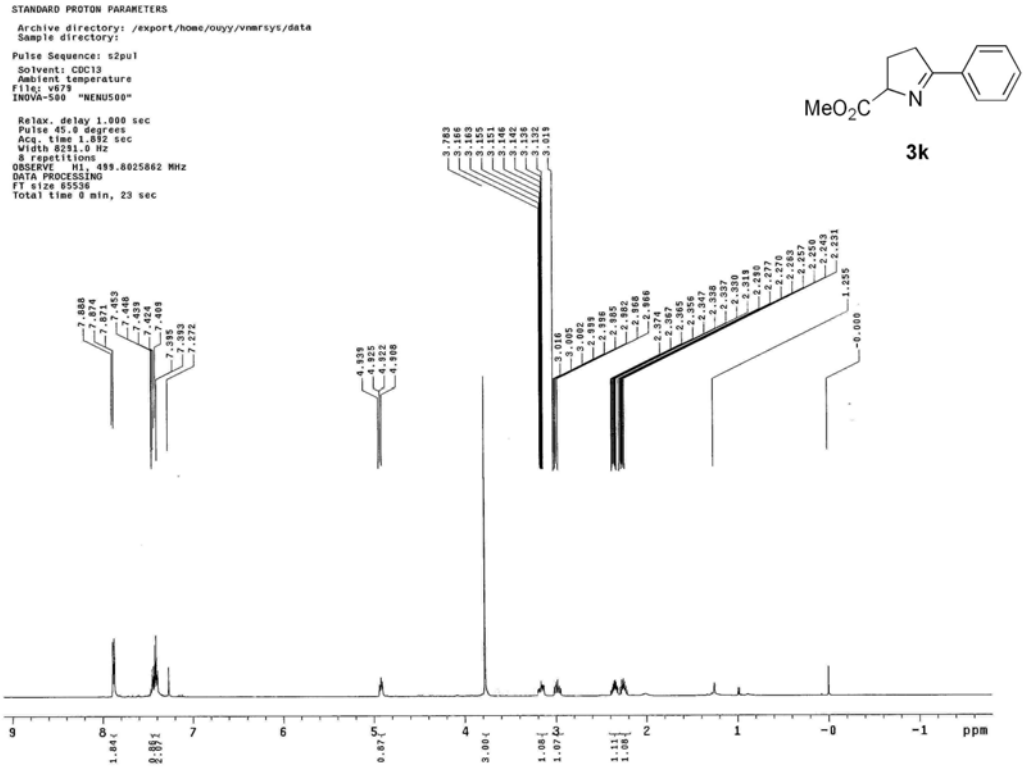

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: u460
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
cis-3i
ppm

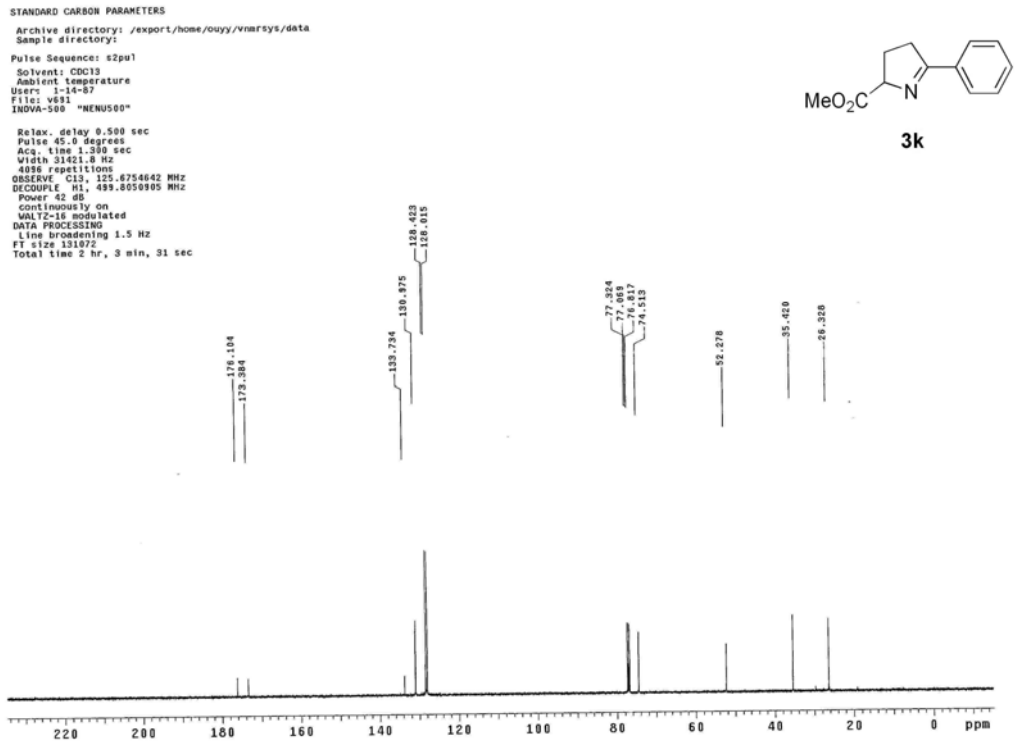

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v975
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025786 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
trans-3j
ppm

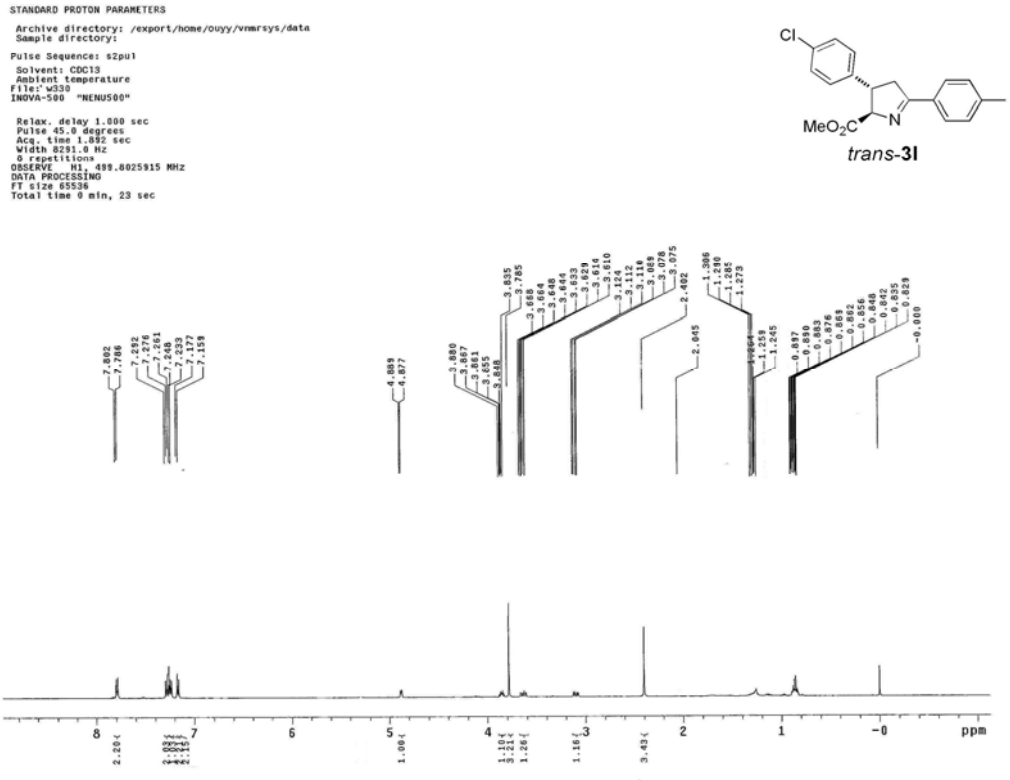

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v332
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754805 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
trans-3j
ppm

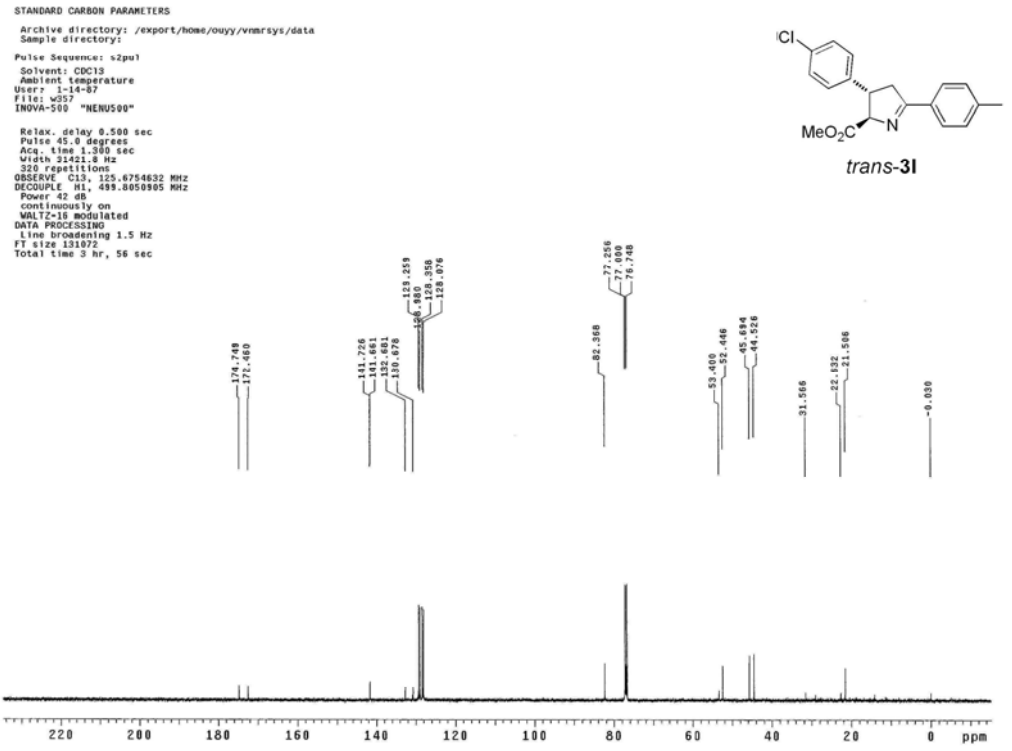

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v976
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025854 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
cis-3j
ppm

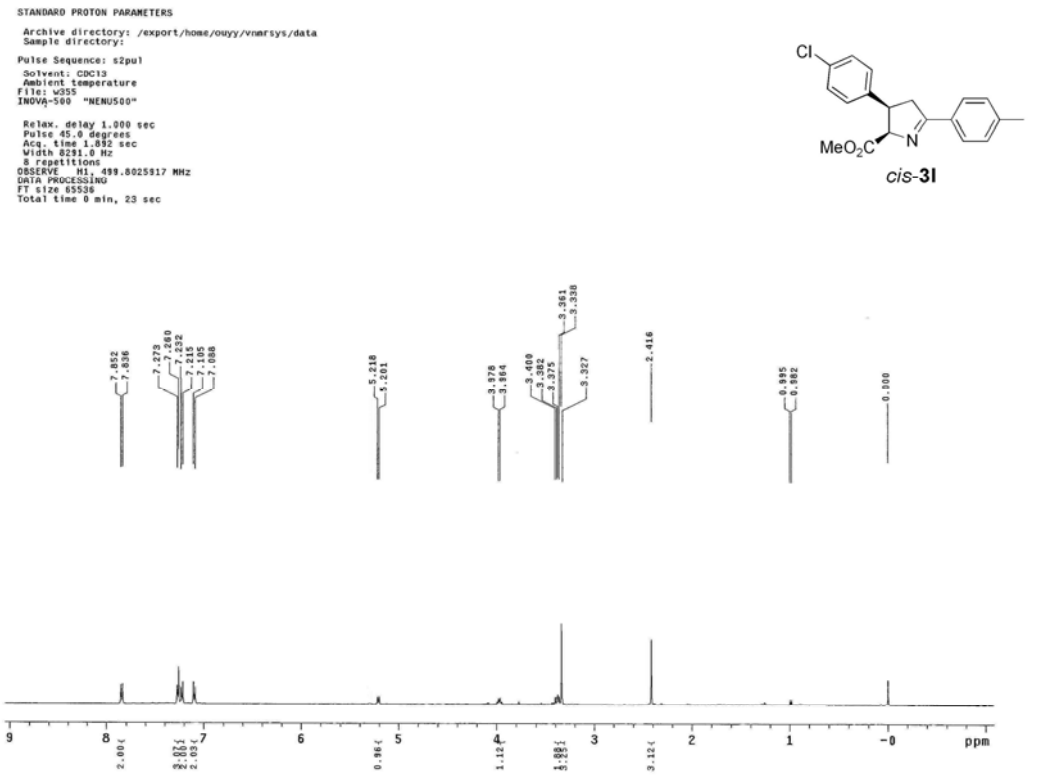

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v993
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
cis-3j
ppm

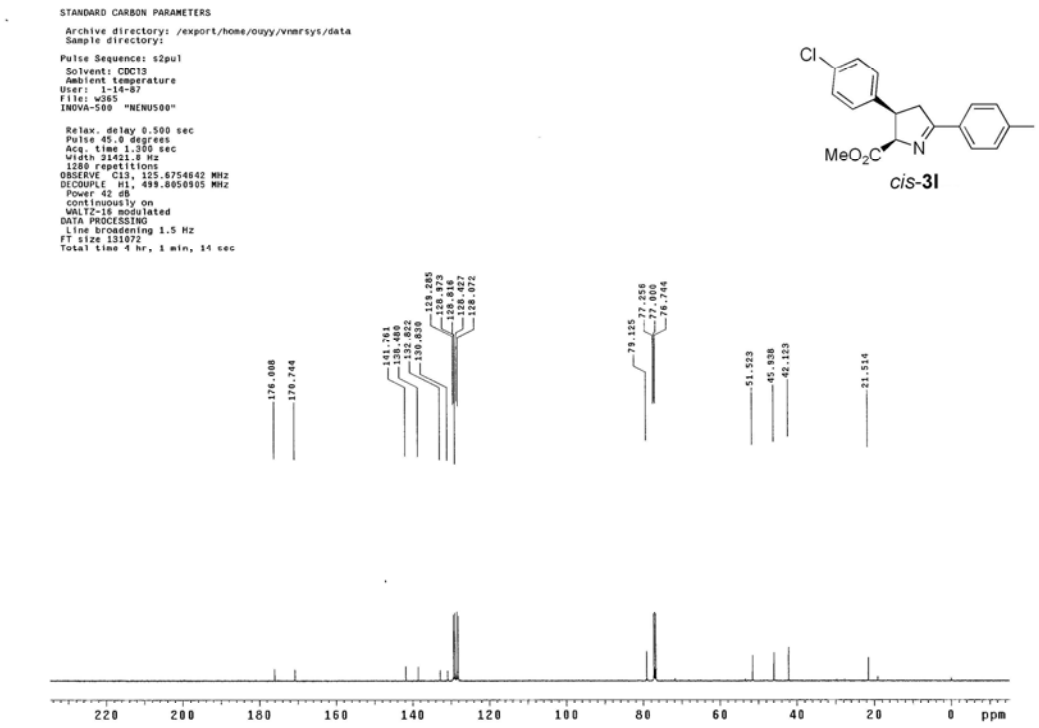

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v679
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025862 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
3k
ppm

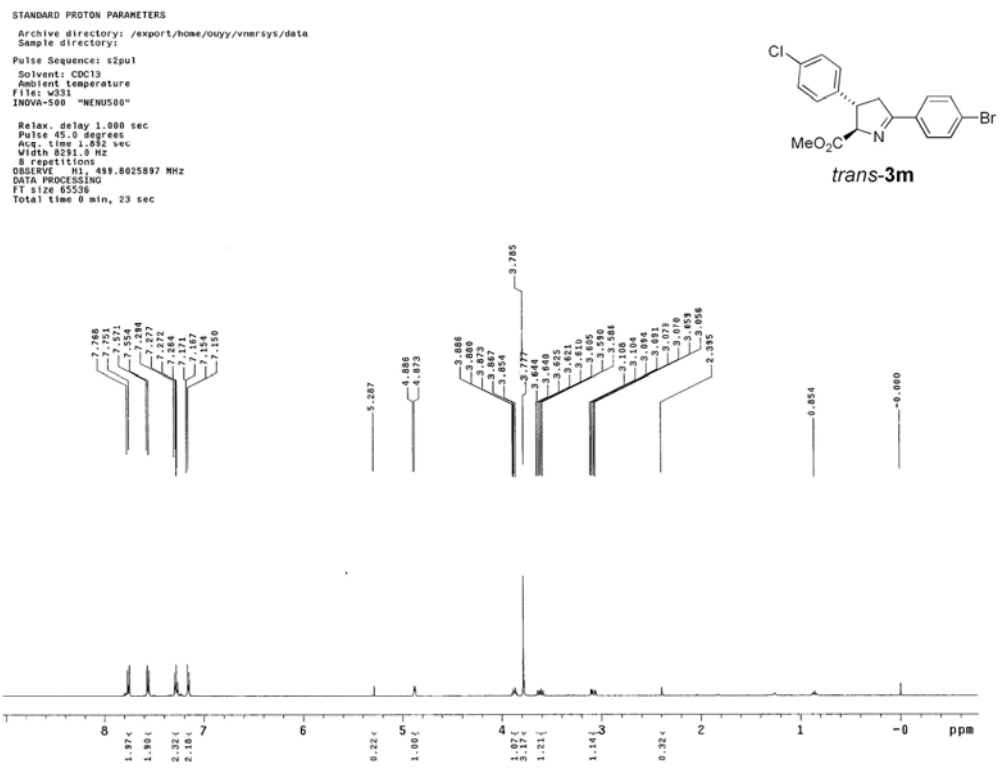

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Users 1-14-87
File: v691
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
3k
ppm

