Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Asymmetric Transformation by Dynamic Crystallization of Achiral Succinimides

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Table of Contents

General experimental:

General. NMR spectra were recorded on $CDCl_3$ solutions on a BRUKER 300 operating 300 MHz, respectively, for ^{1}H- and ^{13}C-NMR spectroscopy. Chemical shifts are reported in parts per million (ppm) relatives to TMS as internal standards. IR spectra were recorded on a JASCO FT/IR-230 spectrometer as KBr disks. Specific rotation was measured by a DIP 370 polarimeter (JASCO).

Preparation of 3,4-diphenylmaleimides 1a-k

3,4-Diphenylmaleimides 1a-k were synthesized from 3,4-diphenylmaleic anhydride and the corresponding amines according to the literature[S1].

Preparation of *cis*-3,4-diphenylsuccinimides 2d and2e

An ethyl acetate solution of 3,4-diphenylmaleimide was stirred under hydrogen atmosphere in the presence of catalytic amount of PtO_2 until the yellowish green color disappeared. PtO_2 was filtered off through celite column, and the filtrate was evaporated. Chromatography (eluent: a mixture of ethyl acetate and hexane) or by crystallization from with a minimum of $CHCl_3$ and an excess of hexane gave the desired cis-3,4-diphenylmaleimide product almost quantitative yield.

***cis*-*N*-propyl-3,4-diphenylsuccinimde 2d** was obtained as colorless crystals: m.p. 81-83 °C; IR (KBr) 1775, 1697 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.03 (t, *J* = 7.4 Hz, 3H), 1.73-1.86 (m, 2H), 3.71 (t, *J* = 7.4 Hz, 2H), 4.46 (s, 2H), 6.78-7.00 (m, 4H), 7.02-7.08 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 11.4, 21.3, 40.8, 52.2, 127.1, 128.1, 129.1, 133.9, 177.1; HRMS (ESI-MS) *m*/*z* calcd for $C_{19}H_{19}O_2N$ + Na 316.1308, found 316.1302

***cis*-*N*-isopropyl-3,4-diphenylsuccinimide 2e** was obtained as colorless crystals: 110-114 °C; IR (KBr) 1772, 1699 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.56 (d, *J* = 6.9 Hz, 6H), 4.41 (s, 2H), 4.64 (sep, *J* = 7.0 Hz, 1H), 6.78-6.81 (m, 4H), 7.02-7.25 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.4, 44.3, 52.1, 127.1, 128.1, 129.1, 134.1, 177.0; HRMS (ESI-MS) *m*/*z* calcd for $C_{19}H_{19}O_2N$ + Na 316.1308, found 316.1304

Preparation of *trans*-3,4-diphenysuccinimides 3a-k

Trans-3,4-diphenylsuccinimides **3** were synthesized from the corresponding *cis* isomers **2**. Crude *cis* isomers **2** generated by hydrogenation of the corresponding 3,4-diphenylmaleimides **1** with hydrogen in the presence of PtO_2 were directly isomerized to *trans* isomers **3** by catalytic amount of DBU. Catalytic amount of DBU (0.10 eq.) was added to a chloroform solution of *cis* isomer and the mixture was stirred for overnight at room temperature. The reaction mixture

was concentrated *in vacuo*, and the residue was chromatographed on silica gel (eluent: a mixture of ethyl acetate and hexane). Crystalline *trans* isomers **3** were purified by crystallization with a minimum of $CHCl_3$ and an excess of hexane. In all cases, *trans* isomers **3** were obtained in almost quantitative yields through hydrogenation and isomerization.

The structure of **3a, 3e**, **3g**, **3h**, and **3j** was unequivocally established by X-ray single crystallographic analysis. Other *trans* isomers did not give available crystals for single crystal X-ray analysis.

***trans*-3,4-diphenylsuccinimde 3a** was obtained as colorless crystals: m.p. 197-199 °C; IR (KBr) 3318, 1795, 1701 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.14 (s, 2H), 7.20-7.23 (m, 4H), 7.31-7.42(m, 6H), 8.62(s, 1H); ^{13}C NMR ($CDCl_3$) δ 56.6, 127.7, 128.2, 129.3, 135.8, 176.5; HRMS (ESI-MS) *m*/*z* calcd for $C_{16}H_{13}O_2N$ + Na 274.0838, found 274.0840.

***trans*-*N*-methyl-3,4-diphenylsuccinimde 3b** was obtained as colorless crystals: m.p. 107-108 °C; IR (KBr) 1774, 1689 cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.13 (s, 3H), 4.05 (s, 2H), 7.15-7.17 (m, 4H), 7.26-7.36 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 25.4, 55.3, 127.6, 127.9, 129.0, 136.2, 176.5; HRMS (ESI-MS) *m*/*z* calcd for $C_{17}H_{15}O_2N$ + Na 288.0995, found 288.0993

***trans*-*N*-ethyl-3,4-diphenylsuccinimde 3c** was obtained as colorless crystals: m.p. 86-87 °C; IR (KBr) 1776, 1693 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, 3H), 3.74 (q, *J* = 7.2 Hz, 2H), 4.03 (s, 2H), 7.16-7.19 (m, 4H), 7.30-7.41 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 13.2, 34.4, 55.5, 127.6, 128.0, 129.3, 136.7, 176.5; HRMS (ESI-MS) *m*/*z* calcd for $C_{18}H_{17}O_2N$ + Na 302.1152, found 302.1149

***trans*-*N*-propyl-3,4-diphenylsuccinimde 3d** was obtained as colorless crystals: m.p. 67-69 °C; IR (KBr) 1774, 1697 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.98 (t, *J* = 7.4 Hz, 3H), 1.67-1.79 (m, 2H), 3.65 (t, *J* = 7.3 Hz, 2H), 4.04 (s, 2H) 7.16-7.19 (m, 4H), 7.32-7.41 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 11.3, 21.1, 40.9, 55.4, 127.6, 128.0, 129.2, 136.6, 176.7; HRMS (ESI-MS) *m*/*z* calcd for $C_{19}H_{19}O_2N$ + Na 316.1308, found 316.1306

***trans*-*N*-isopropyl-3,4-diphenylsuccinimde 3e** was obtained as colorless crystals: m.p. 152-153 °C; IR (KBr) 1773, 1696 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.48 (d, *J* = 6.9 Hz, 3H), 1.50 (d, *J* = 6.9 Hz, 3H), 3.97 (s, 2H), 4.54 (sep, *J* = 6.9 Hz, 1H), 7.15-7.18 (m, 4H), 7.28-7.39 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 19.2, 19.4, 44.4, 55.2, 127.5, 127.9, 129.2, 136.9, 176.6; HRMS (ESI-MS) *m*/*z* calcd for $C_{19}H_{19}O_2N$ + Na 316.1308, found 316.1304

***trans*-*N*-benzyl-3,4-diphenylsuccinimde 3f** was obtained as colorless crystals: m.p. 86-88 °C; IR (KBr) 1783, 1708 cm^{-1}; ^1H NMR ($CDCl_3$) δ 4.05 (s, 2H), 4.77 and 4.86 (ABq, *J* = 14.0 Hz, 2H), 7.10-7.13 (m, 4H), 7.24-7.38 (m, 9H), 7.43-7.46 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 43.0, 55.4, 127.6, 128.0, 128.1, 128.7, 129.2, 135.7, 136.4, 176.3; HRMS (ESI-MS) *m*/*z* calcd for $C_{23}H_{19}O_2N$ + Na 364.1308, found 364.1303

***trans*-*N*-phenethyl-3,4-diphenylsuccinimde 3g** was obtained as colorless crystals: m.p. 121-123 °C; IR (KBr) 1773, 1707 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.99-3.15 (m, 2H), 3.88-4.08 (m, 4H), 6.97-7.00(m, 4H), 7.25-7.36 (m, 11H) ; ^{13}C NMR ($CDCl_3$) δ 33.1, 40.1, 55.5, 126.8, 127.6, 127.9, 128.6, 129.09, 129.15, 136.5, 127.4, 176.5; HRMS (ESI-MS) *m*/*z* calcd for $C_{24}H_{21}O_2N$ + Na 378.1465, found 378.1460

***trans*-*N*-phenyl-3,4-diphenylsuccinimde 3h** was obtained as colorless crystals: m.p. 236-238 °C; IR (KBr) 1775, 1709 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 4.24 (s, 2H), 7.25-7.53 (m, 15H); ^{13}C NMR ($CDCl_3$) δ 55.4, 126.5, 127.7, 128.2, 128.8, 129.2, 129.3, 131.9, 136.5, 175.7; HRMS (ESI-MS) *m*/*z* calcd for $C_{22}H_{17}O_2N$ + H 328.1332, found 328.1332

***trans*-*N*-(2-methylphenyl)-3,4-diphenylsuccinimde 3i** was obtained as colorless crystals: m.p. 176-177 °C; IR (KBr) 1779, 1710 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.25 (s, 3H), 4.24 (d, J = 5.4 Hz, 2H), 4.28 (d, J = 5.4 Hz, 2H), 7.17-7.19 (m, 1H), 7.25-7.44 (m, 13H); ^{13}C NMR ($CDCl_3$) δ 18.0, 55.7, 55.9, 127.1, 127.6, 127.7, 128.0, 128.2, 129.4, 129.7, 131.1, 131.3, 135.6, 136.6, 175.5, 175.7; HRMS (ESI-MS) *m*/*z* calcd for $C_{23}H_{19}O_2N$ + Na 364.1308, found 364.1304

***trans*-*N*-(3-methylphenyl)-3,4-diphenylsuccinimde 3j** was obtained as colorless crystals: m.p. 175-178 °C; IR (KBr) 1777, 1706 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.41 (s, 3H), 4.22 (s, 2H), 7.17-7.44 (m, 14H); ^{13}C NMR ($CDCl_3$) δ 21.3, 55.4, 123.6, 127.1, 127.6, 128.1, 129.0, 129.3, 129.7, 131.7, 126.6, 139.3, 175.8; HRMS (ESI-MS) *m*/*z* calcd for $C_{23}H_{19}O_2N$ + H 342.1489, found 342.1487

***trans*-*N*-(4-methylphenyl)-3,4-diphenylsuccinimde 3k** was obtained as colorless crystals: m.p. 170-171 °C; IR (KBr) 1776, 1711 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.39 (s, 3H), 4.22 (s, 2H), 7.25-7.43 (m, 14H); ^{13}C NMR ($CDCl_3$) δ 21.2, 55.4, 126.2, 127.6, 128.1, 129.2, 129.3, 129.8, 136.6, 138.9, 175.8; HRMS (ESI-MS) *m*/*z* calcd for $C_{23}H_{19}O_2N$ + H 342.1489, found 342.1487

X-Ray single crystal analysis of *trans*-3,4-diphenylsuccinimide 3a

Crystal data of **3a** (recrystallized from a mixture of $CHCl_3$ and hexane); $C_{16}H_{13}NO_2$, *Mr* = 251.27, Orthorhombic space group *P*bcn, a = 13.1134(11) Å, b = 13.6354(11) Å, c = 7.2439(6) Å, V = 1295.26(19) $Å^3$, Z = 4, ρ = 1.289 g cm^{-3}, in the final least-square refinement cycles on F^2, the model converged to R_1 = 0.0376, wR_2 = 0.0911, and GOF = 1.035 for 1154 reflections. CCDC 927187.