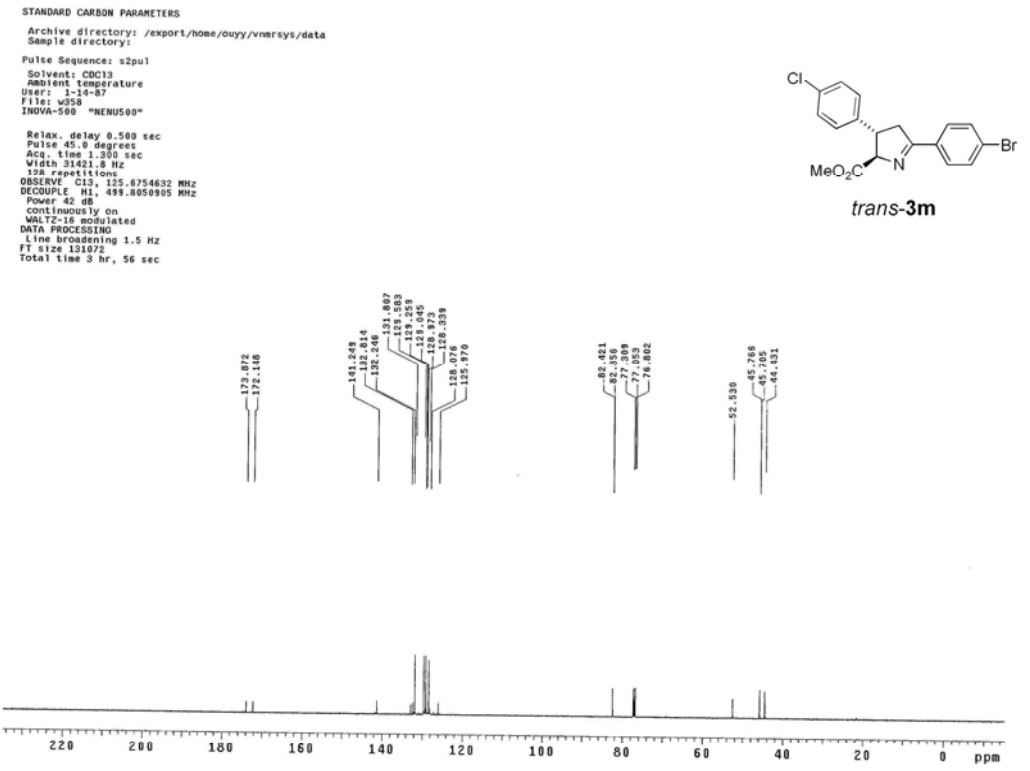

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File:w330
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025915 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
MeO2C
N
trans-3l
ppm

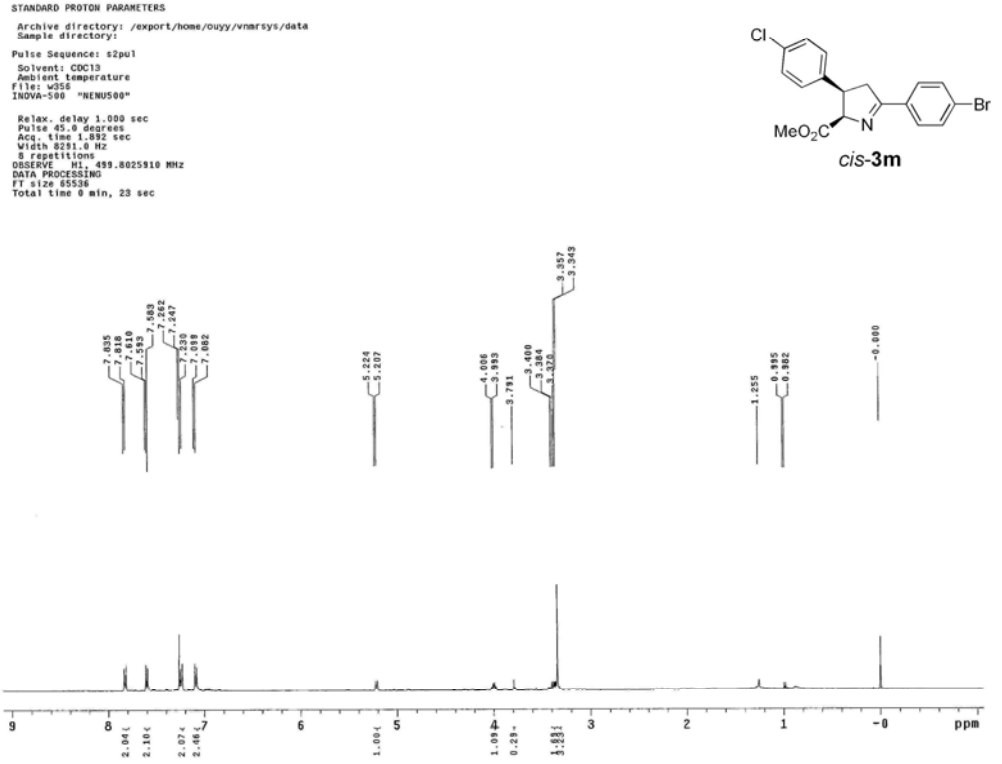

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: w357
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
MeO2C
N
trans-3l
ppm

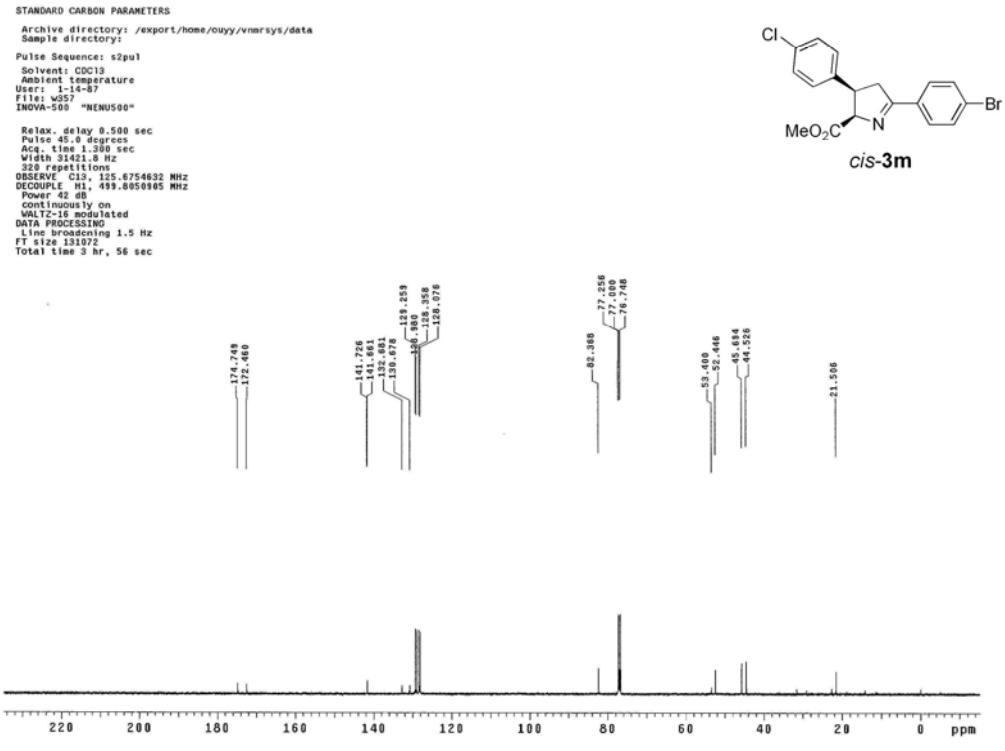

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w355
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025917 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
MeO2C
N
cis-3l
7.852
7.836
7.279
7.260
7.232
7.215
7.195
7.088
5.218
5.201
3.978
3.964
3.400
3.382
3.375
3.361
3.338
3.327
2.416
0.995
0.982
0.000
9
8
7
6
5
4
3
2
1
-0
ppm

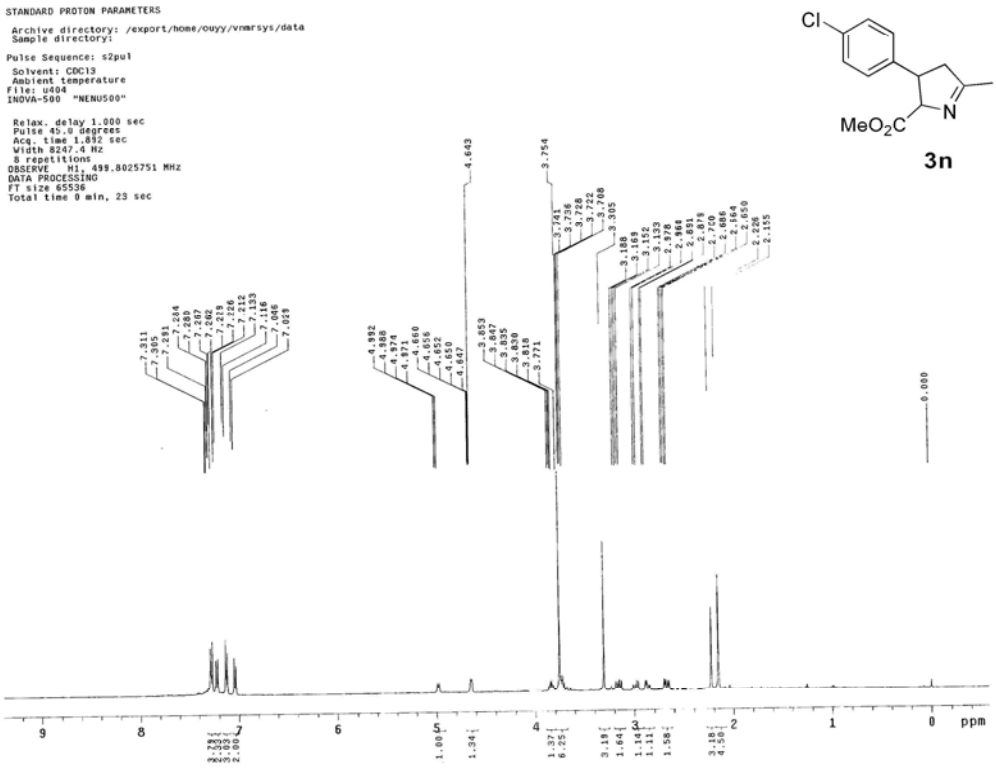

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: w365
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1280 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 4 hr, 1 min, 14 sec
Cl
MeO2C
N
cis-3l
170.808
170.744
161.761
138.465
132.822
130.830
129.285
129.173
128.816
128.427
128.072
79.125
77.256
77.000
76.744
51.523
45.938
42.123
21.514
220
200
180
160
140
120
100
80
60
40
20
0
ppm

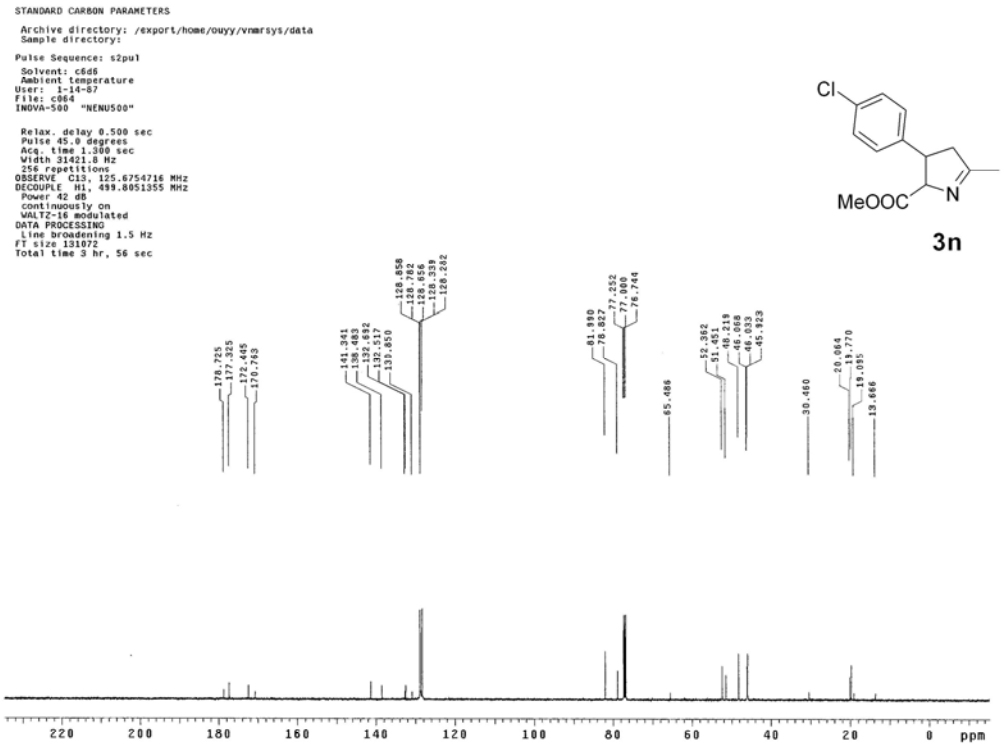

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w331
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.652 sec
Width 8281.0 Hz
8 repetitions
OBSERVE H1, 499.8025897 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
Br
MeO2C
N
trans-3m
ppm

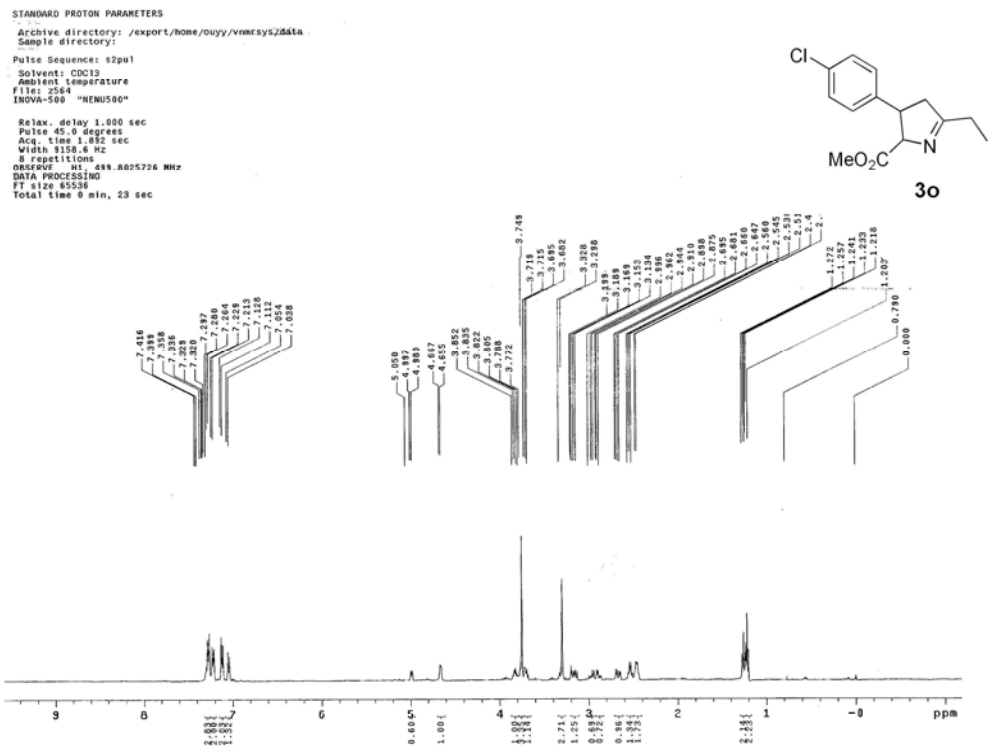

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: w358
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
Br
MeO2C
N
trans-3m
ppm

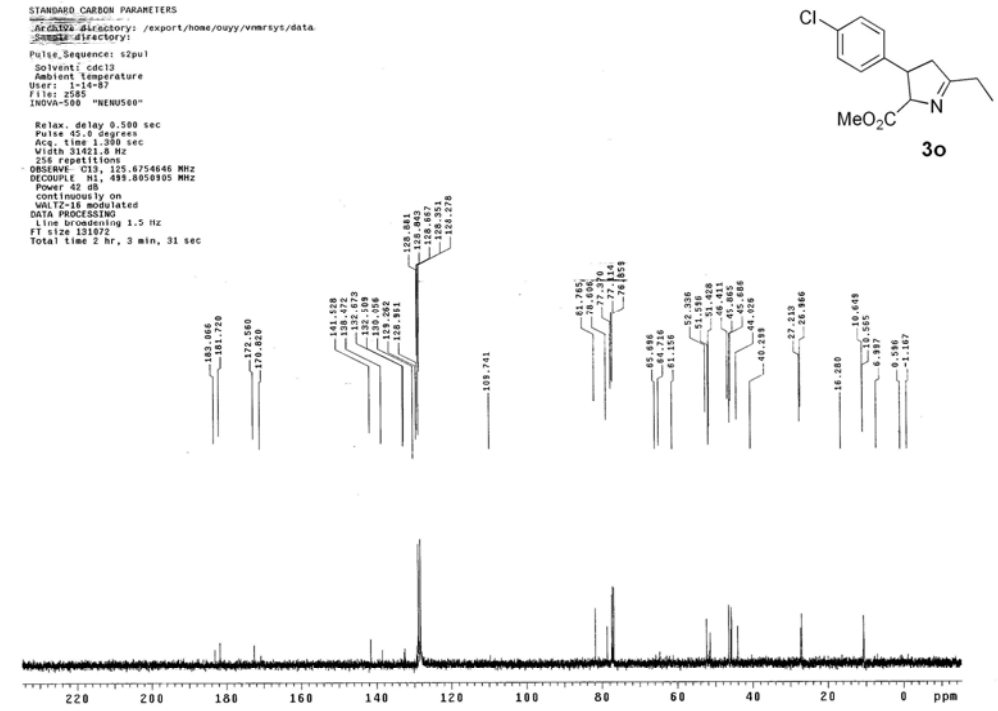

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
file: w356
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025910 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
Br
MeO2C
N
cis-3m
ppm

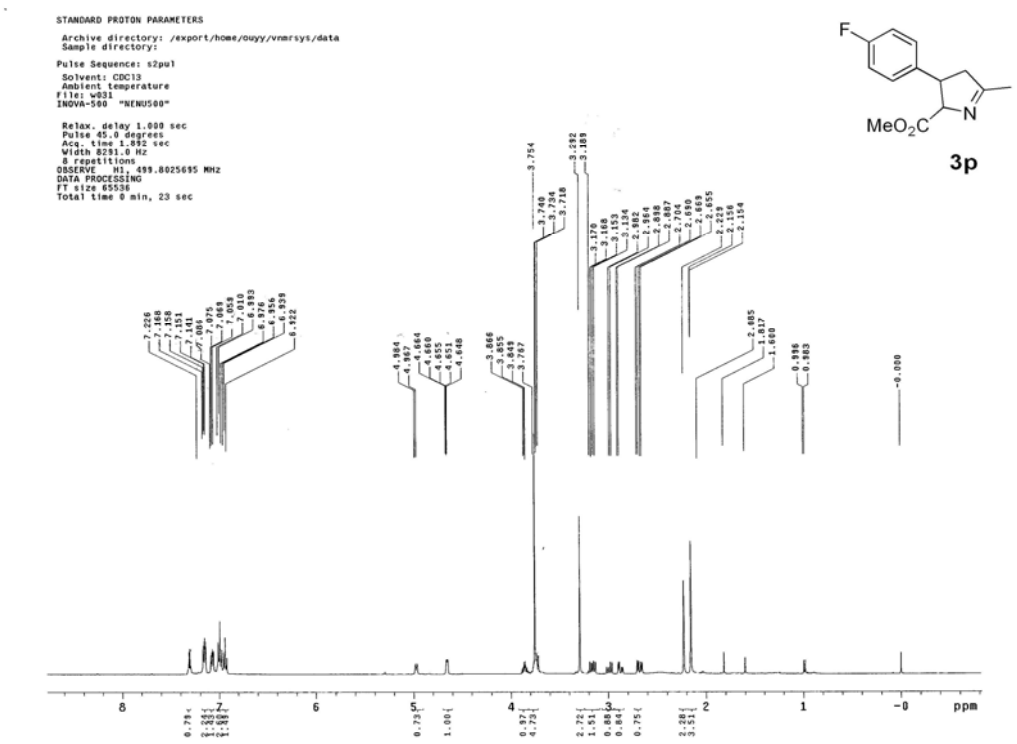

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
file: w357
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
Br
MeO2C
N
cis-3m
ppm

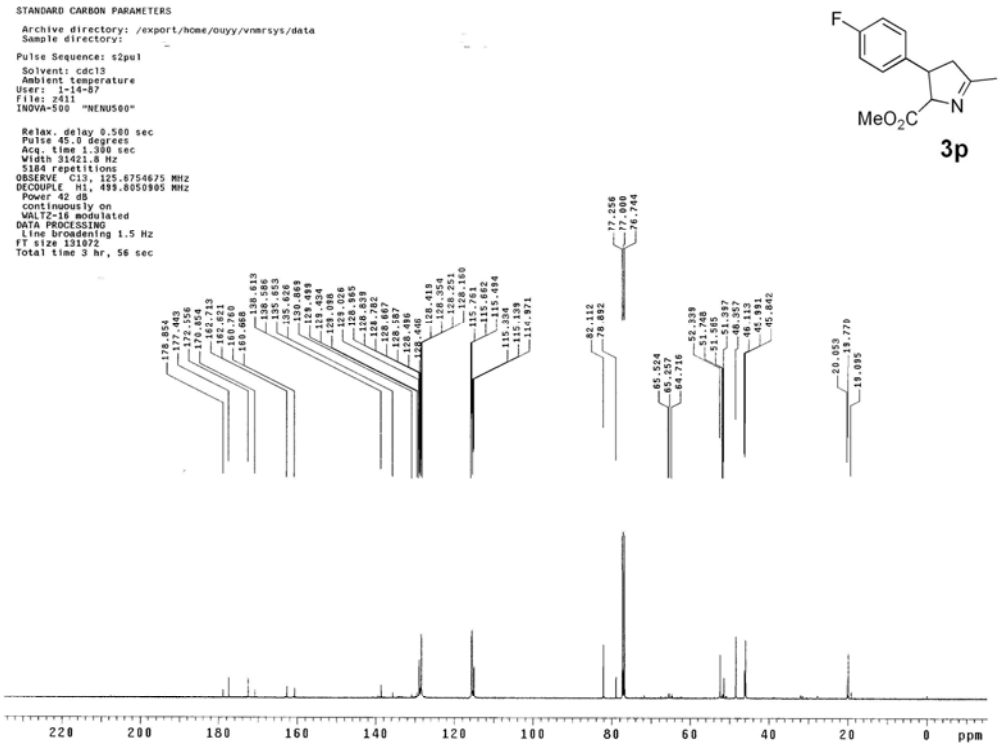

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u404
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8025751 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
MeO2C
N
3n
ppm

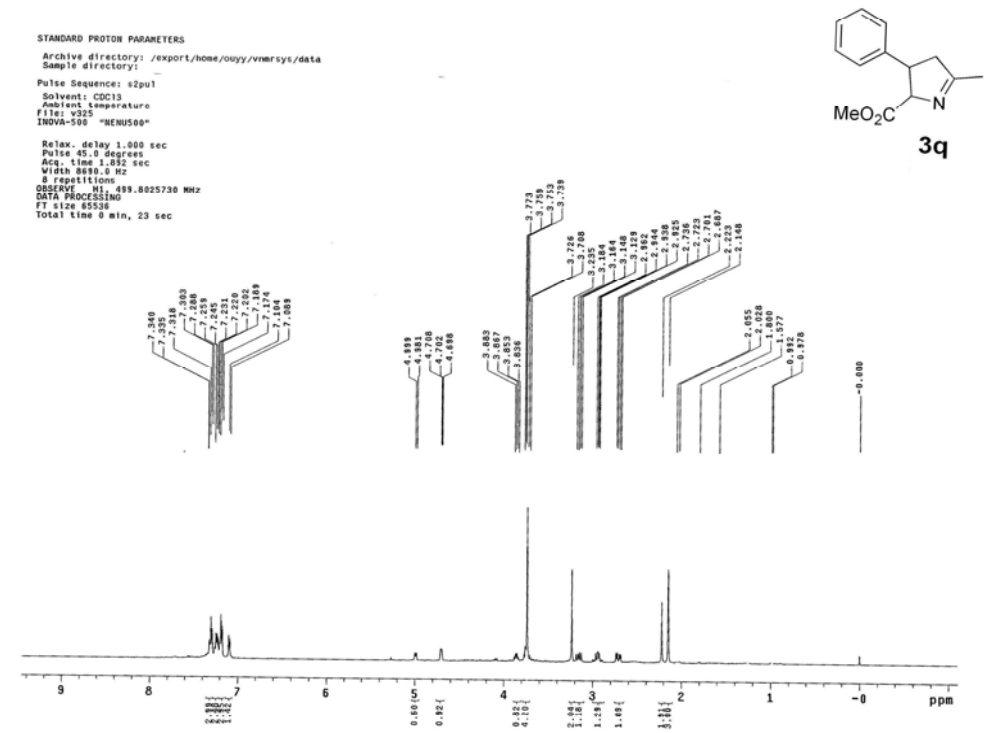

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
User: 1-14-87
File: c064
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754716 MHz
DECOUPLE H1, 499.8051355 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
MeOOC
N
3n
ppm

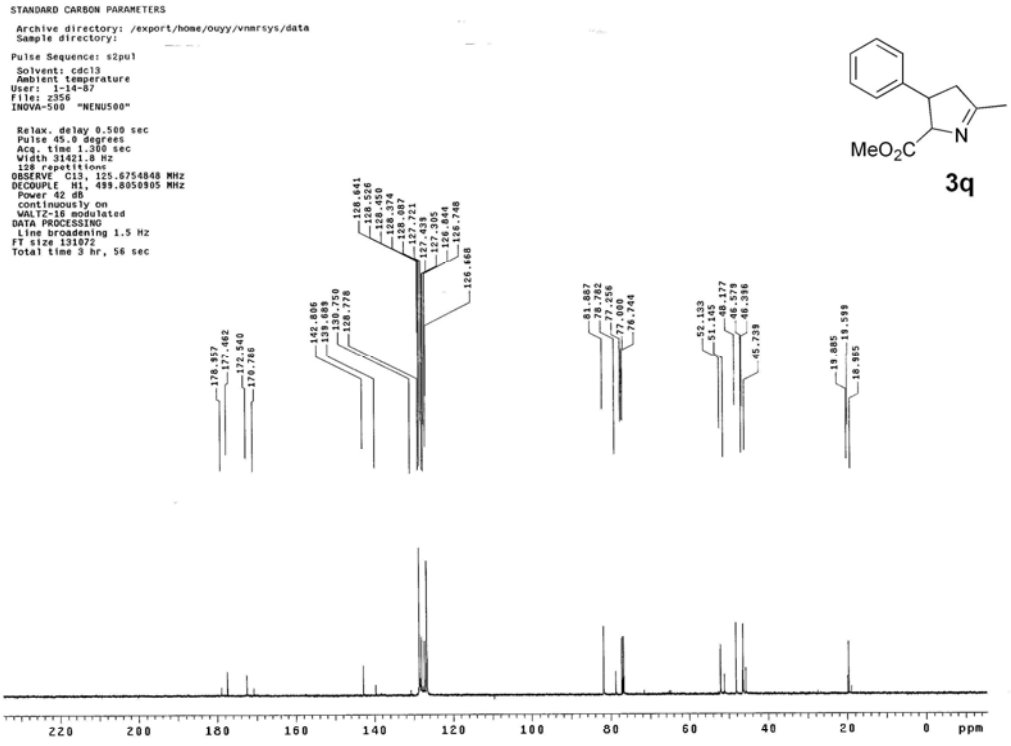

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z564
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9158.6 Hz
8 repetitions
OBSERVE H1, 499.8825726 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
MeO2C
N
3o
ppm

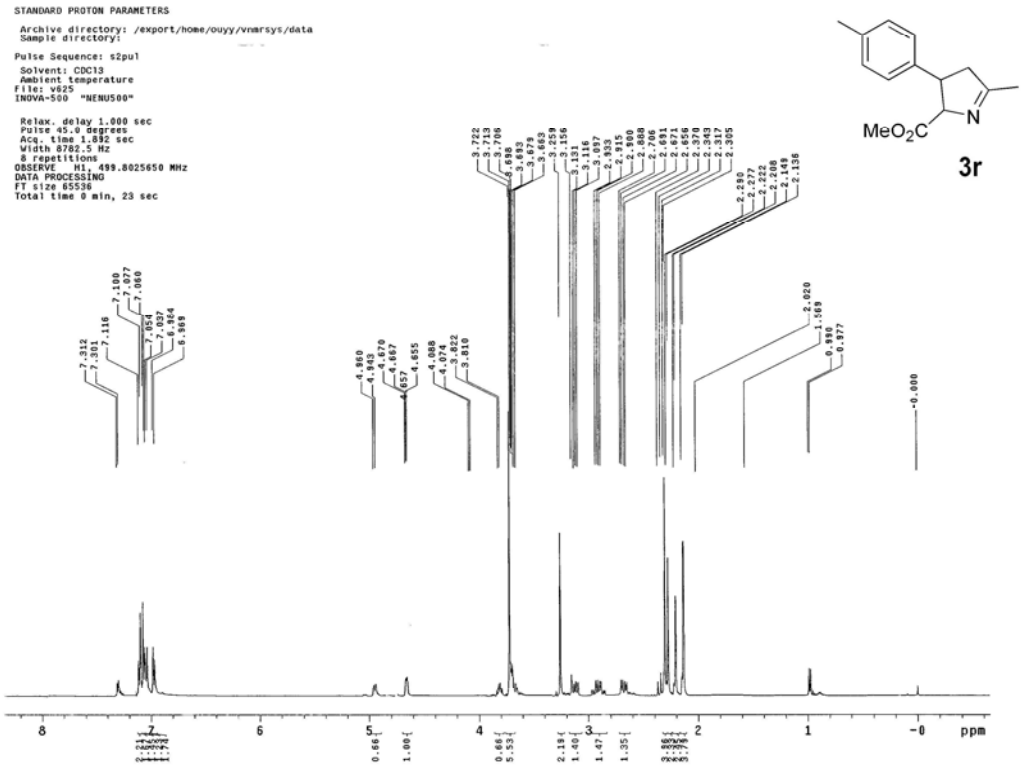

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z585
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050305 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Cl
MeO2C
N
3o
ppm

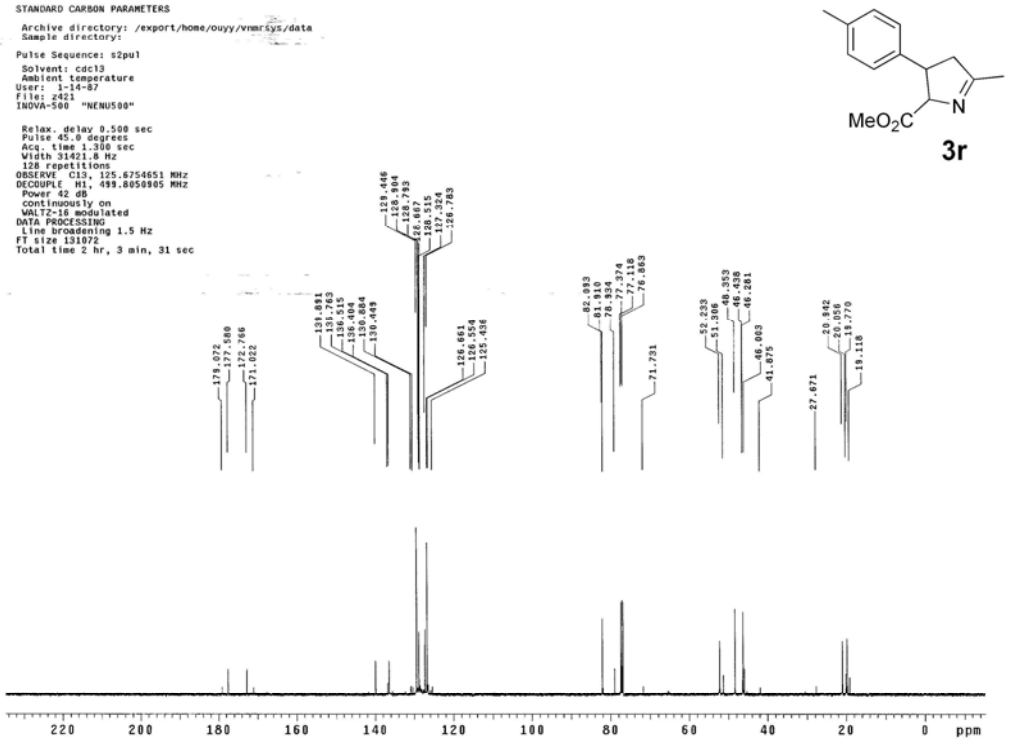

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: w831
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8291.0 Hz
8 repetitions
OBSERVE H1, 499.8025695 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
F
MeO2C
N
3p
ppm