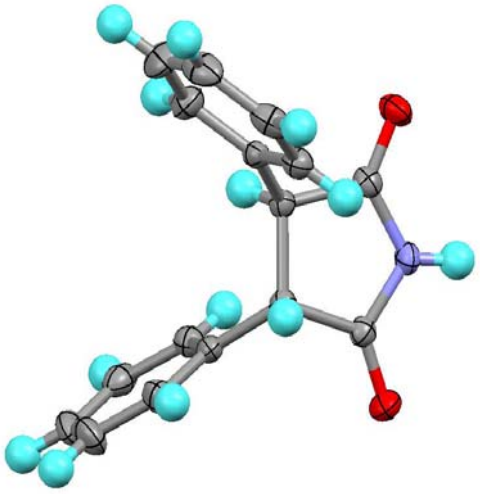

Figure S1. Ortep view of **3a** showing the atoms and thermal ellipsoids at 50% probability.

X-Ray single crystal analysis of *trans*-*N*-isopropyl-3,4-diphenylsuccinimide 3e

Crystal data of **3e** (recrystallized from a mixture of $CHCl_3$ and hexane); $C_{19}H_{19}NO_2$, *Mr* = 293.35, Orthorhombic space group $P2_12_12$, *a* = 17.0588(3)Å, *b* = 5.30480(10) Å, *c* = 8.58540(10) Å, *V* = 776.92(2) Å^3, *Z* = 2, ρ = 1.254 g cm^{-3}, in the final least-square refinement cycles on F^2, the model converged to R_1 = 0.0291, wR_2 = 0.0751, and GOF = 1.115 for 1267 reflections. CCDC 927188.

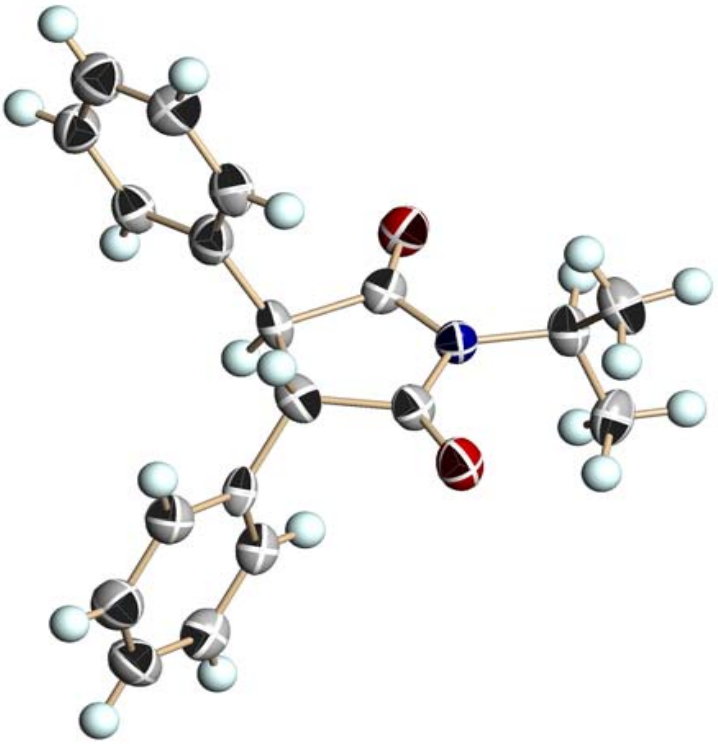

Figure S2. Ortep view of **3e** showing the atoms and thermal ellipsoids at 50% probability.

X-Ray single crystal analysis of *trans*-*N*-phenethyl-3,4-diphenylsuccinimide 3g

Crystal data of **3g** (recrystallized from a mixture of $CHCl_3$ and hexane); $C_{24}H_{21}NO_2$, *Mr* = 355.42, Orthorhombic space group *P*bca, *a* = 11.729(4) Å, *b* = 8.588(3) Å, *c* = 37.580(13) Å, *V* = 3785(2) Å^3, *Z* = 8, ρ = 1.247 g cm^{-3}, in the final least-square refinement cycles on F^2, the model converged to R_1 = 0. 1009, wR_2 = 0. 2307, and GOF = 1.107 for 3613 reflections. CCDC 927189.

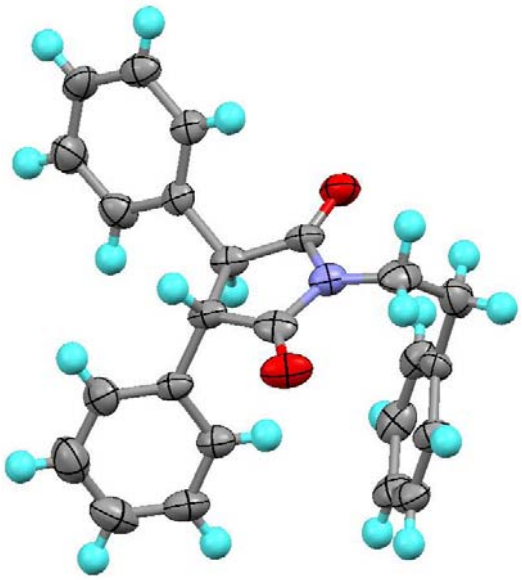

Figure S3. Ortep view of **3g** showing the atoms and thermal ellipsoids at 50% probability.

X-Ray single crystal analysis of *trans*-*N*-phenyl-3,4-diphenylsuccinimide 3h

Crystal data of **3h** (recrystallized from a mixture of $CHCl_3$ and hexane); $C_{22}H_{17}NO_2$, *Mr* = 327.37, Orthorhombic space group *P*bcn, *a* = 17.1516(3) Å, *b* = 11.5890(2) Å, *c* = 8.4053(2) Å, *V* = 1670.72(6) $Å^3$, *Z* = 4, ρ = 1.301 g cm^{-3}, in the final least-square refinement cycles on F^2, the model converged to R_1 = 0.0357, wR_2 = 0.1003, and GOF = 1.023 for 1498 reflections. CCDC 927190.

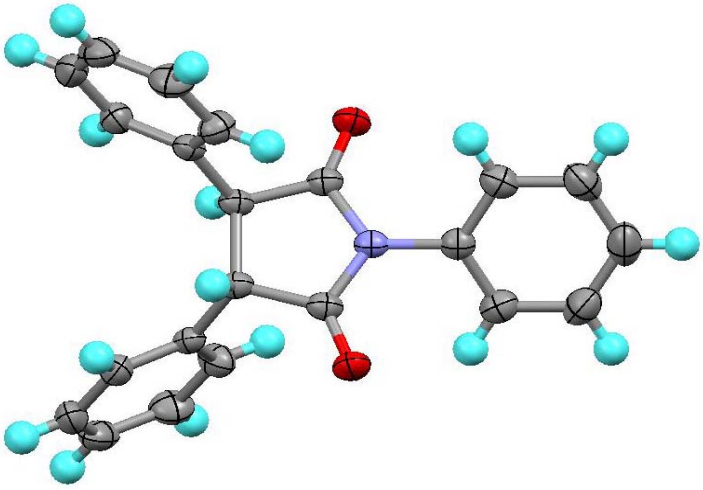

Figure. S4. Ortep view of **3h** showing the atoms and thermal ellipsoids at 50% probability.

X-Ray single crystal analysis of *trans*-*N*-(3-methylphenyl)-3,4-diphenylsuccinimide 3j

Crystal data of **3j** (recrystallized from a mixture of $CHCl_3$ and hexane); $C_{23}H_{19}NO_2$, *Mr* = 341.39, Monoclinic, space group $P2_1/c$, *a* = 8.8218(6) Å, *b* = 17.4333(12) Å, *c* = 11.8783(9) Å, β = 100.3140(10) °, *V* = 1797.3(2) $Å^3$, *Z* = 4, ρ = 1.262 g cm^{-3}, in the final least-square refinement cycles on F^2, the model converged to R_1 = 0.0447, wR_2 = 0.1096, and GOF = 1.037 for 3190 reflections. Data CCDC 927191.

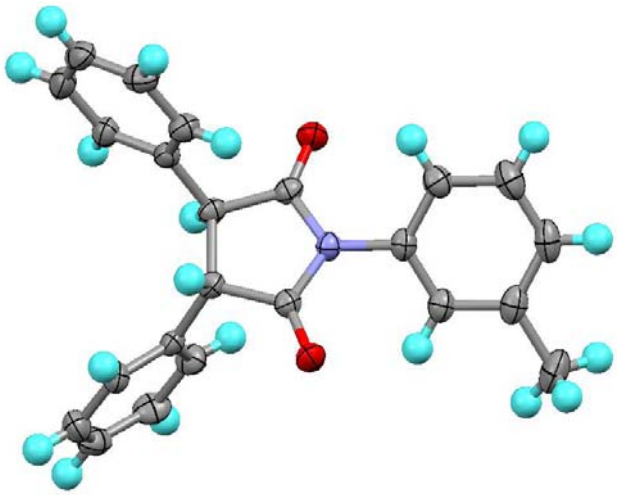

Figure S5. Ortep view of **3j** showing the atoms and thermal ellipsoids at 50% probability.