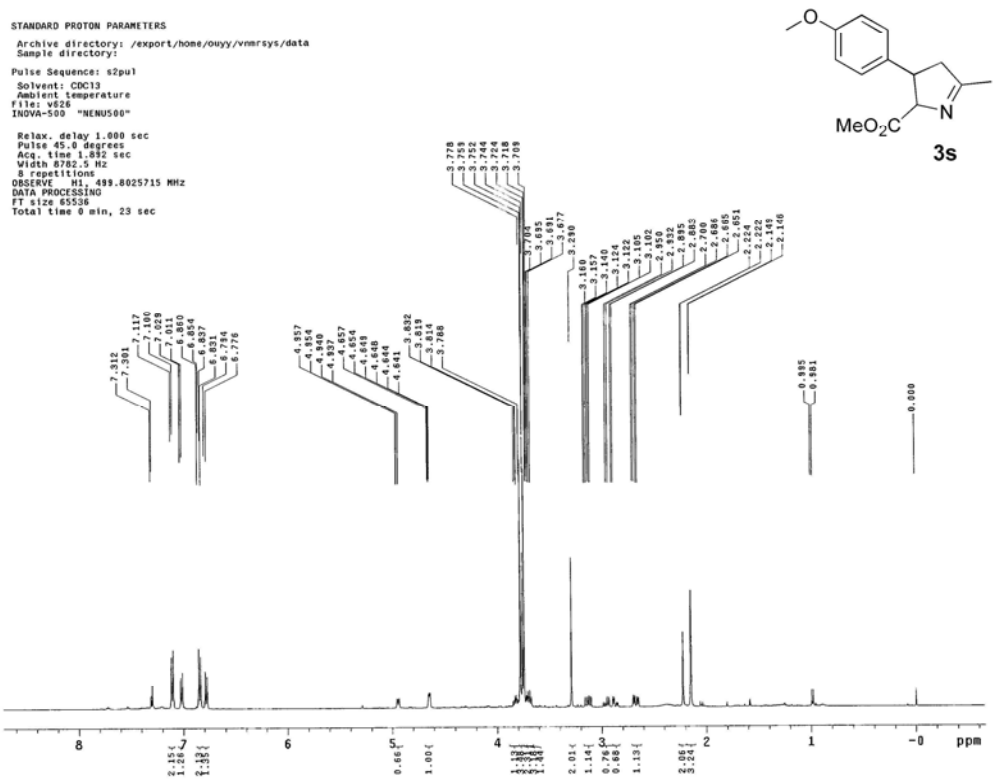

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z411
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5184 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
F
MeO2C
N
3p
ppm

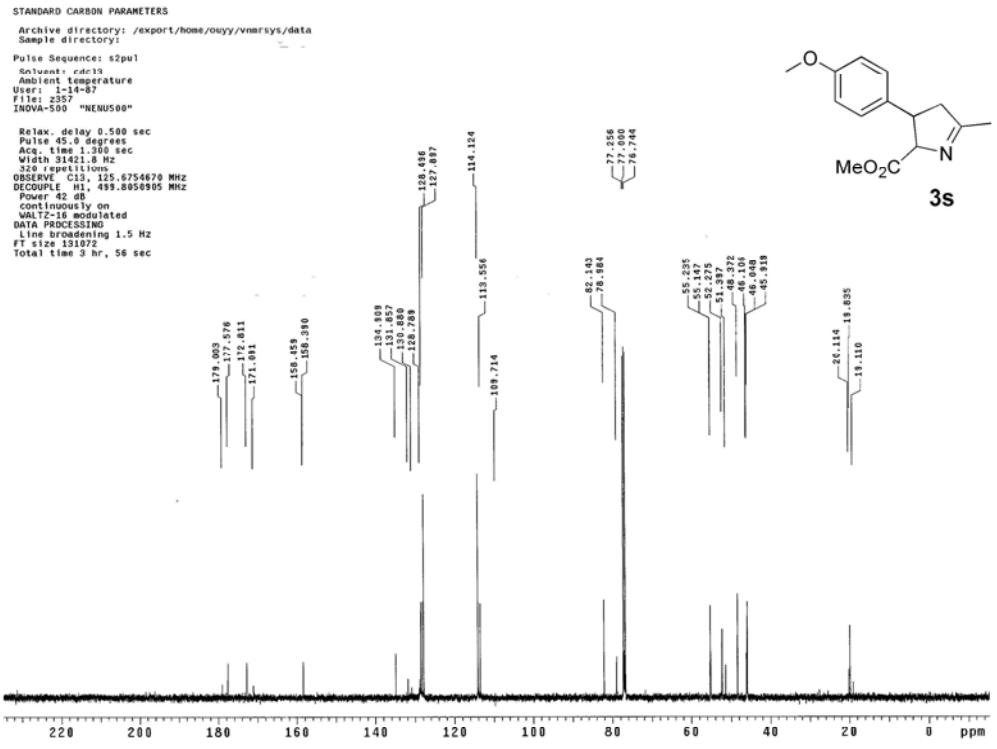

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v325
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8680.6 Hz
8 repetitions
OBSERVE H1, 499.8025730 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
3q
ppm

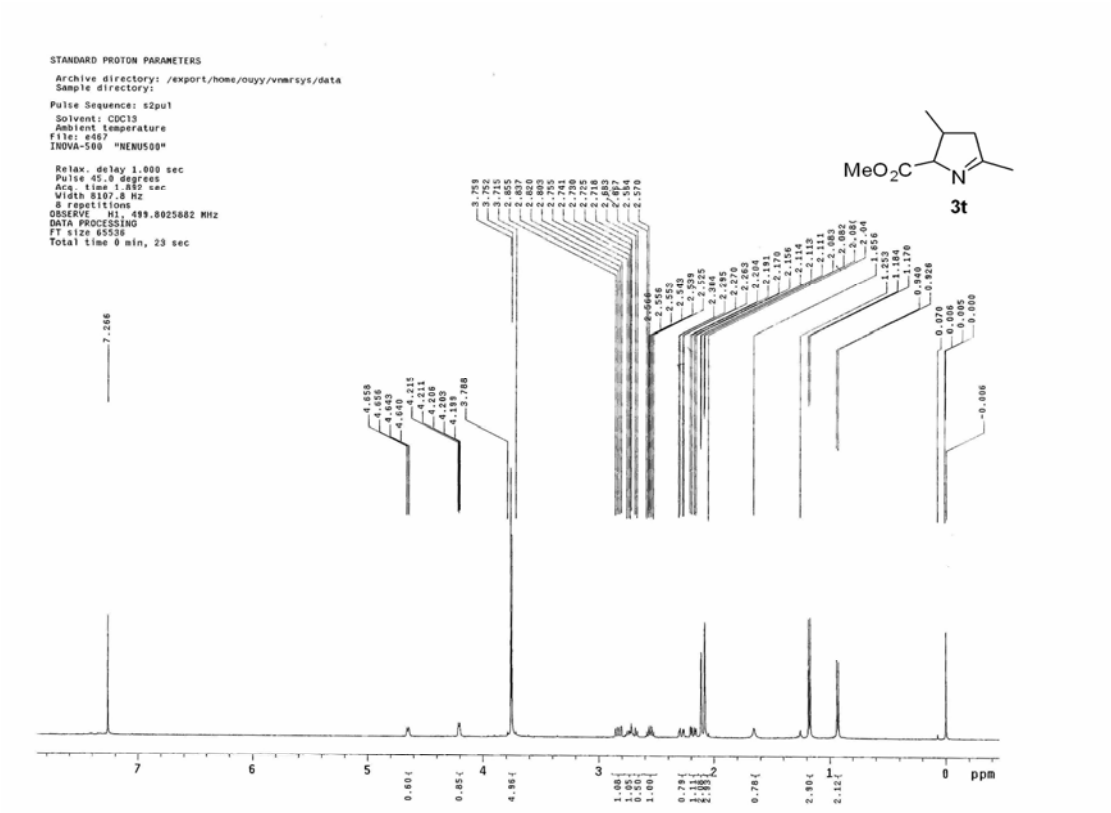

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z356
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
3q
ppm

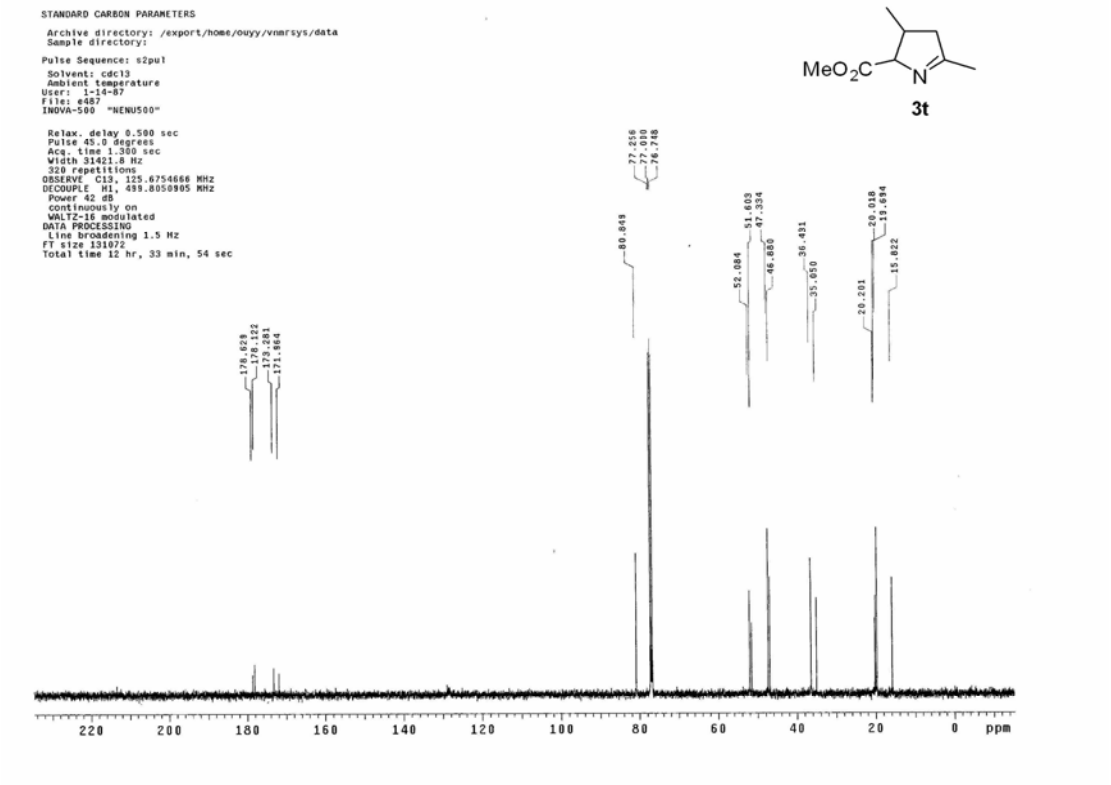

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v625
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8782.5 Hz
8 repetitions
OBSERVE H1, 499.8025650 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
3r
ppm

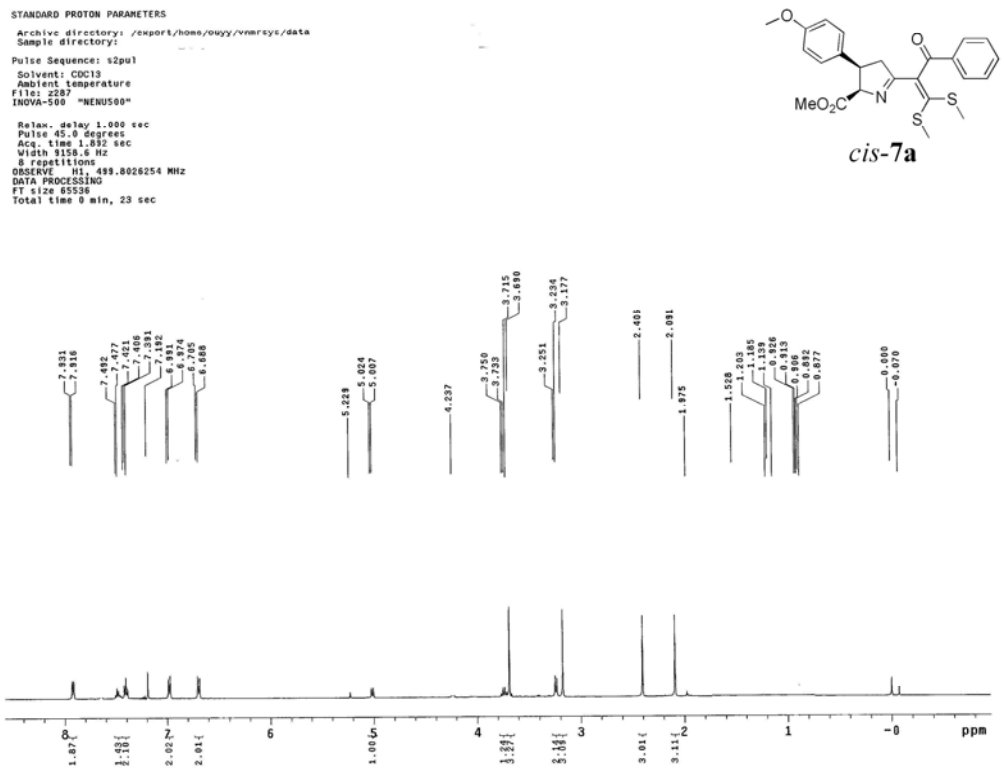

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z421
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
N
3r
ppm

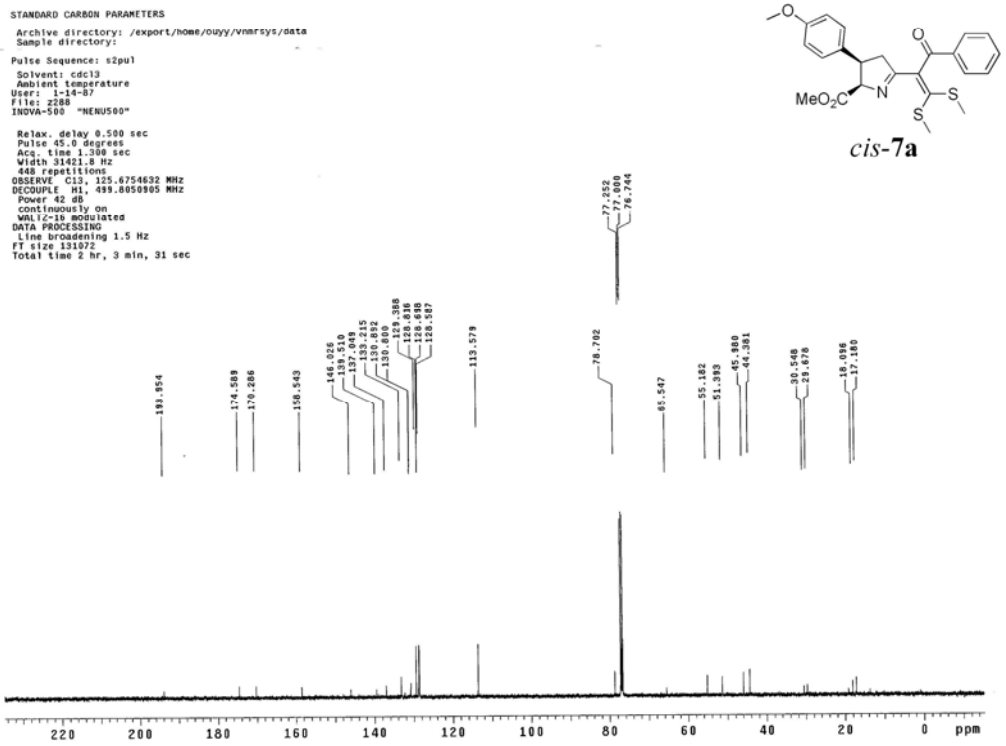

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: v626
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8782.5 Hz
8 repetitions
OBSERVE H1, 499.8025715 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
O
3s
ppm

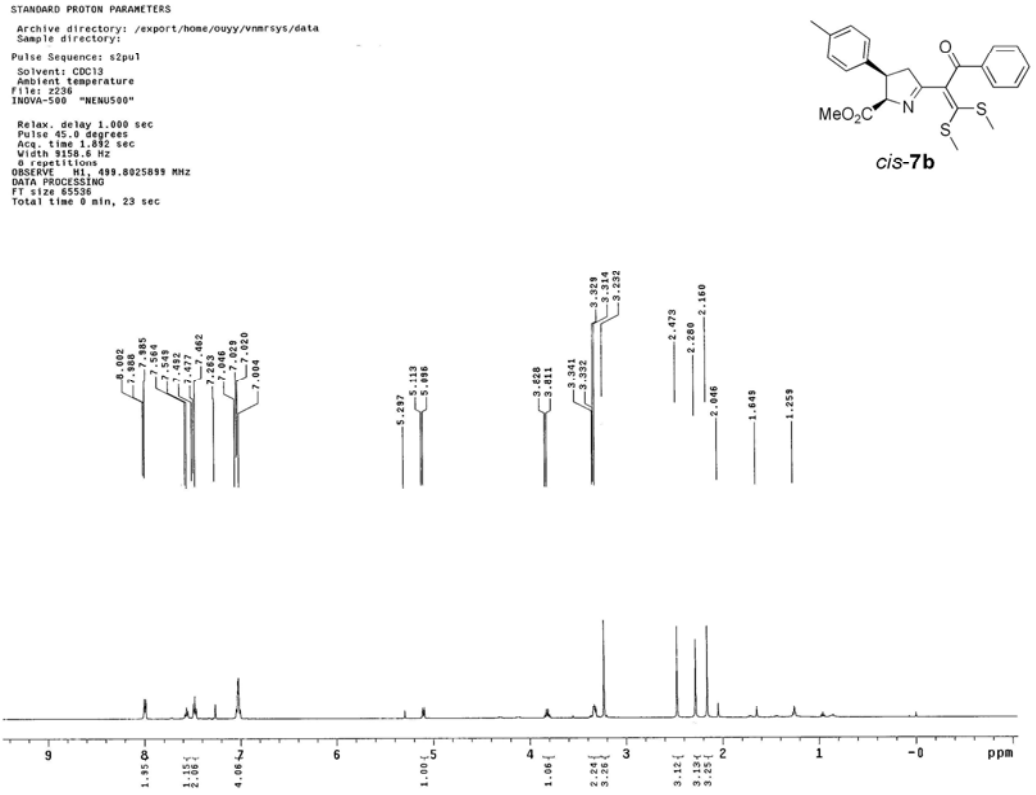

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Ambient temperature
User: 1-14-87
File: 2357
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8056905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
O
3s
ppm

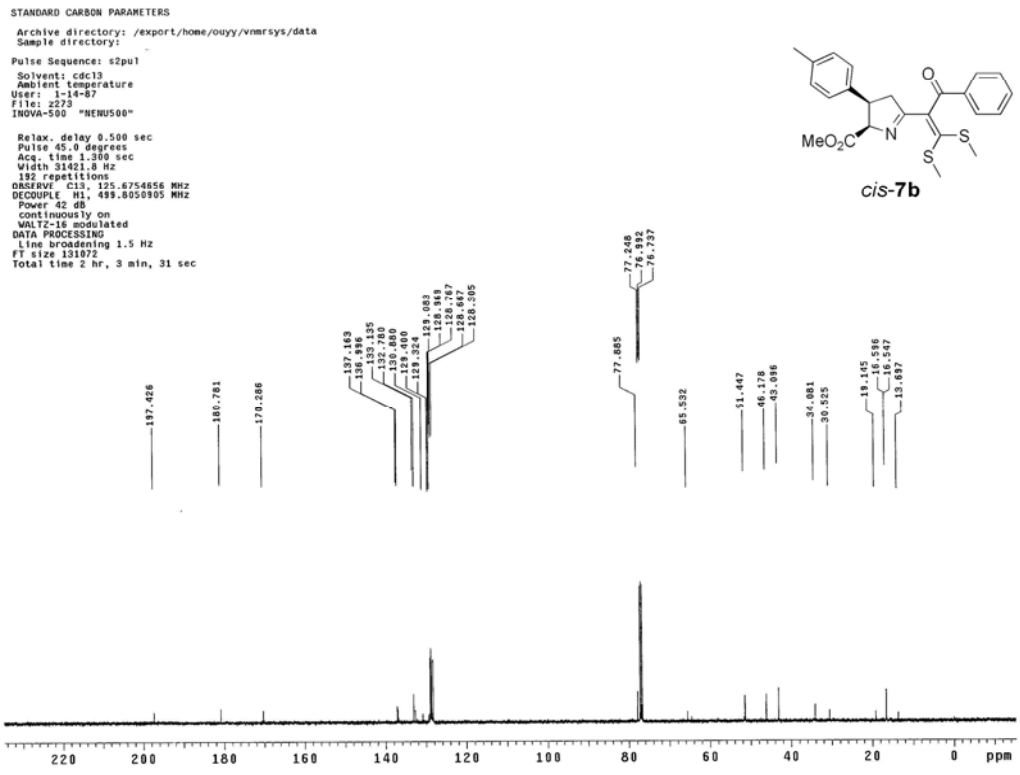

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: e467
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.802 sec
Width 8107.8 Hz
8 repetitions
OBSERVE H1, 499.8025682 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
3t
ppm

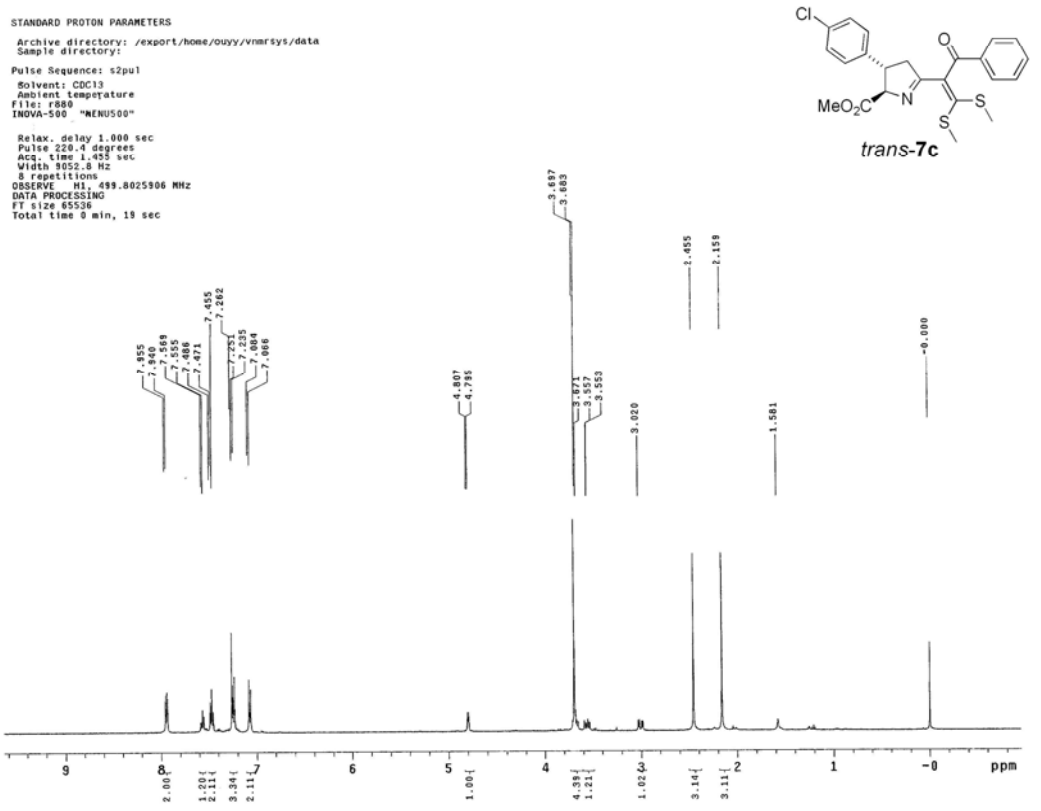

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: e487
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754666 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 12 hr, 33 min, 54 sec
MeO2C
N
3t
ppm

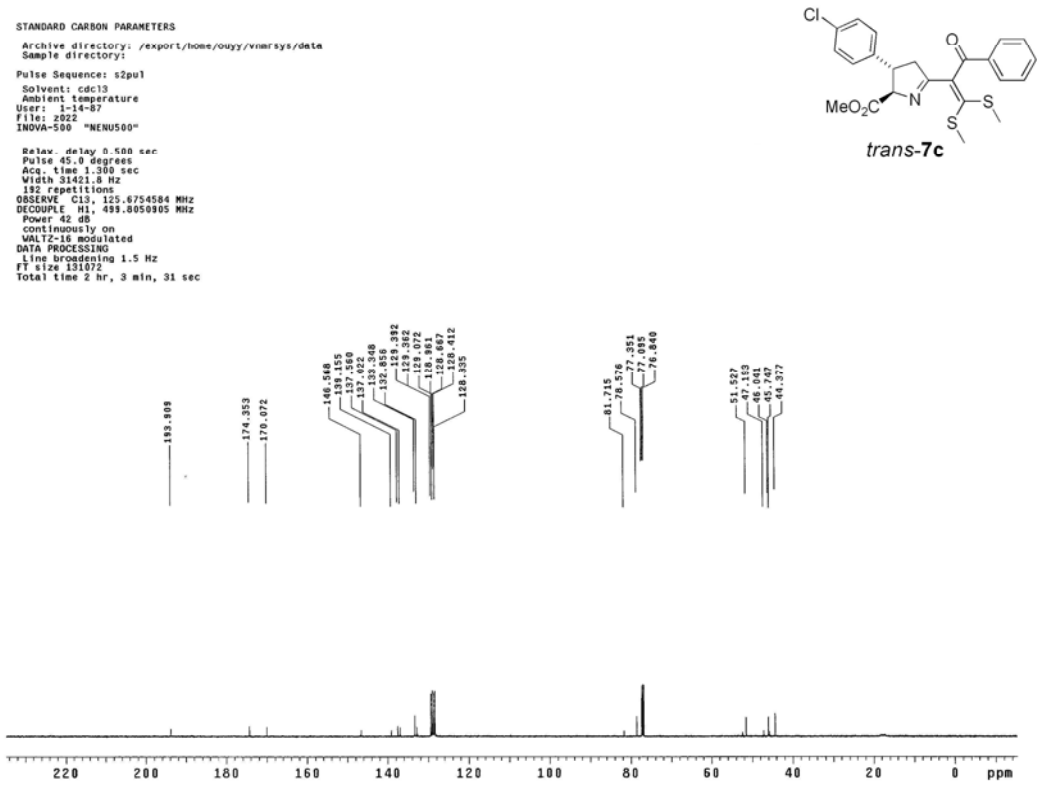

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z287
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9158.6 Hz
8 repetitions
OBSERVE H1, 499.8026254 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
cis-7a
ppm

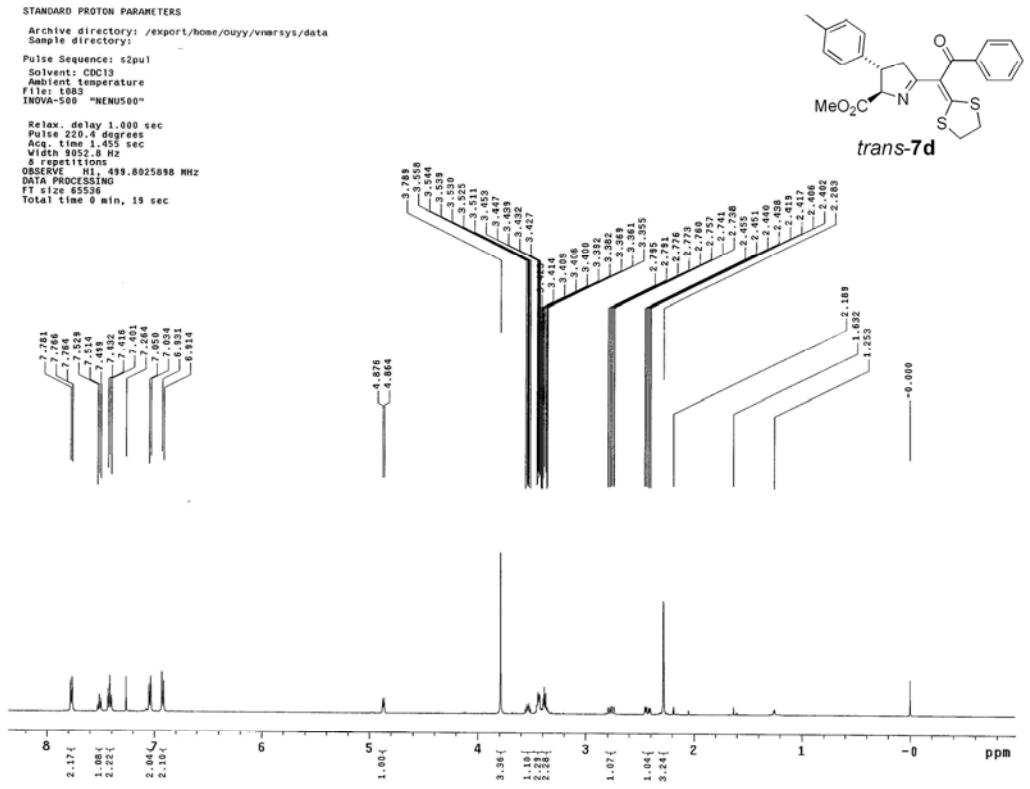

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z288
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
448 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
cis-7a
ppm

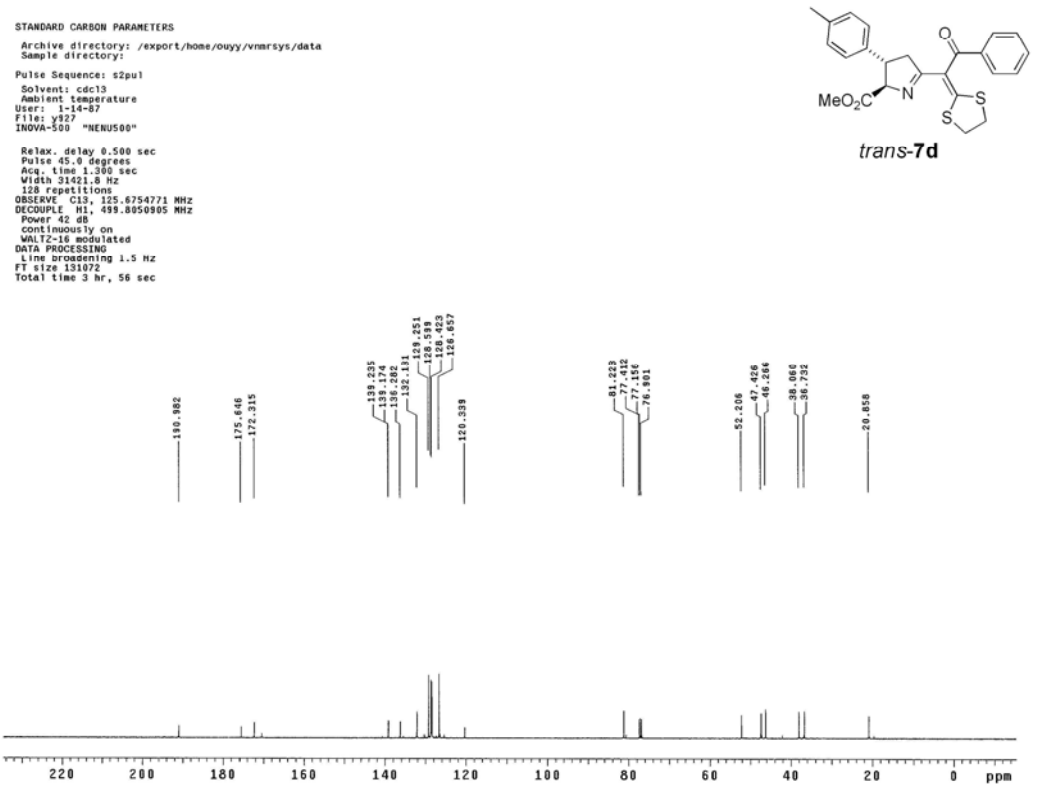

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z236
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9158.6 Hz
0 repetitions
OBSERVE H1, 499.8025899 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
cis-7b
ppm