Kinetic studies for racemization of 3d, 3e, and 3i

Each optically active *trans*-3,4-diphenylsuccinimide **3** was dissolved into a $CHCl_3$ solution (5.0 x 10^{-2} mol L^{-1}) containing catalytic amount of DBU and the change of optical rotation was monitored by an DIP 370 polarimeter (JASCO) at 20 °C. The activation parameters were obtained from the Eyring equation. The first-order kinetic plots of the decay profile angle of rotation was shown as a plot of ln(*ee*) versus time (eq. 1), and the rate of racemization (k_{rac}) was calculated from the slope of the line. The free energy barrier ($\Delta G^{\ddagger}$) for racemization is calculated according to the Eyring equation (eq. 2), and then half-life were calculated according to equation (eq. 3).

$$\ln(ee)=k_{rac}t \qquad \text{(eq. 1)}$$

$$k_{rac}=(\boldsymbol{k}T/\boldsymbol{h})\exp(-\Delta G^{\ddagger}/\boldsymbol{R}T) \qquad \text{(eq. 2)}$$

$$t_{1/2} = \ln 2/2k_{rac} \qquad \text{(eq. 3)}$$

k_{rac} : rate of racemization, ***h*** : Planck constant, ***k*** : Boltzmann constant, ***R*** : gas constant, ***T*** : temperature

Table S1. Kinetic parameters for racemization of *trans*-3,4-diphenylsuccinimides **3d**, **3e**, **3i**[a]

trans-**3**	DBU (eq)	$t_{1/2}$ (sec)	$\Delta G^{\ddagger}$ (kcal mol^{-1})
3d	0.10	120	20.6
3e	0.10	186	20.8
3i	0.05	99	20.4

[a]Measurement conditions: 5.0 x 10^{-2} mol L^{-1} of each succinimide in $CHCl_3$ at 20 °C.

Method for crystallization

Crystallization of achiral *cis*-2 or racemic *trans*-3 by evaporating solvent (method A): [S2]

A mixed solution of chloroform (2 ml) and hexane (1 ml) containing 30 mg of *cis*-**2d,** *cis*-**2e**, or racemic *trans*-**3i** and 0.05 - 0.10 mol % of DBU was stirred in a test tube or a vial at room temperature until all solvent evaporated. After the solidified substrate was dissolved in chloroform and DBU was removed through short silica gel column, the enantiomer excess (ee) of *trans*-**3** was analysed by HPLC using chiral column (Daicel Ind. CHIRALPAK AD-H for **3d**; CHIRALPAK IA-3 for **3e**; CHIRALPAK IA for **3i**).

References

[S1] 3,4-Dipnenylmaleimides were synthesized from 3,4-diphenylmaleic anhydride and corresponding amines. (a) Zehavi, U. *J. Org. Chem.* **1976**, 2821-2825. (b) Aoyama, H; Sakamoto, M.; Omote, Y. *J. Am. Chem. Soc.* **1980**, *102*, 6902-6903.

[S2] Yagishita, F.; Ishikawa, H.; Onuki, T.; Hachiya, S.; Mino, T.; Sakamoto, M. *Angew. Chem. Int. Ed.* **2012**, *51*, 13023 –13025.