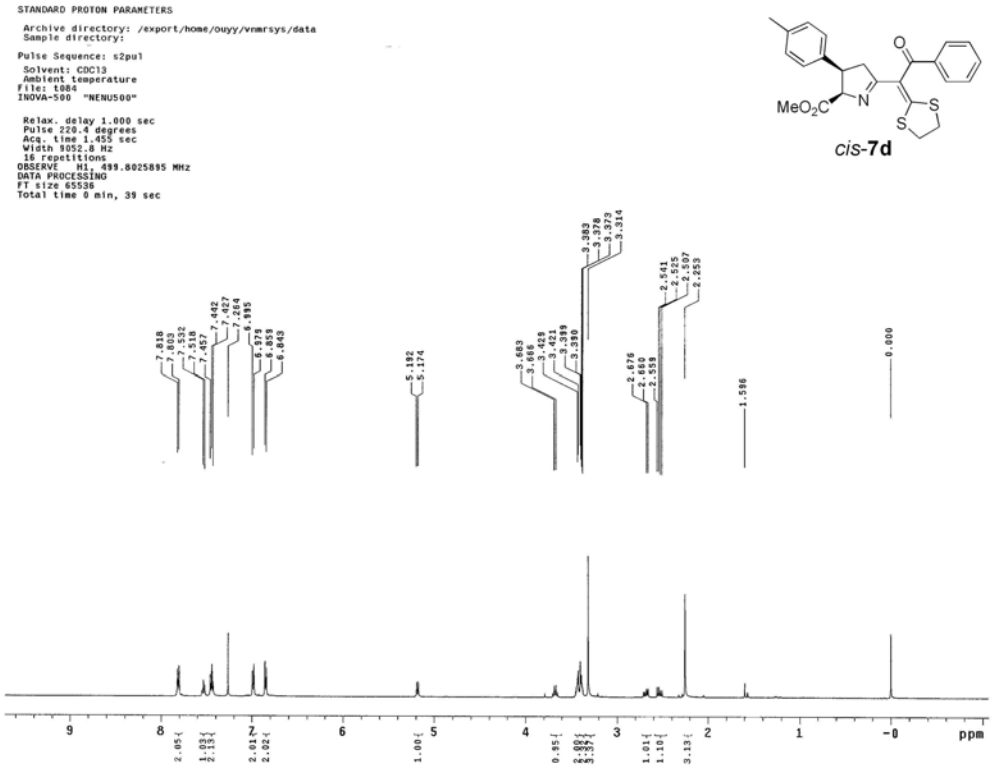

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z273
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
cis-7b
ppm

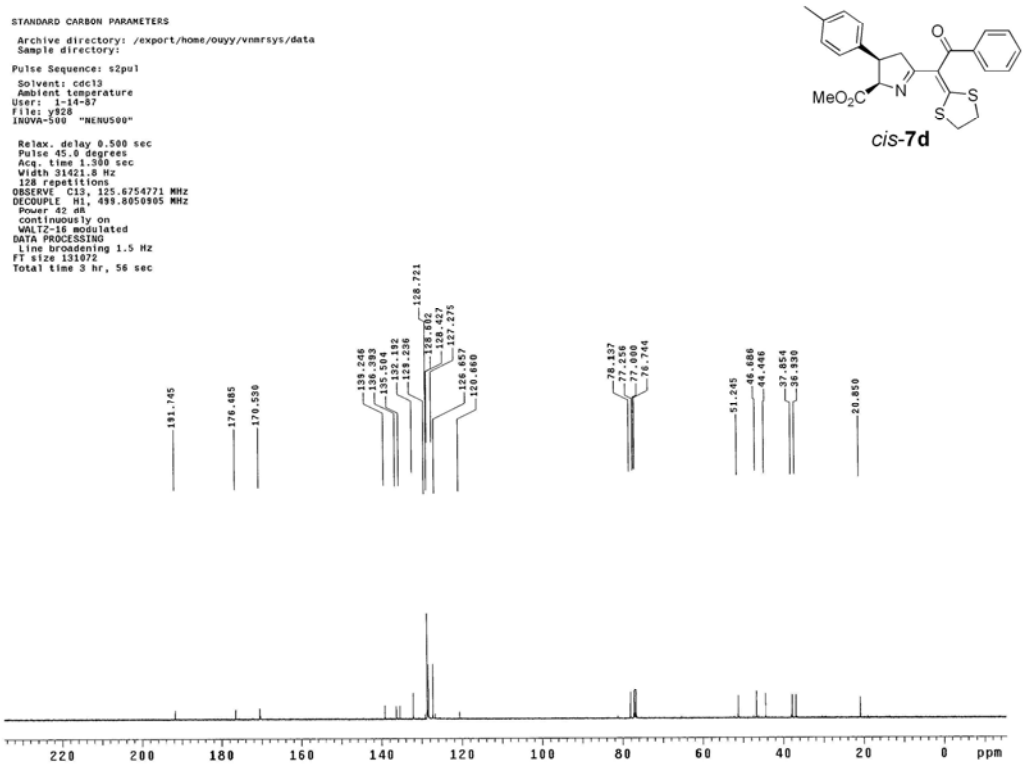

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: r880
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 220.4 degrees
Acq. time 1.455 sec
Width 9052.8 Hz
8 repetitions
OBSERVE H1, 499.8025906 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 19 sec
trans-7c
ppm

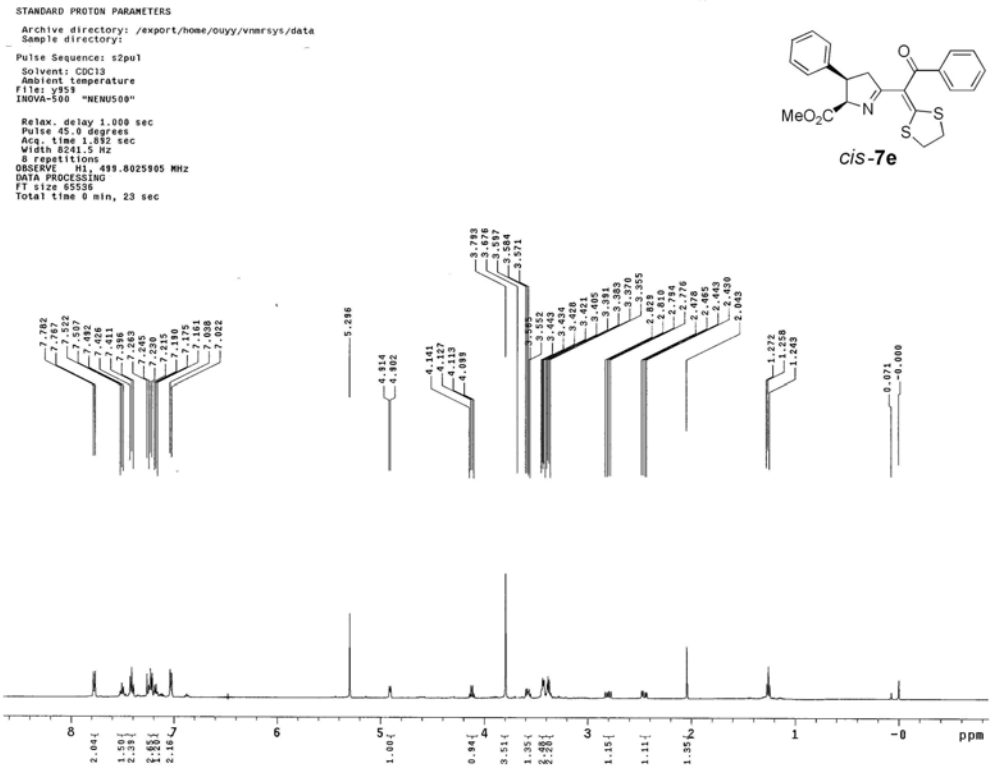

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z022
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754584 MHz
DECOUPLE H1, 499.8050305 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
trans-7c
ppm

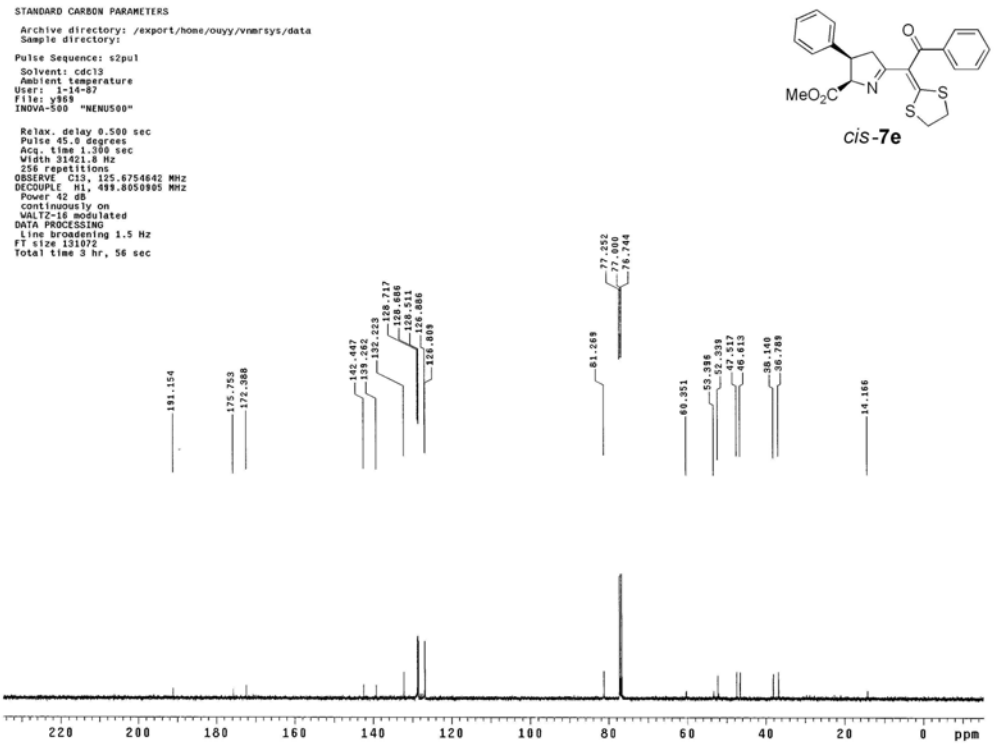

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t083
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 220.4 degrees
Acq. time 1.455 sec
Width 9052.8 Hz
8 repetitions
OBSERVE H1, 499.8025898 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 19 sec
trans-7d
ppm

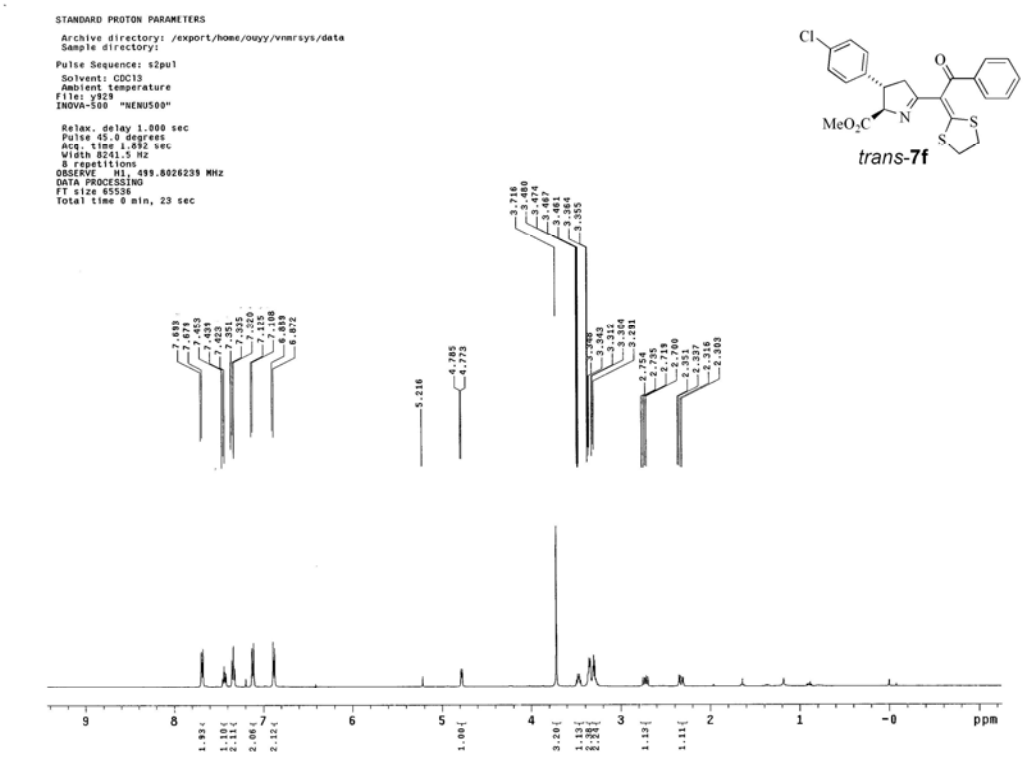

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y927
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754771 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
trans-7d
ppm

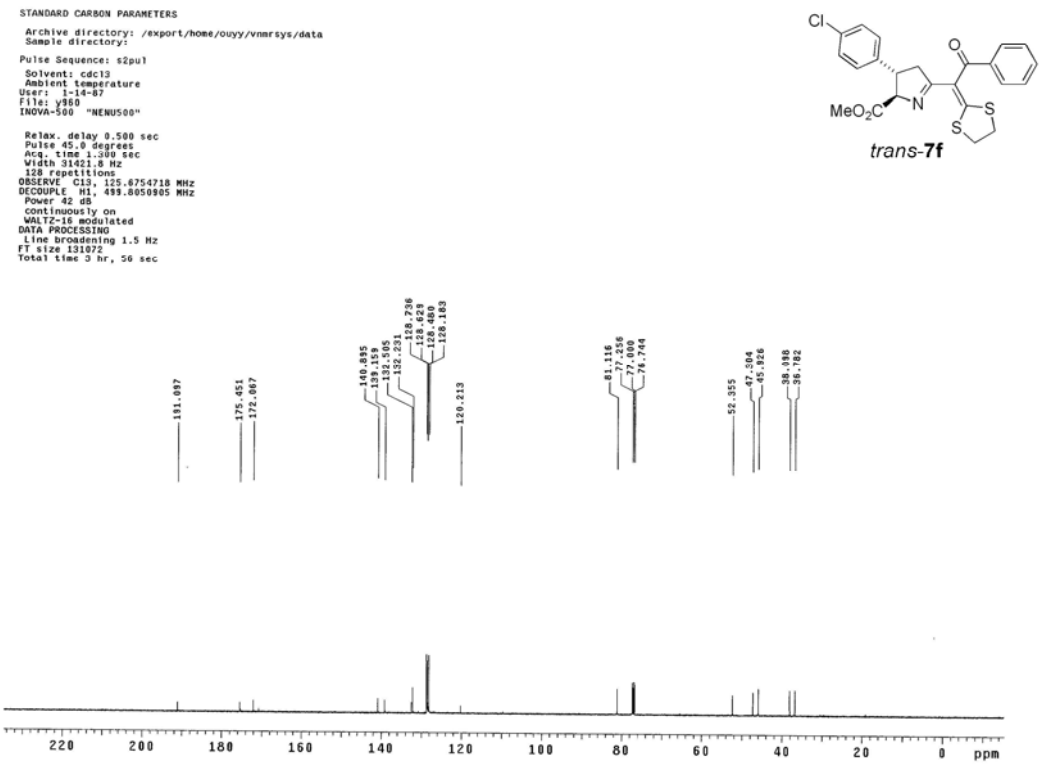

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t084
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 220.4 degrees
Acq. time 1.455 sec
Width 9052.8 Hz
16 repetitions
OBSERVE H1, 499.8025895 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 39 sec
MeO2C
N
O
S
S
cis-7d
ppm

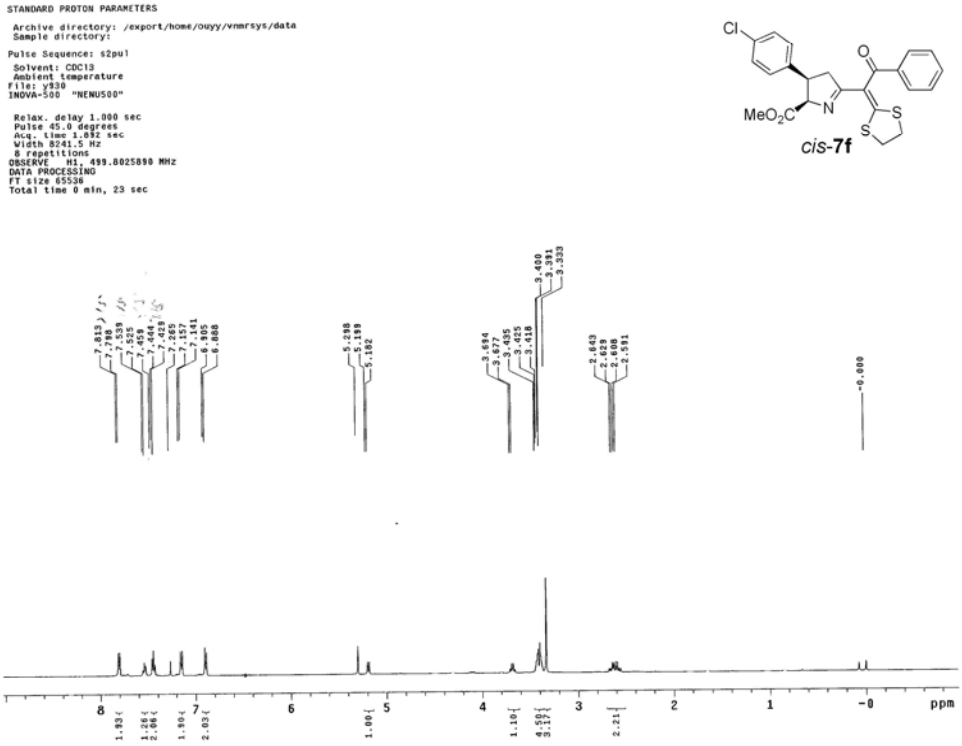

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y826
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754771 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
O
S
S
cis-7d
ppm