Figure S7. ^{1}H NMR of *cis*-*N*-propyl-3,4-diphenylsuccinimide 2d

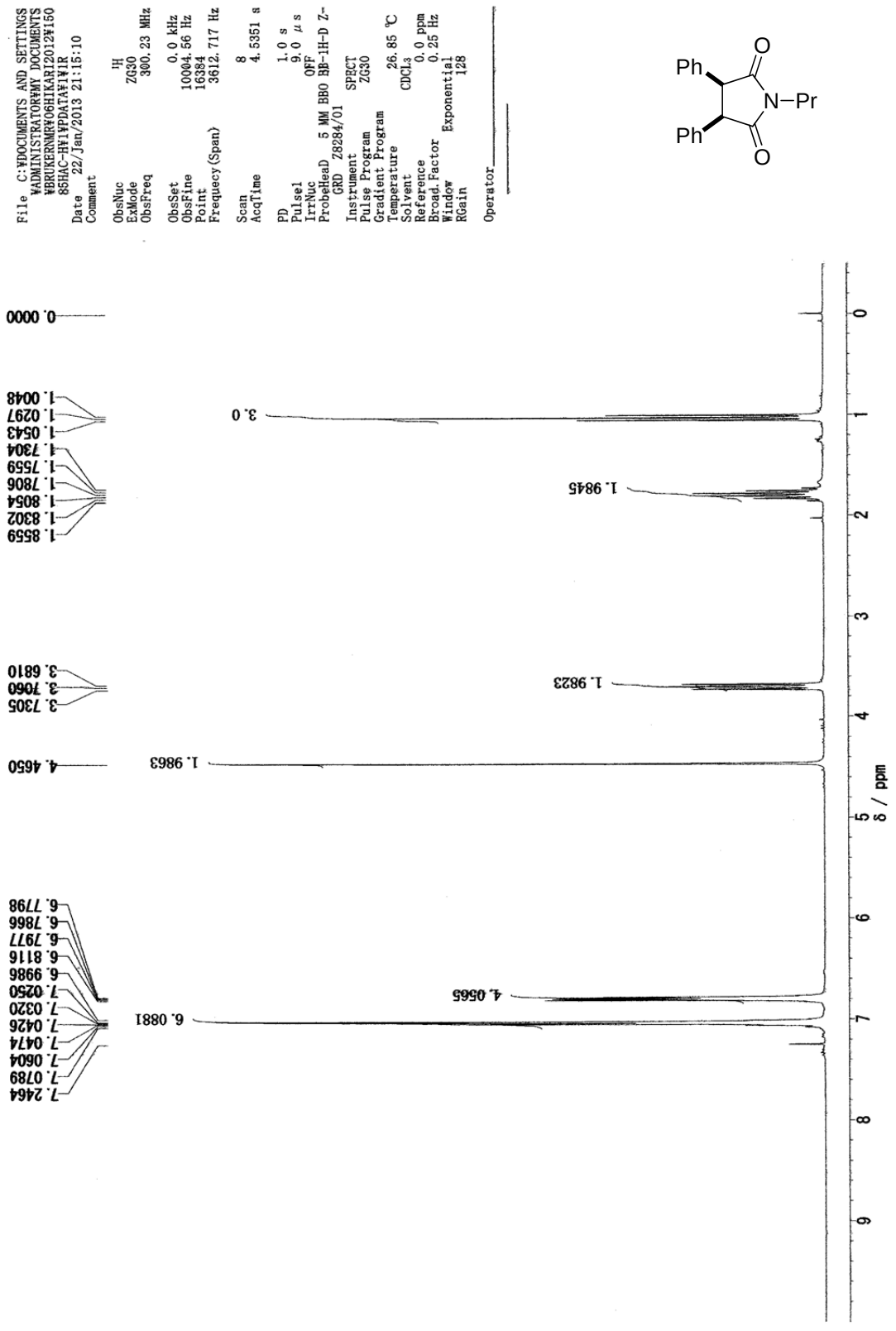

Figure S8. ^{13}C NMR of *cis*-*N*-propyl-3,4-diphenylsuccinimide 2d

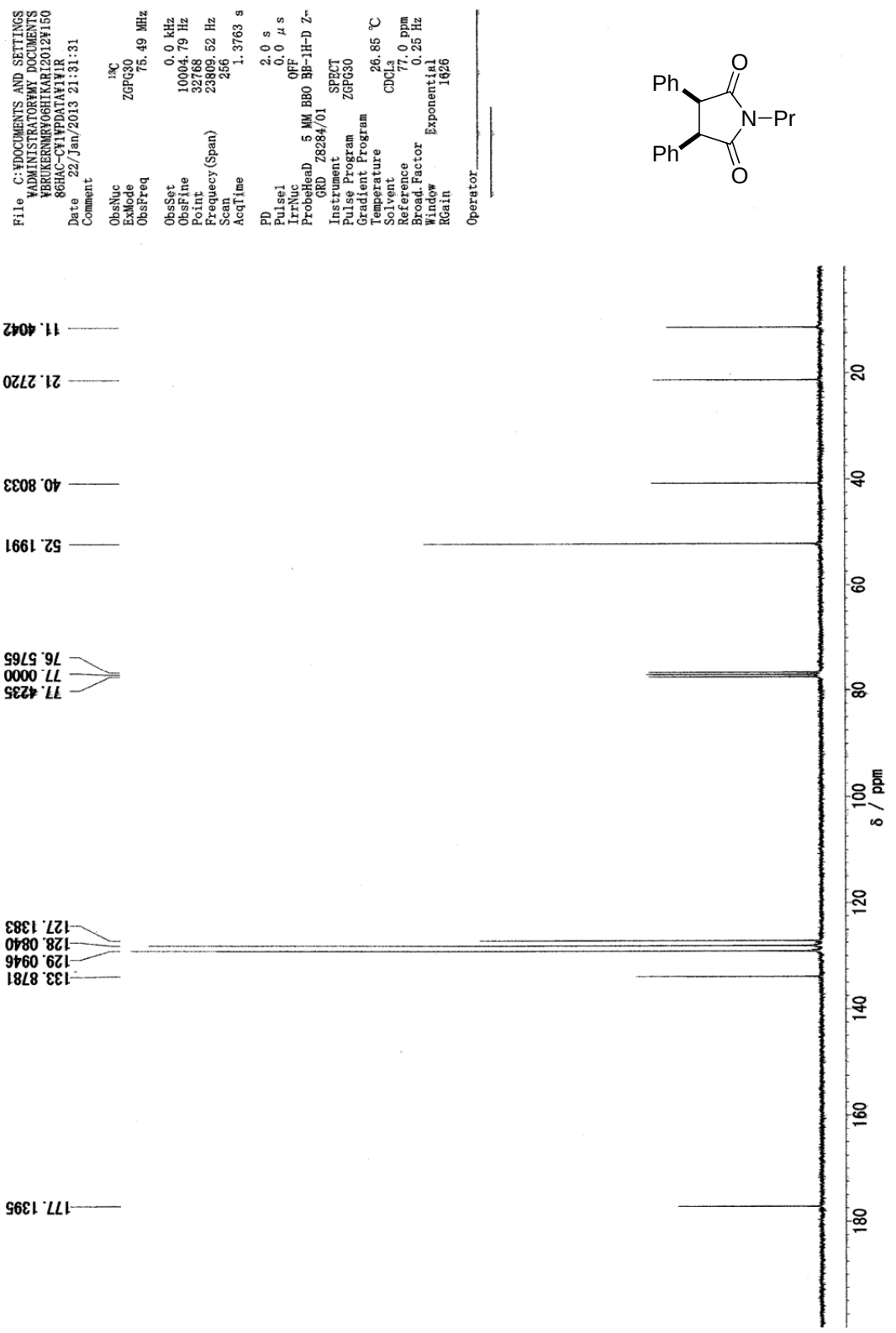

Figure S9. ^{1}H NMR spectrum for *cis*-*N*-isopropyl-3,4-diphenylsuccinimide 2e

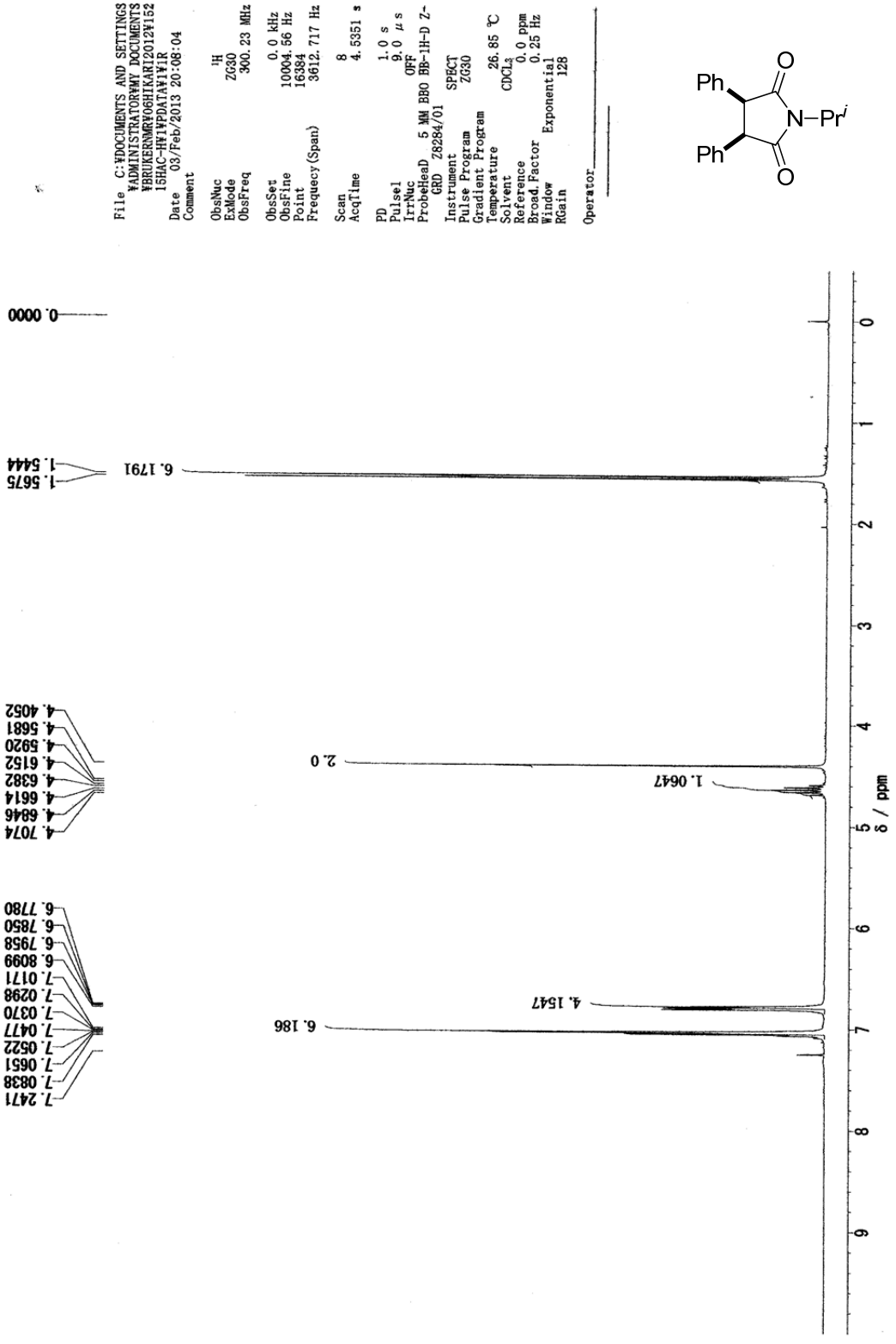

Figure S10. ^{13}C NMR spectrum for *cis*-*N*-isopropyl-3,4-diphenylsuccinimide 2e

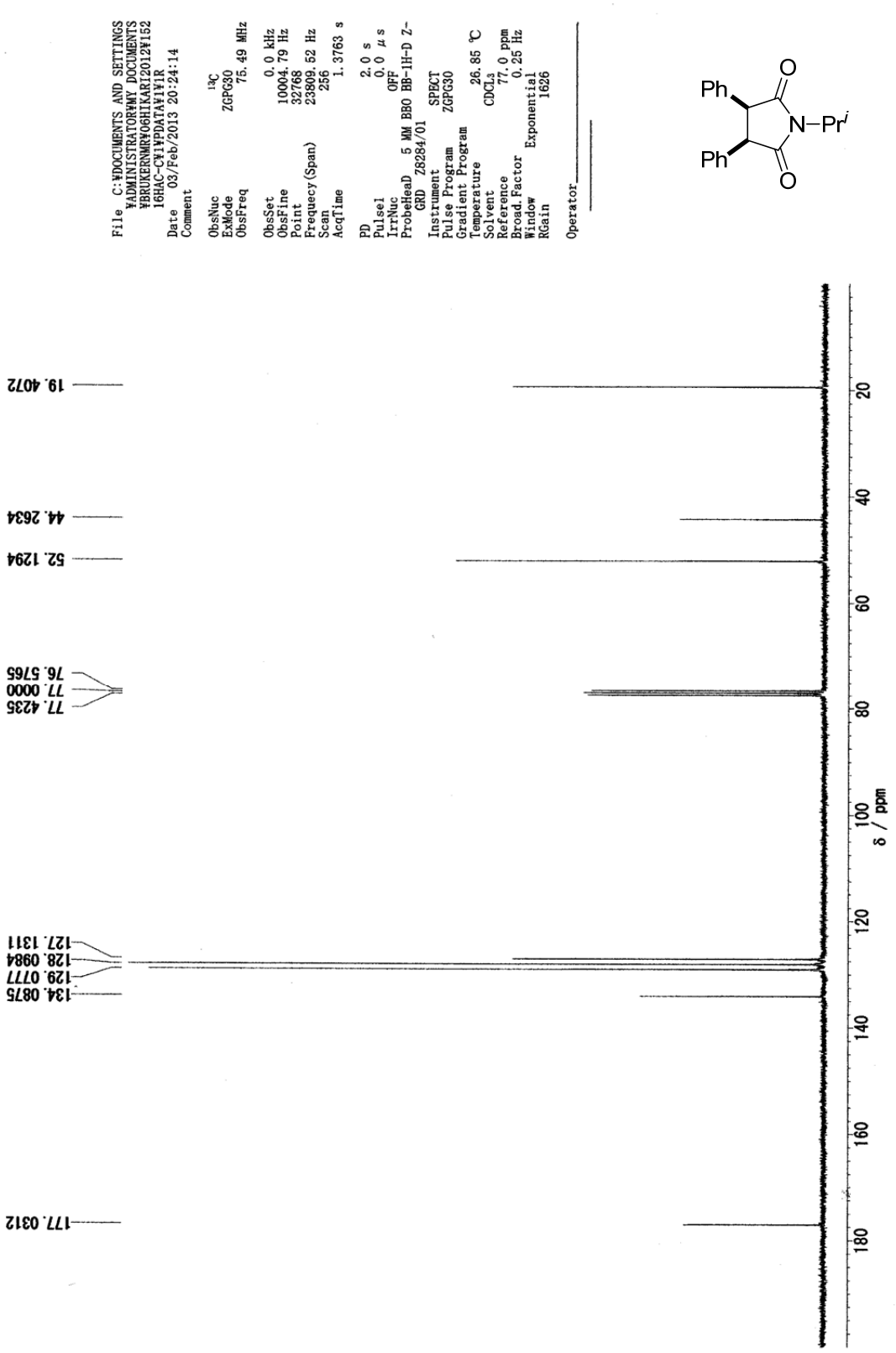

Figure S11. ^{1}H NMR spectrum for *trans*-3,4-diphenylsuccinimide 3a

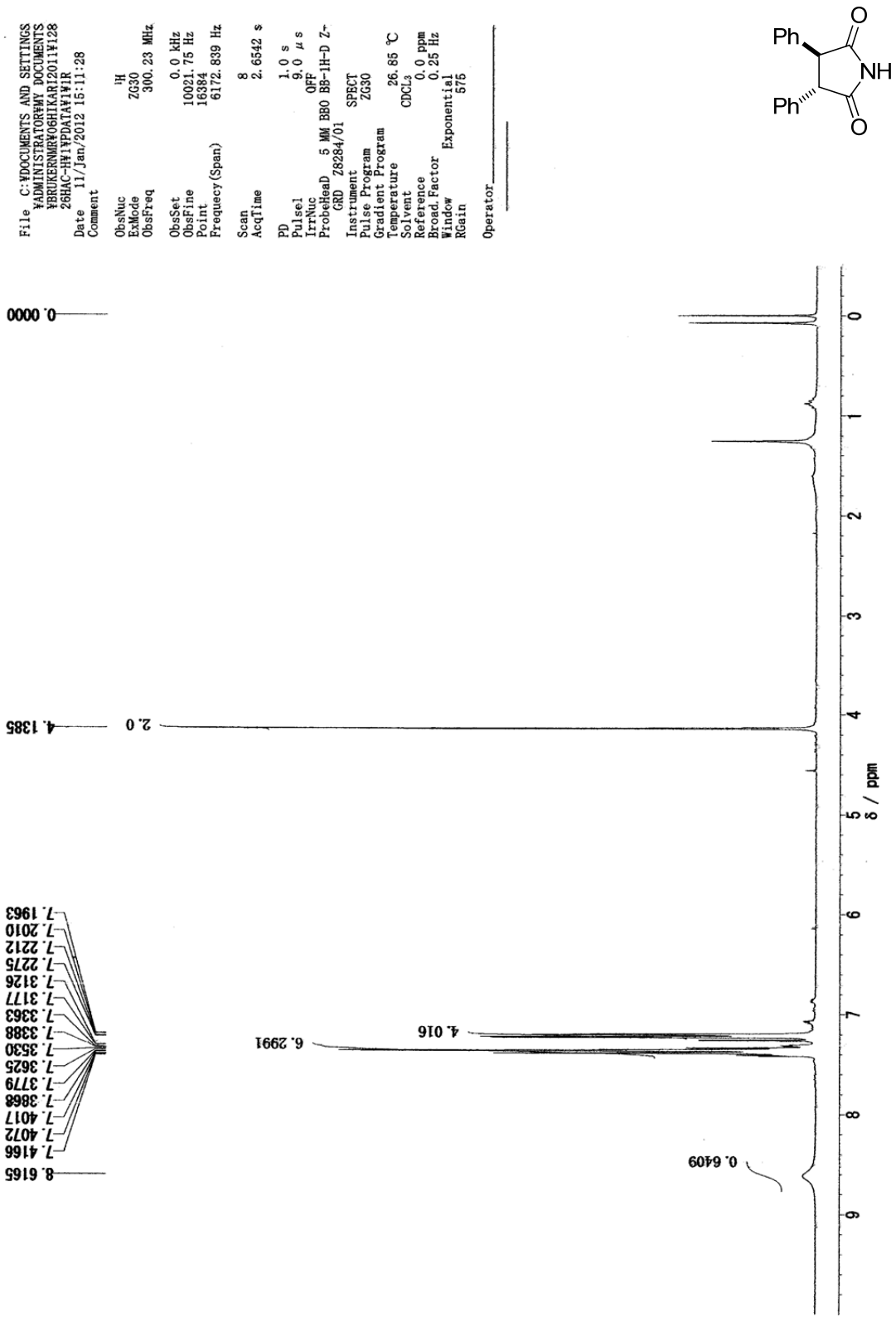

Figure S12. ^{13}C NMR spectrum for *trans*-3,4-diphenylsuccinimide 3a

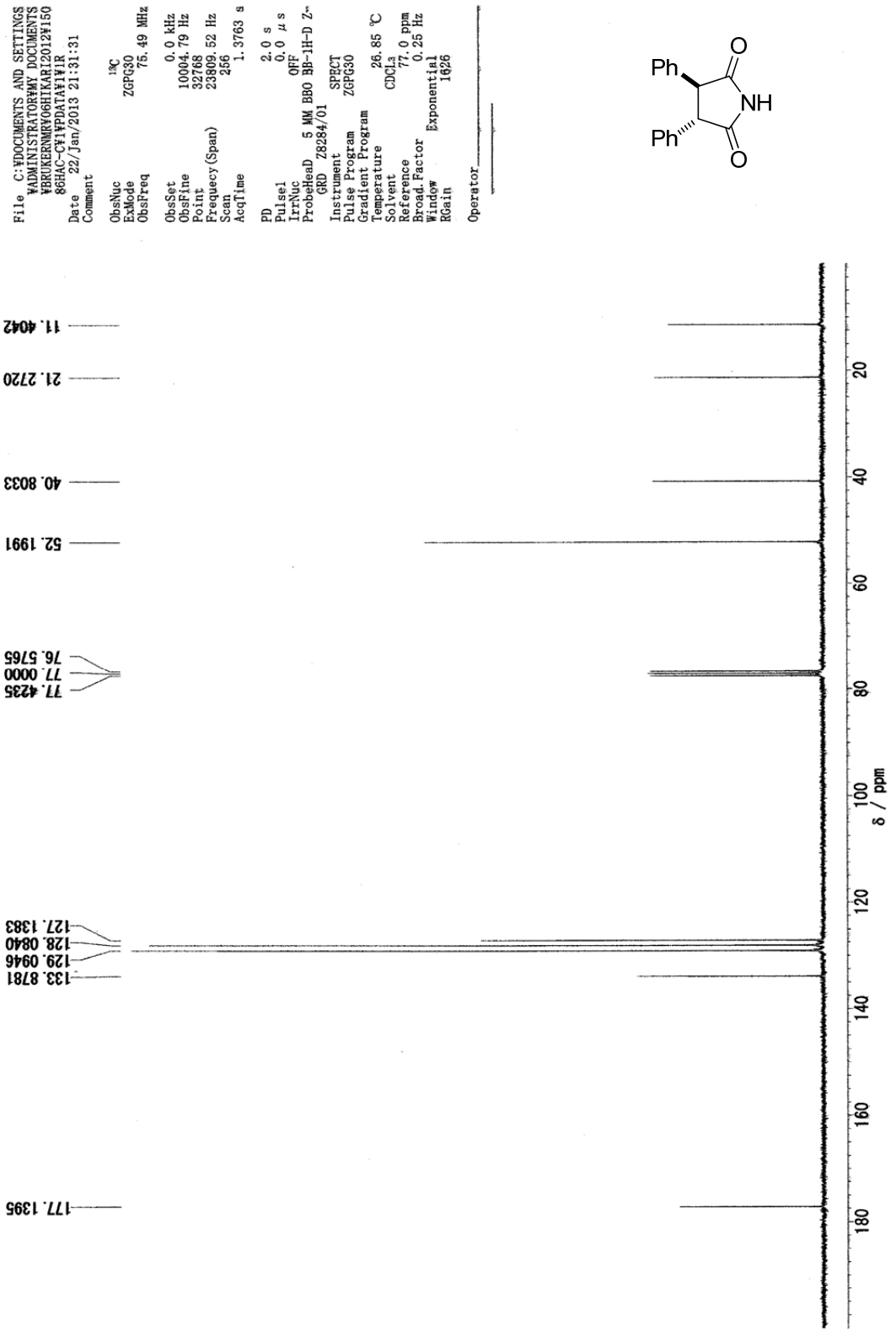

Figure S13. ^{1}H NMR spectrum for *trans*-*N*-methyl-3,4-diphenylsuccinimide 3b

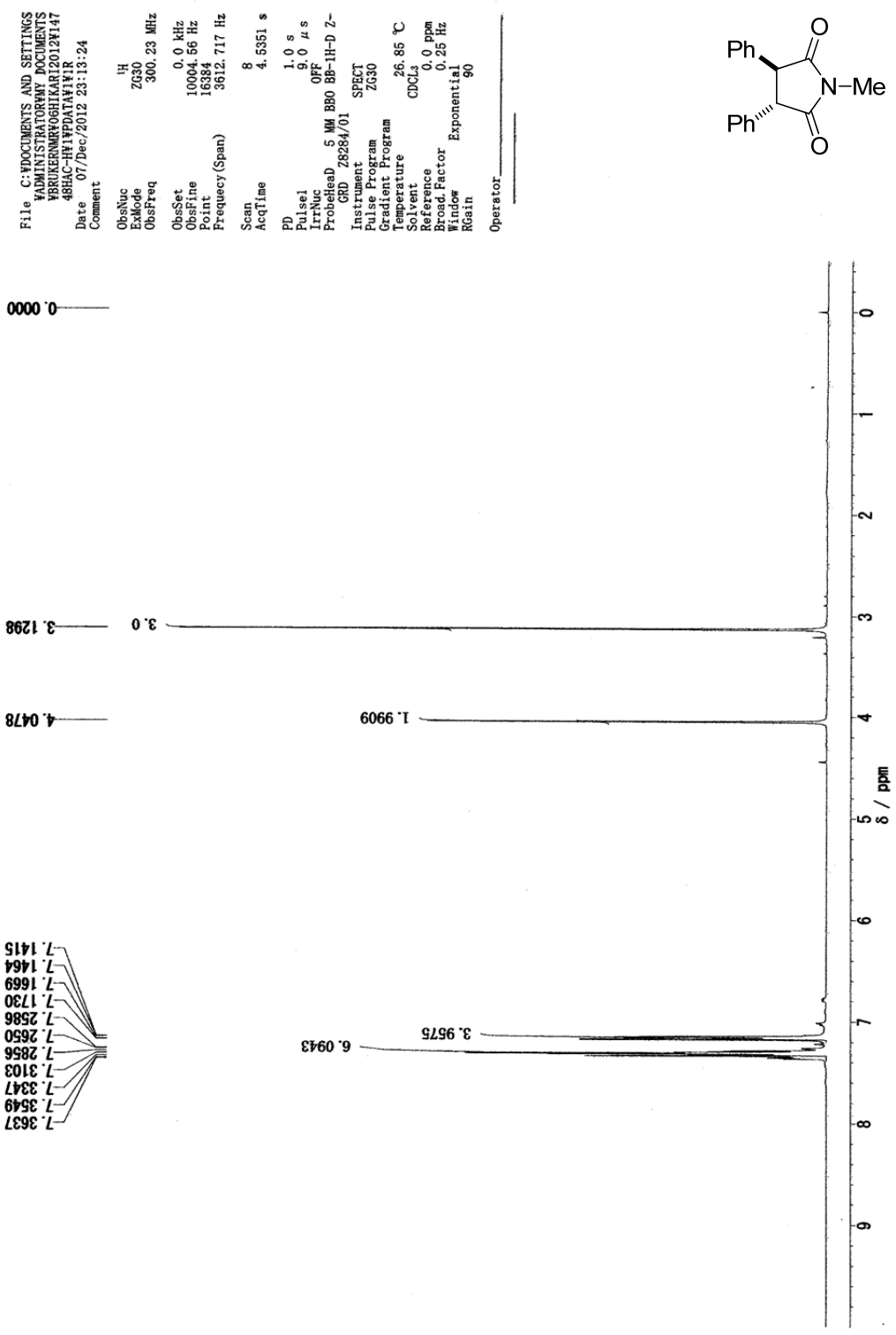

Figure S14. ^{13}C NMR spectrum for *trans*-*N*-methyl-3,4-diphenylsuccinimide 3b