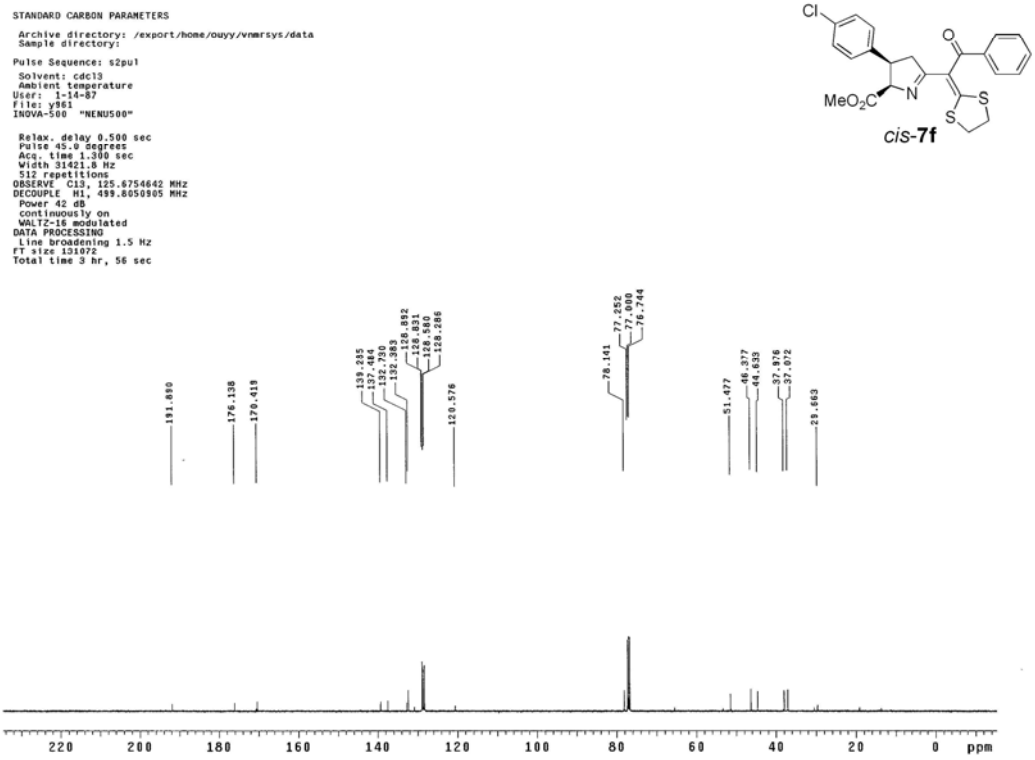

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y959
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8241.5 Hz
8 repetitions
OBSERVE H1, 499.8025905 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
N
O
S
S
cis-7e
ppm

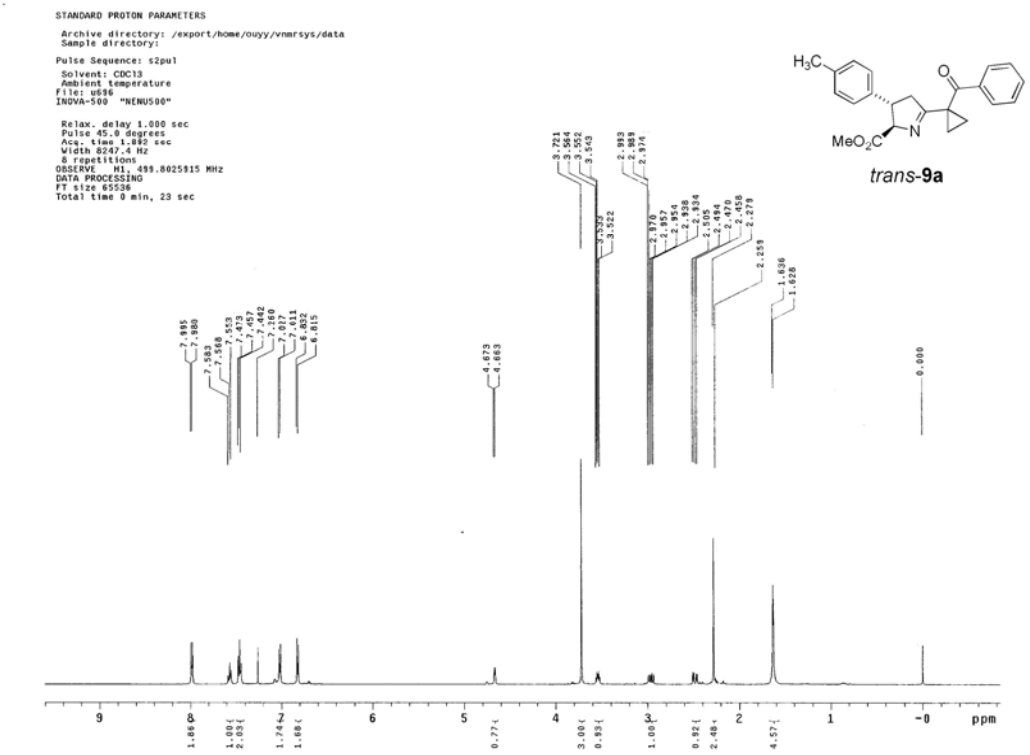

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y969
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
MeO2C
N
O
S
S
cis-7e
ppm

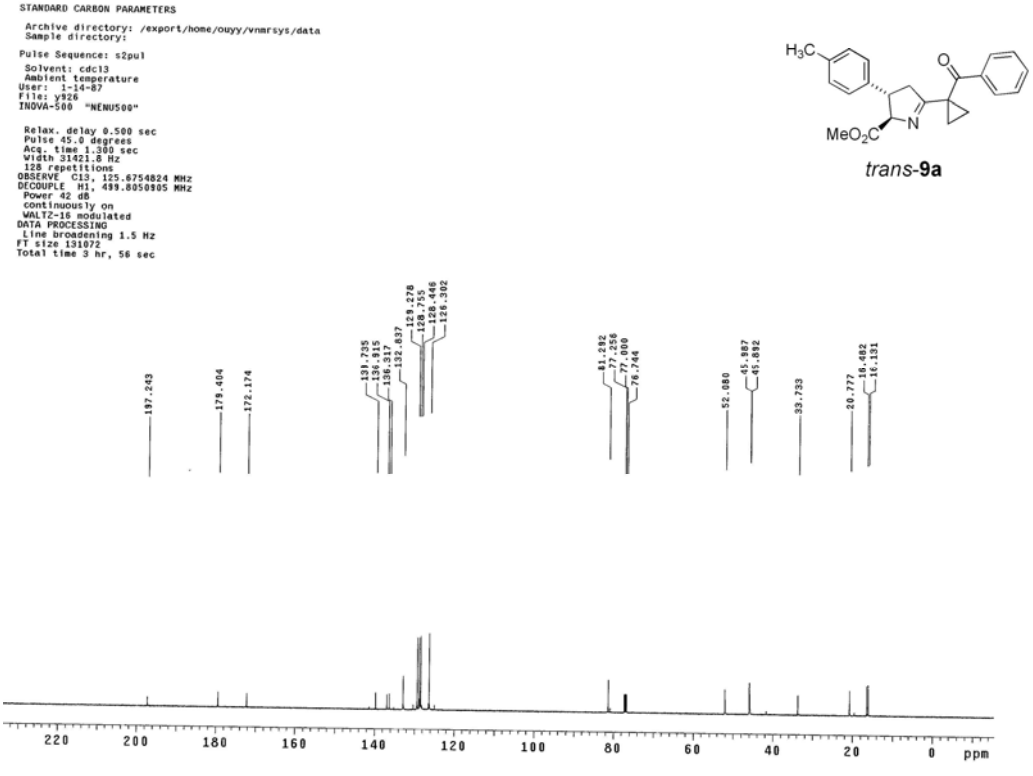

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y929
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.692 sec
Width 8241.5 Hz
8 repetitions
OBSERVE H1, 499.8026239 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
O
MeO2C
N
S
S
trans-7f
ppm

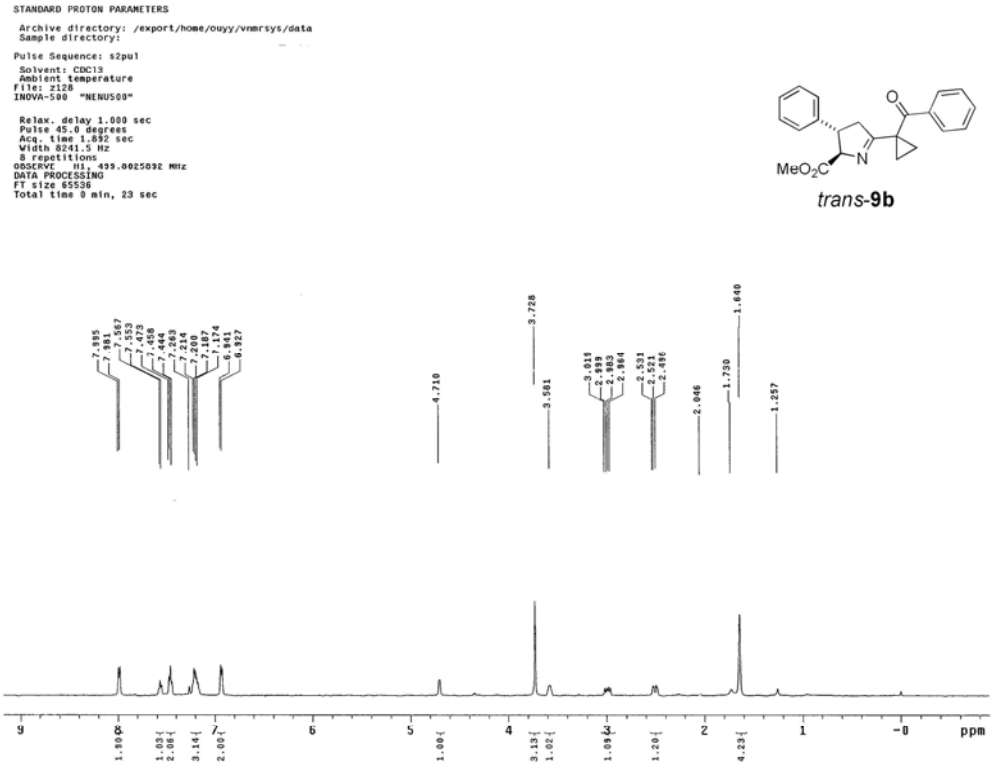

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y960
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754718 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
O
MeO2C
N
S
S
trans-7f
ppm

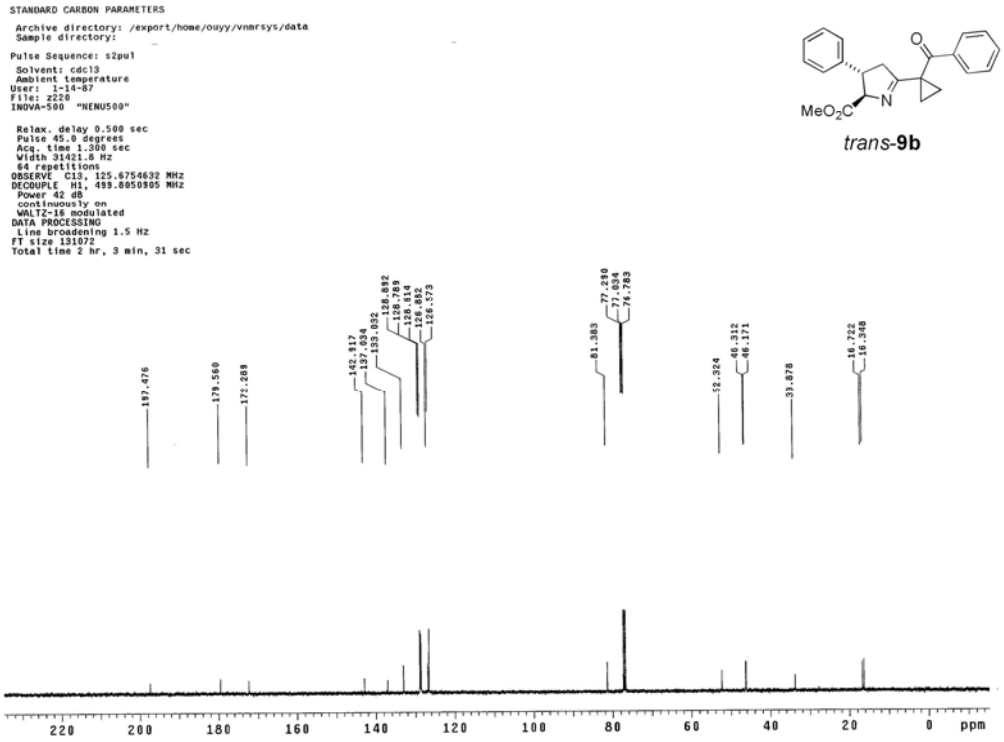

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y930
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.832 sec
Width 8241.5 Hz
8 repetitions
OBSERVE H1, 499.8025898 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
O
MeO2C
N
S
S
cis-7f
7.815
7.788
7.558
7.555
7.459
7.444
7.428
7.405
7.157
7.141
6.905
6.886
5.298
5.199
5.182
3.694
3.677
3.435
3.425
3.418
3.400
3.391
3.333
2.643
2.629
2.608
2.591
0.000
1.93
1.26
2.06
1.90
2.03
1.00
1.10
4.50
3.17
2.21
8
7
6
5
4
3
2
1
-0
ppm

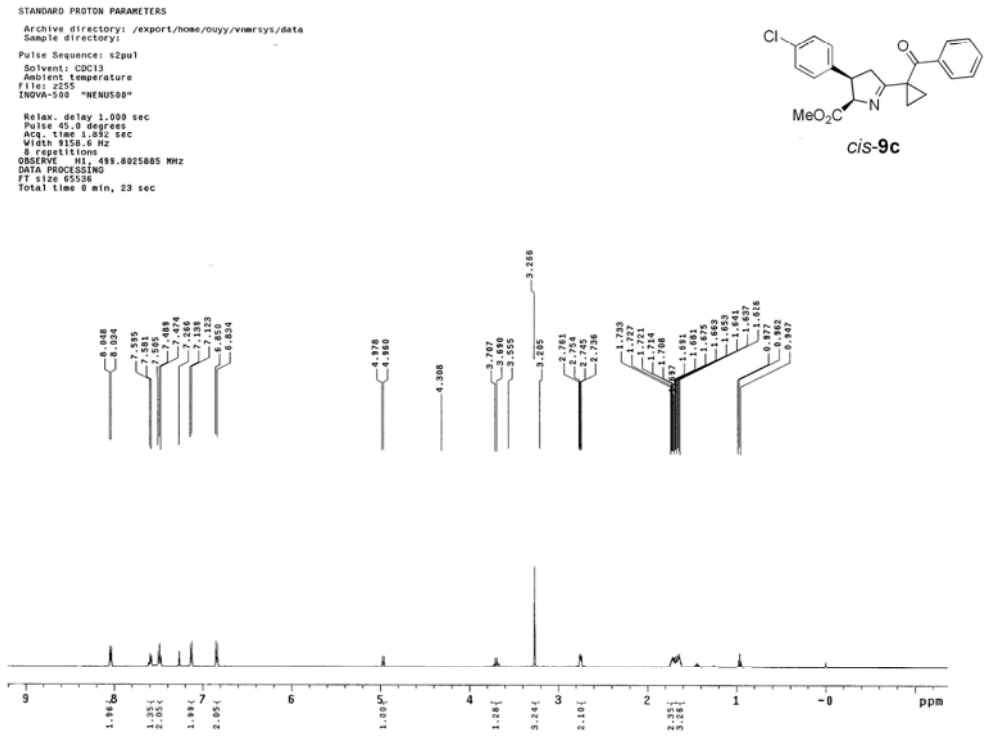

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y961
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Cl
O
MeO2C
N
S
S
cis-7f
191.690
176.136
170.418
139.035
137.484
132.730
132.383
130.892
130.431
128.580
128.286
120.576
78.141
77.252
77.000
76.744
51.477
46.377
44.633
37.976
37.072
29.663
220
200
180
160
140
120
100
80
60
40
20
0
ppm