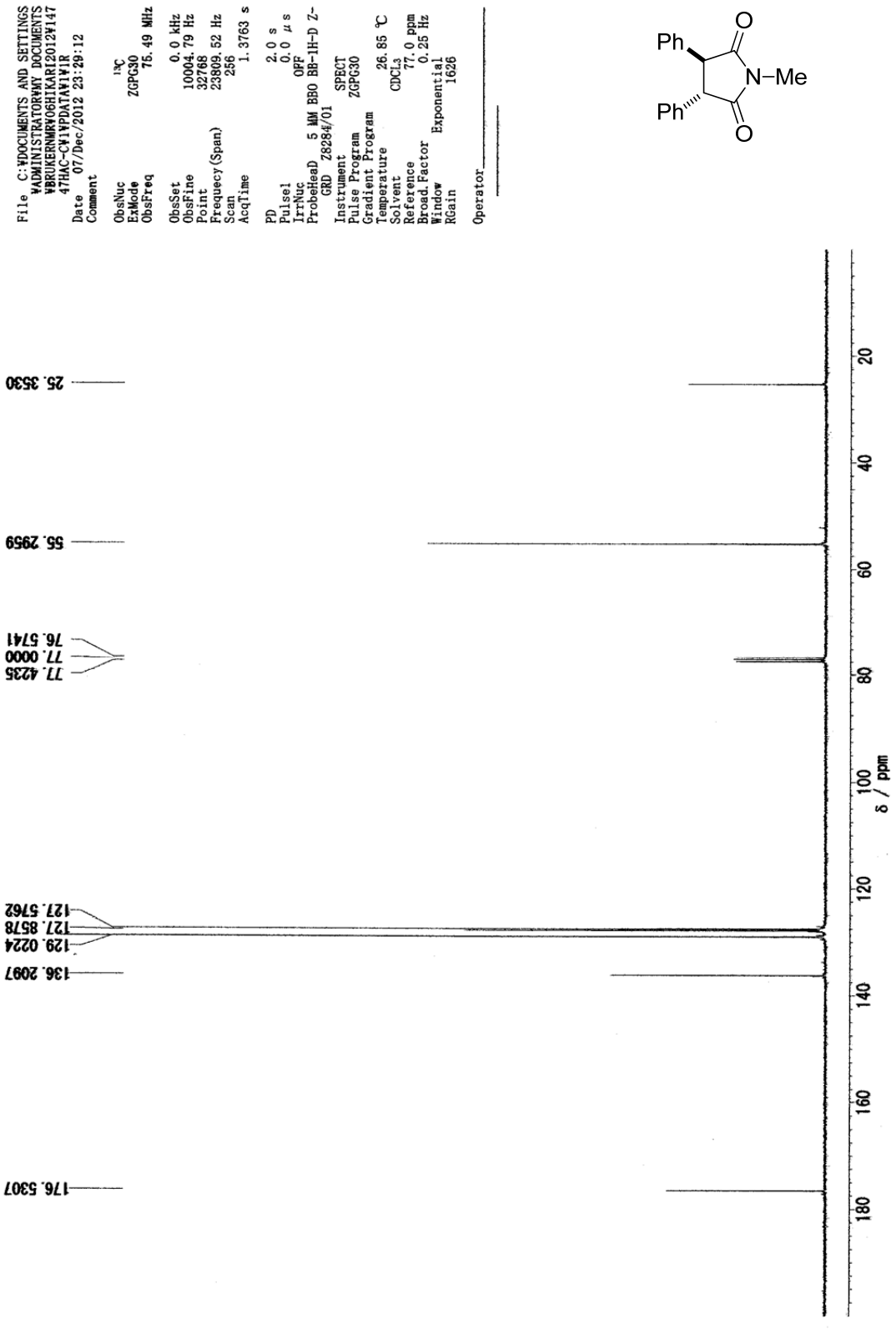

Figure S15. ^{1}H NMR spectrum for *trans*-*N*-ethyl-3,4-diphenylsuccinimide 3c

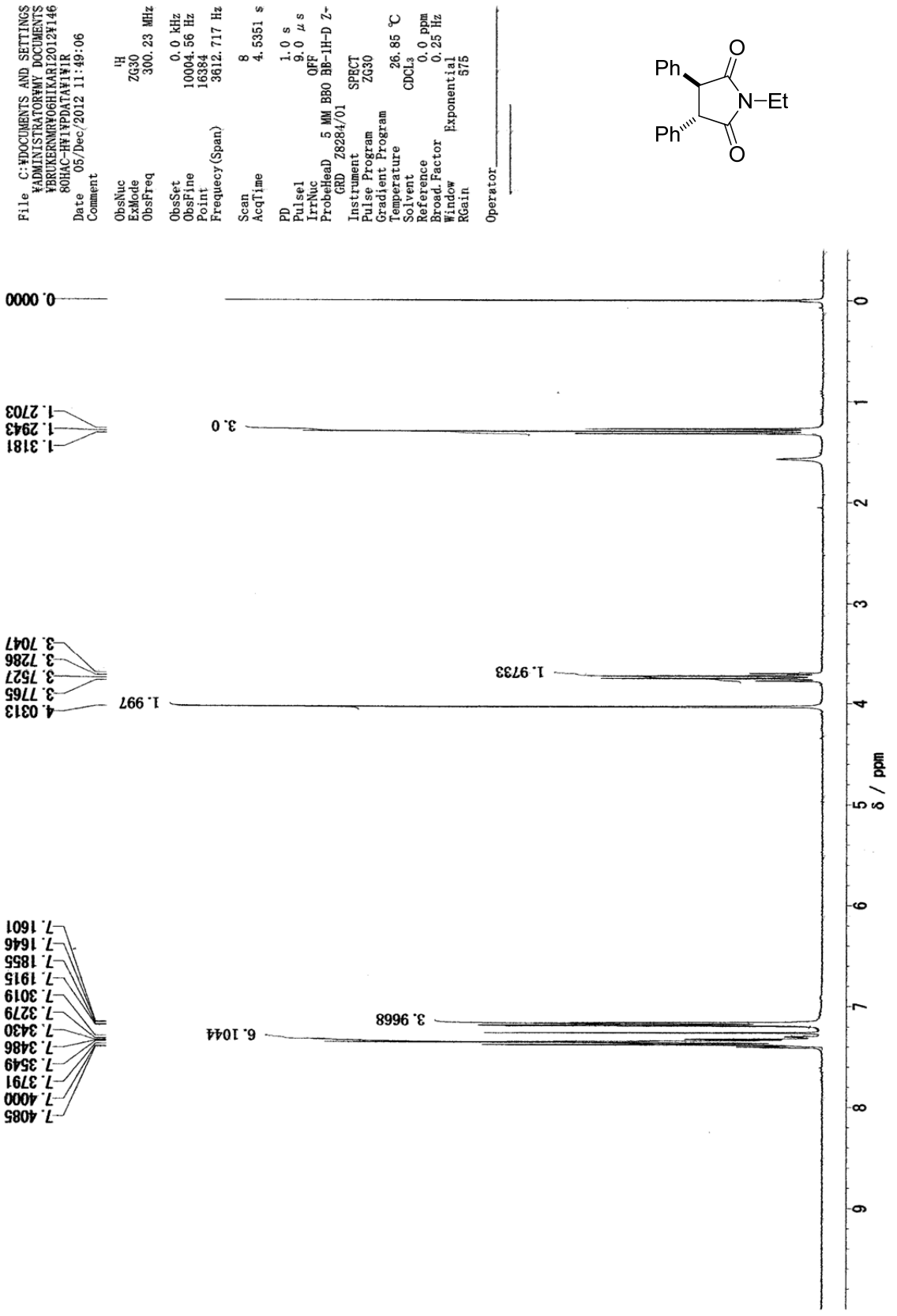

Figure S16. ^{13}C NMR spectrum for *trans*-*N*-ethyl-3,4-diphenylsuccinimide 3c

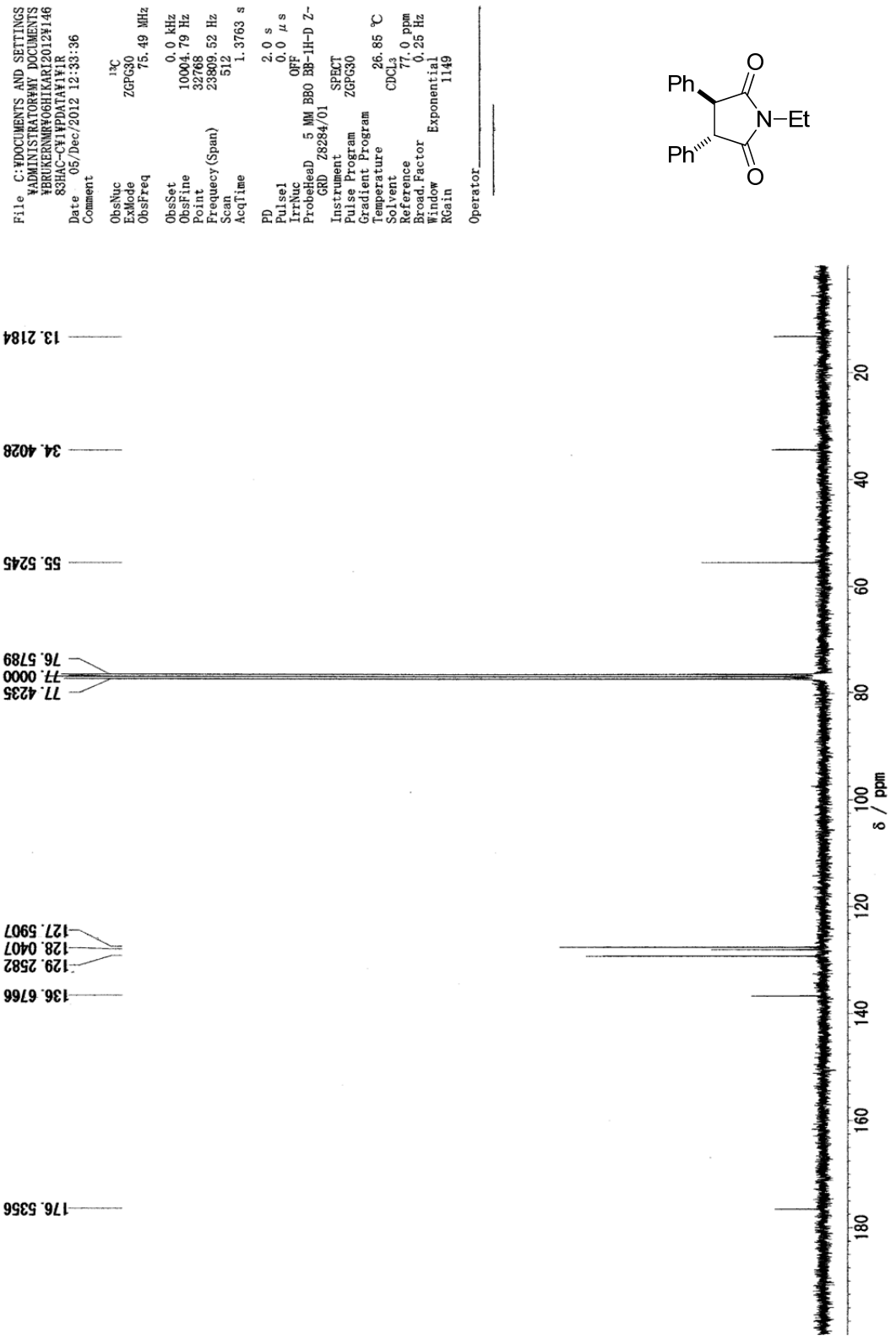