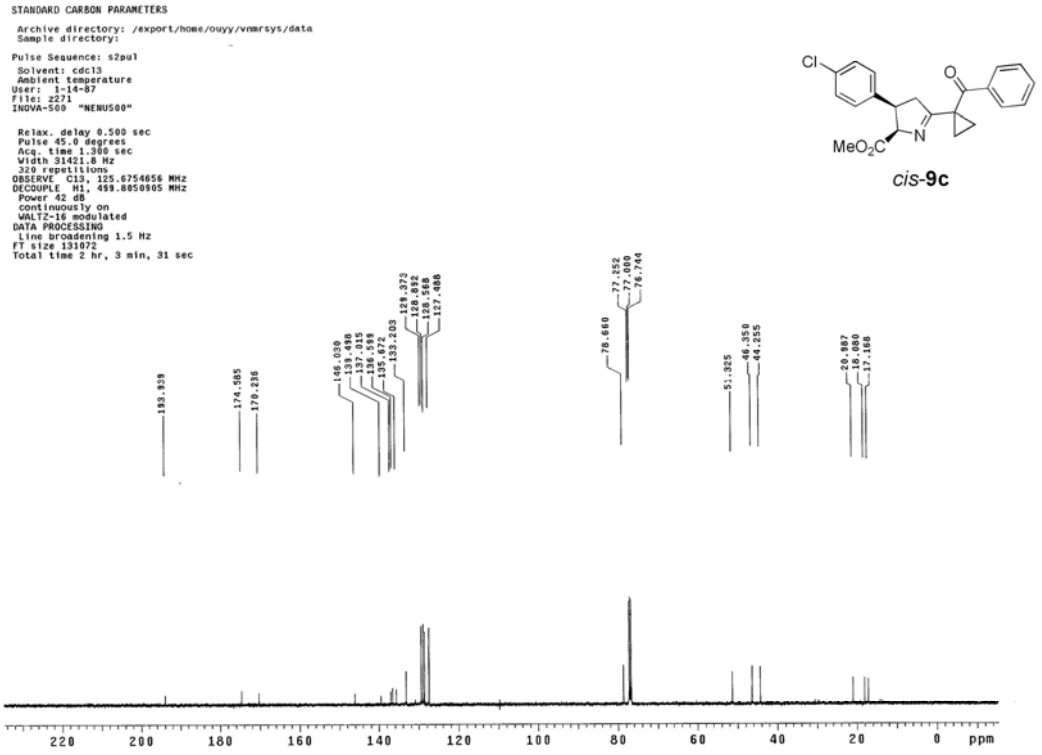

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u696
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8247.4 Hz
8 repetitions
OBSERVE H1, 499.8025815 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
trans-9a
ppm

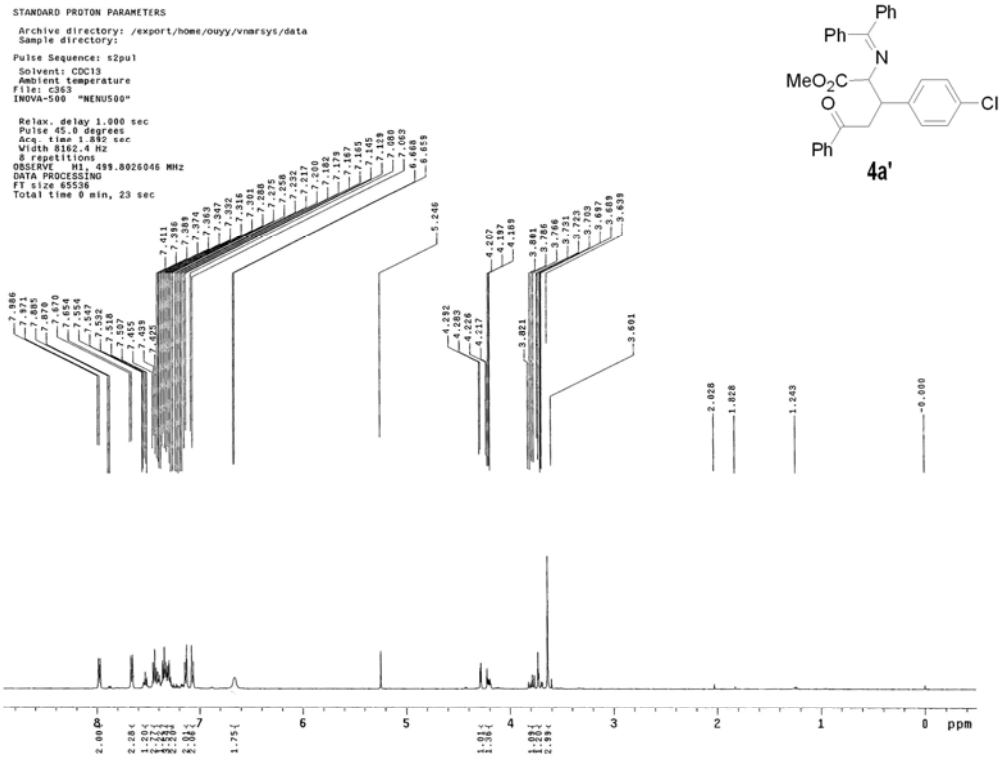

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: y926
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754824 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
trans-9a
ppm

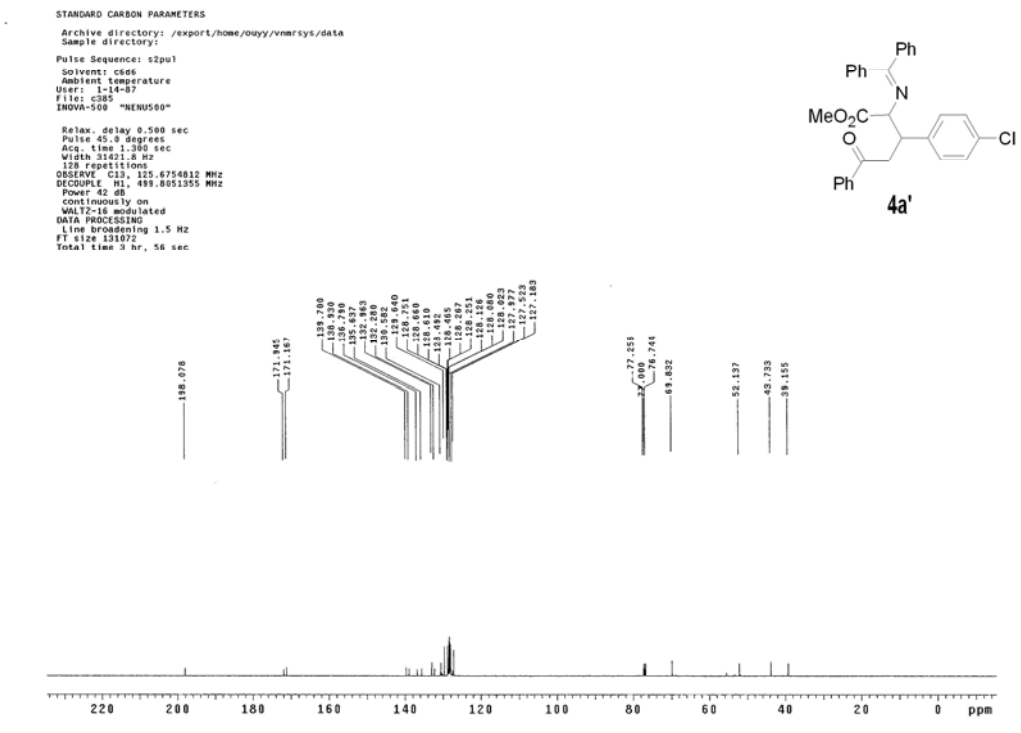

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z128
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8241.5 Hz
8 repetitions
OBSERVE H1, 499.8025692 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO2C
trans-9b
7.895
7.881
7.567
7.553
7.475
7.459
7.444
7.263
7.254
7.240
7.187
7.174
6.941
6.927
4.710
3.728
3.581
3.011
2.999
2.983
2.964
2.531
2.521
2.498
2.046
1.730
1.640
1.257
ppm

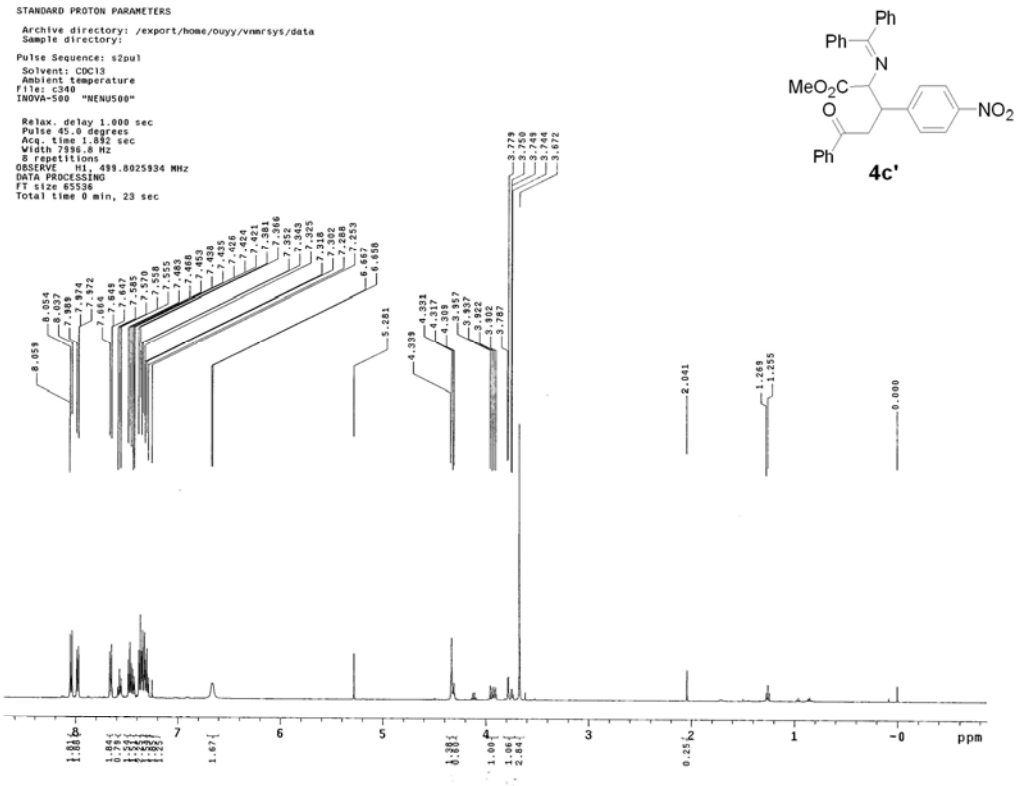

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z220
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050305 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
MeO2C
trans-9b
197.478
179.560
172.269
142.917
137.634
133.032
128.892
128.769
128.614
128.492
126.573
81.383
77.288
77.036
76.783
62.324
46.312
46.171
33.878
16.722
16.348
ppm

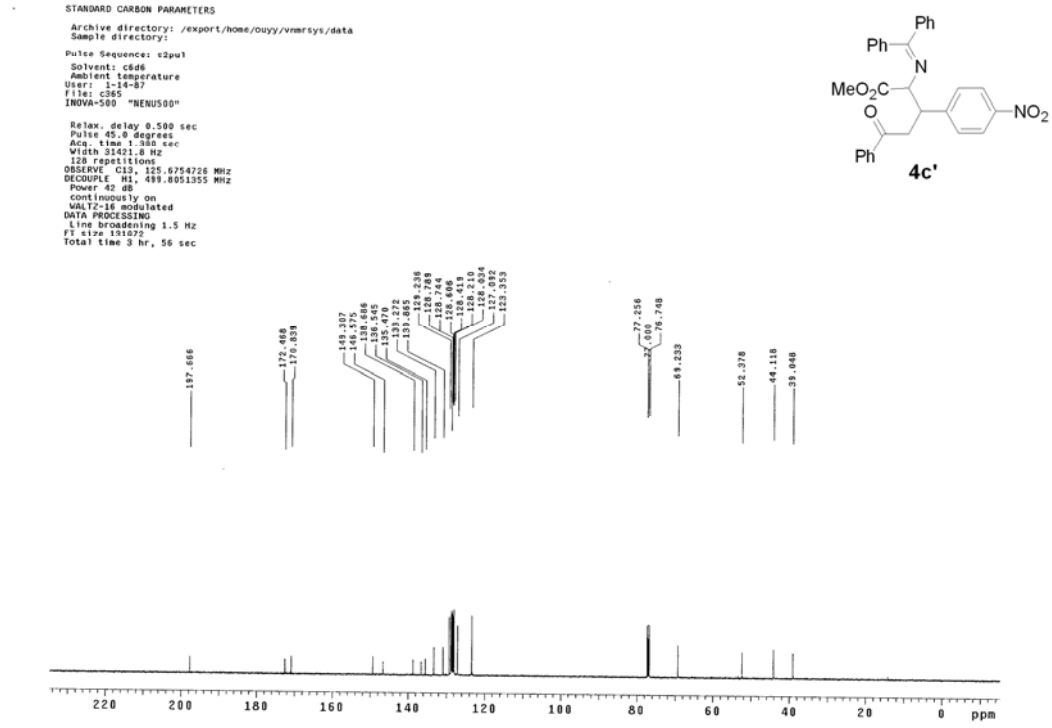

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z255
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9158.6 Hz
8 repetitions
OBSERVE H1, 499.8025885 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
O
N
MeO2C
cis-9c
8.048
8.034
7.595
7.581
7.505
7.489
7.474
7.266
7.139
7.123
6.836
6.834
4.978
4.960
4.308
3.707
3.690
3.555
3.266
3.265
2.761
2.754
2.745
2.738
1.733
1.727
1.721
1.714
1.708
1.697
1.691
1.681
1.675
1.663
1.653
1.641
1.637
1.616
0.977
0.968
0.947
9
8
7
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: z271
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
Cl
O
N
MeO2C
cis-9c
193.939
174.585
170.296
146.039
139.498
137.015
136.579
135.672
133.203
129.373
128.892
128.586
127.488
77.252
77.000
76.744
70.660
51.325
46.350
44.255
20.987
18.080
17.168
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: c363
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8162.4 Hz
8 repetitions
OBSERVE H1, 499.8026046 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Ph
Ph
N
MeO2C
O
Cl
Ph
4a'
ppm

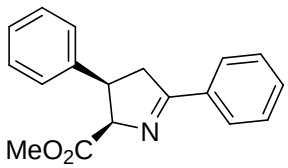

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
User: 1-14-07
File: c385
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754812 MHz
DECOUPLE H1, 499.8051355 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Ph
Ph
N
MeO2C
O
Cl
Ph
4a'
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: c340
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7896.8 Hz
8 repetitions
OBSERVE H1, 499.8025934 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Ph
Ph
N
MeO2C
O
NO2
Ph
4c'
ppm

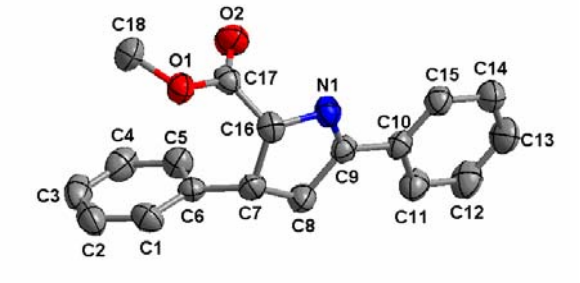

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: c6d6
Ambient temperature
User: 1-14-87
File: c365
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754726 MHz
DECOUPLE H1, 499.8051355 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
Ph
Ph
N
MeO2C
O
NO2
Ph
4c'
ppm

V. Crystal data and ORTEP drawing of compound *cis*-**3d**

1. ORTEP drawing of compound **3d**



cis-**3d**



2. Crystal data

cis-**3d**: $C_{18}H_{17}N_1O_2$, pale-yellow, Mr = 279.33, monoclinic, space group P2(1)/c, a = 7.6540(13), b = 16.830(3), c = 11.568(2) Å, α=90.000, β= 99.578(2), γ= 90.000º, V = 1469.4(4) $Å_3$, Z = 4, T = 293(2) K, 7868 reflections (2863 unique), 191 refined parameters, R1 = 0.0448 (I > 2σ(I)), wR2(F2) = 0.1091. The hydrogen atoms were refined as rigid groups. CCDC deposition number: 844259 (**3d**). These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).