Figure S17. ^{1}H NMR spectrum for *trans*-*N*-propyl-3,4-diphenylsuccinimide 3d

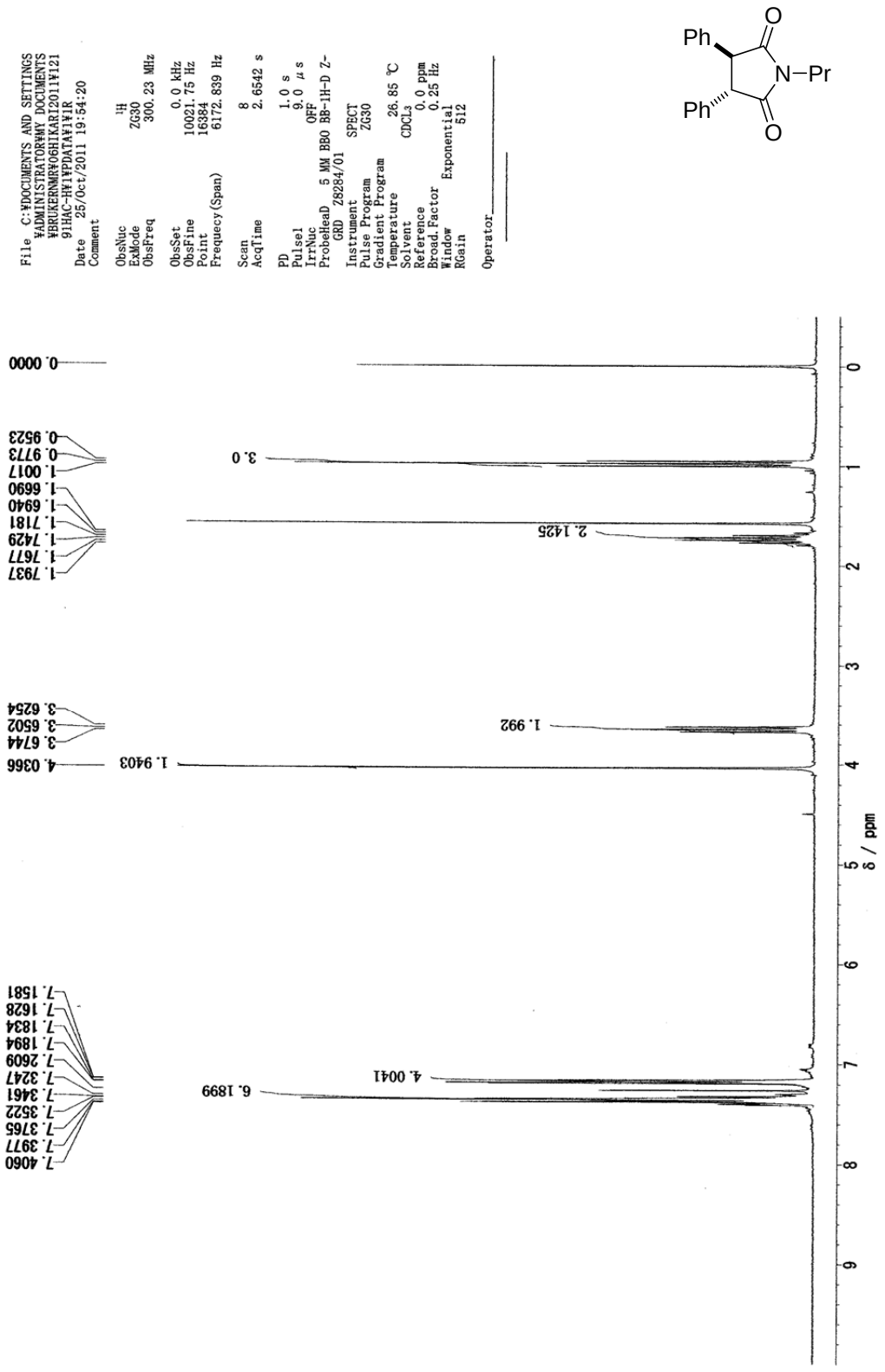

Figure S18. ^{13}C NMR spectrum for *trans*-*N*-propyl-3,4-diphenylsuccinimide 3d

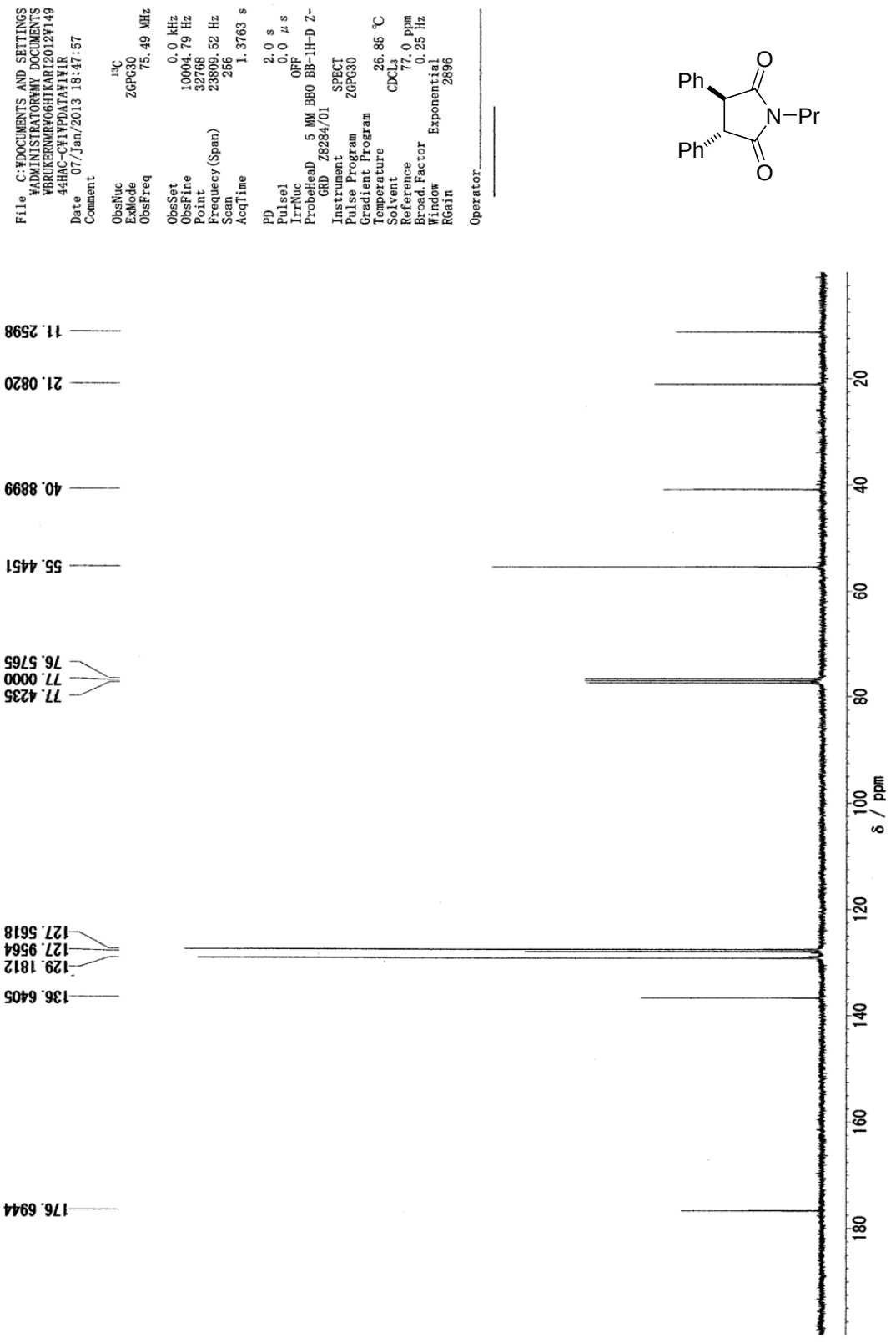

Figure S19. ^{1}H NMR spectrum for *trans*-*N*-isopropyl-3,4-diphenylsuccinimide 3e

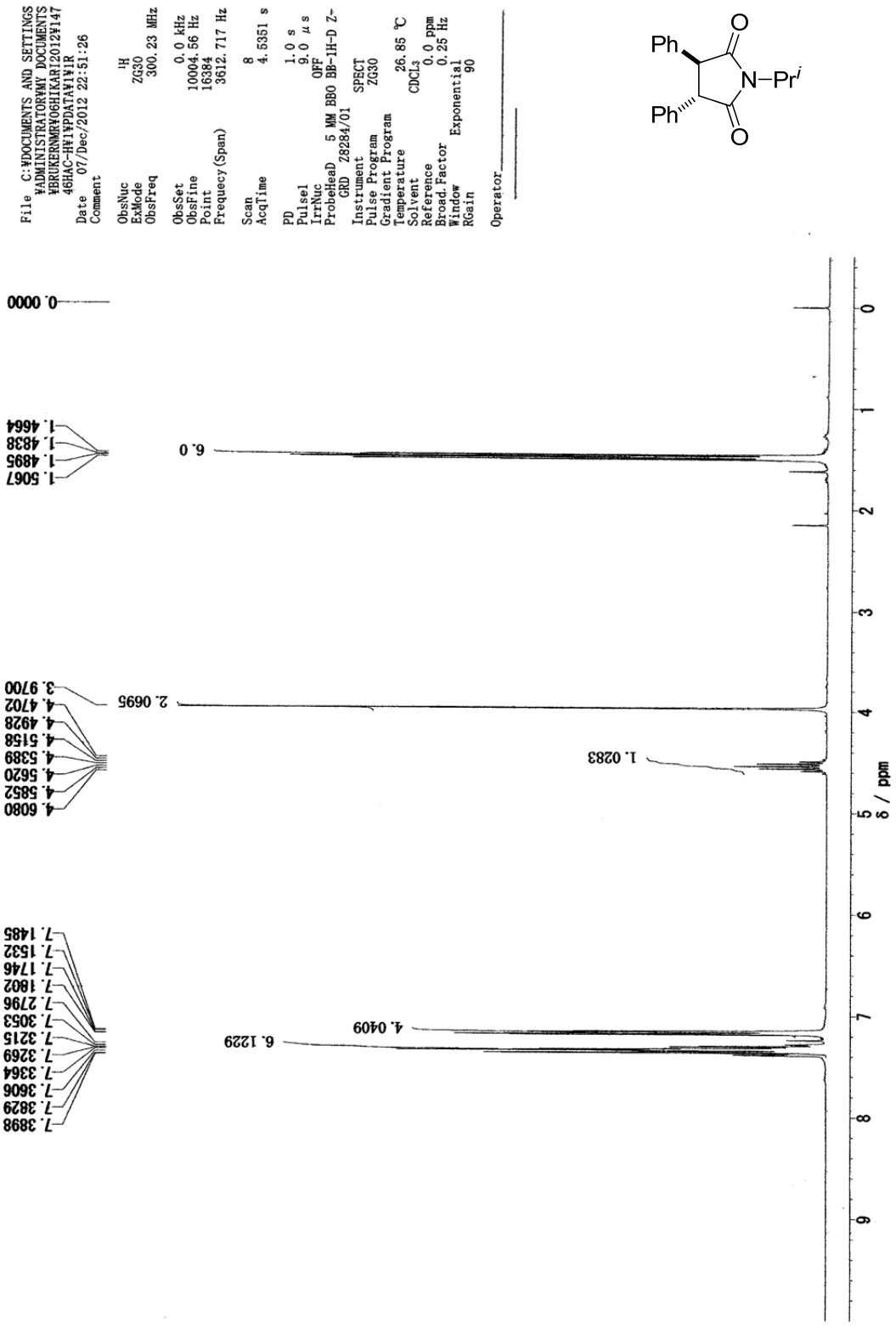

Figure S20. ^{13}C NMR spectrum for *trans*-*N*-isopropyl-3,4-diphenylsuccinimide 3e

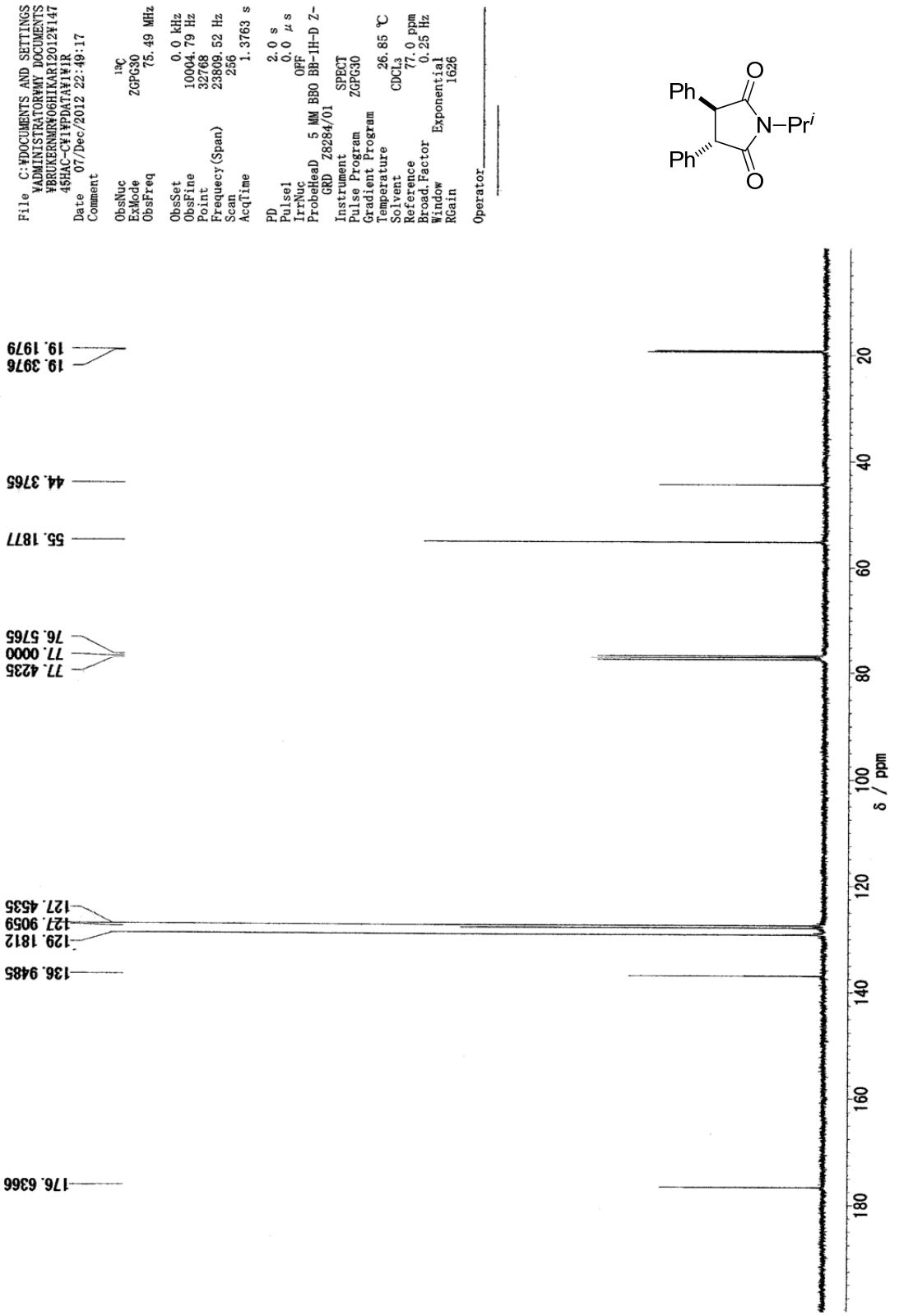

Figure S21. ^{1}H NMR spectrum for *trans*-*N*-benzyl-3,4-diphenylsuccinimide 3f

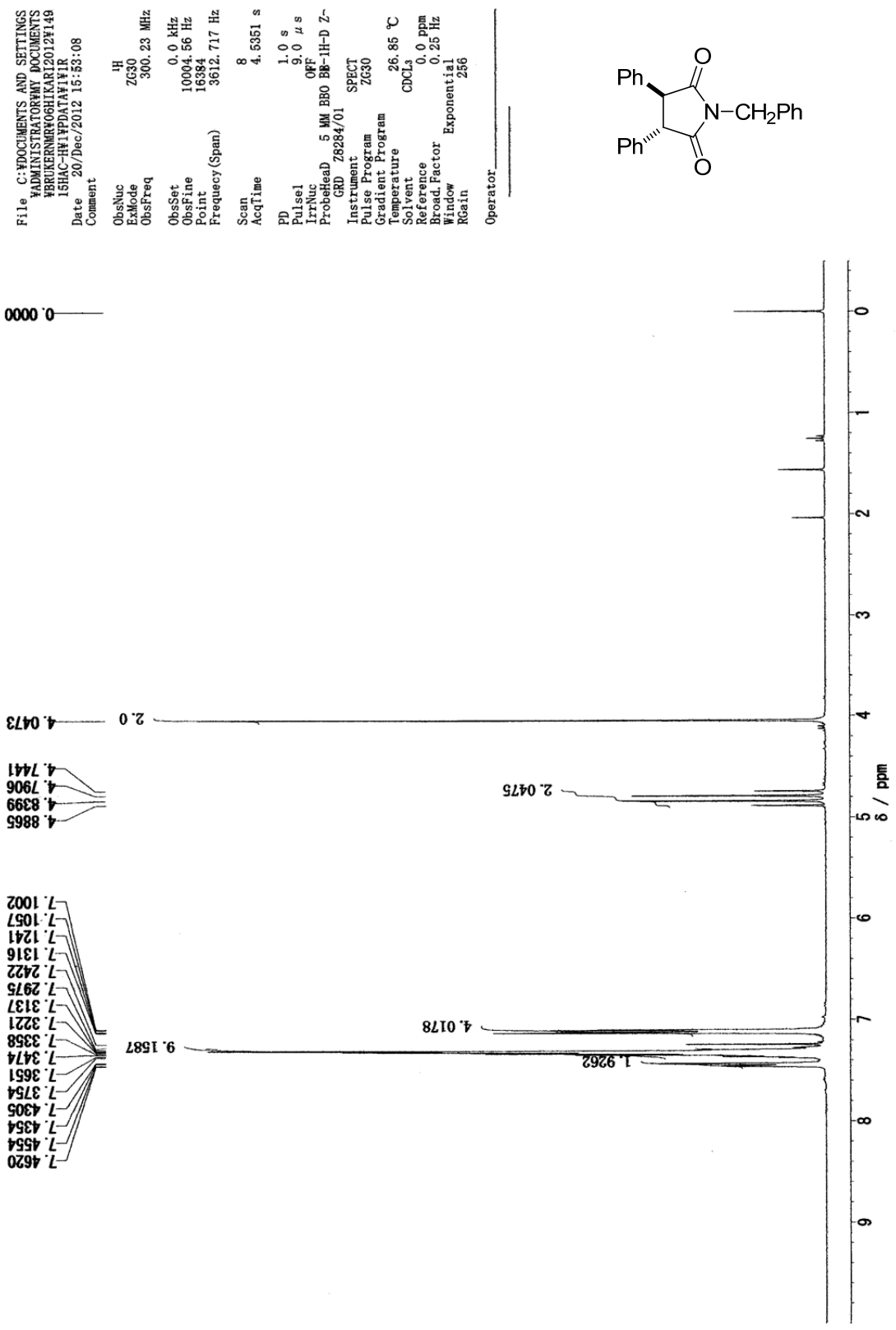

Figure S22. ^{13}C NMR spectrum for *trans*-*N*-benzyl-3,4-diphenylsuccinimide 3f

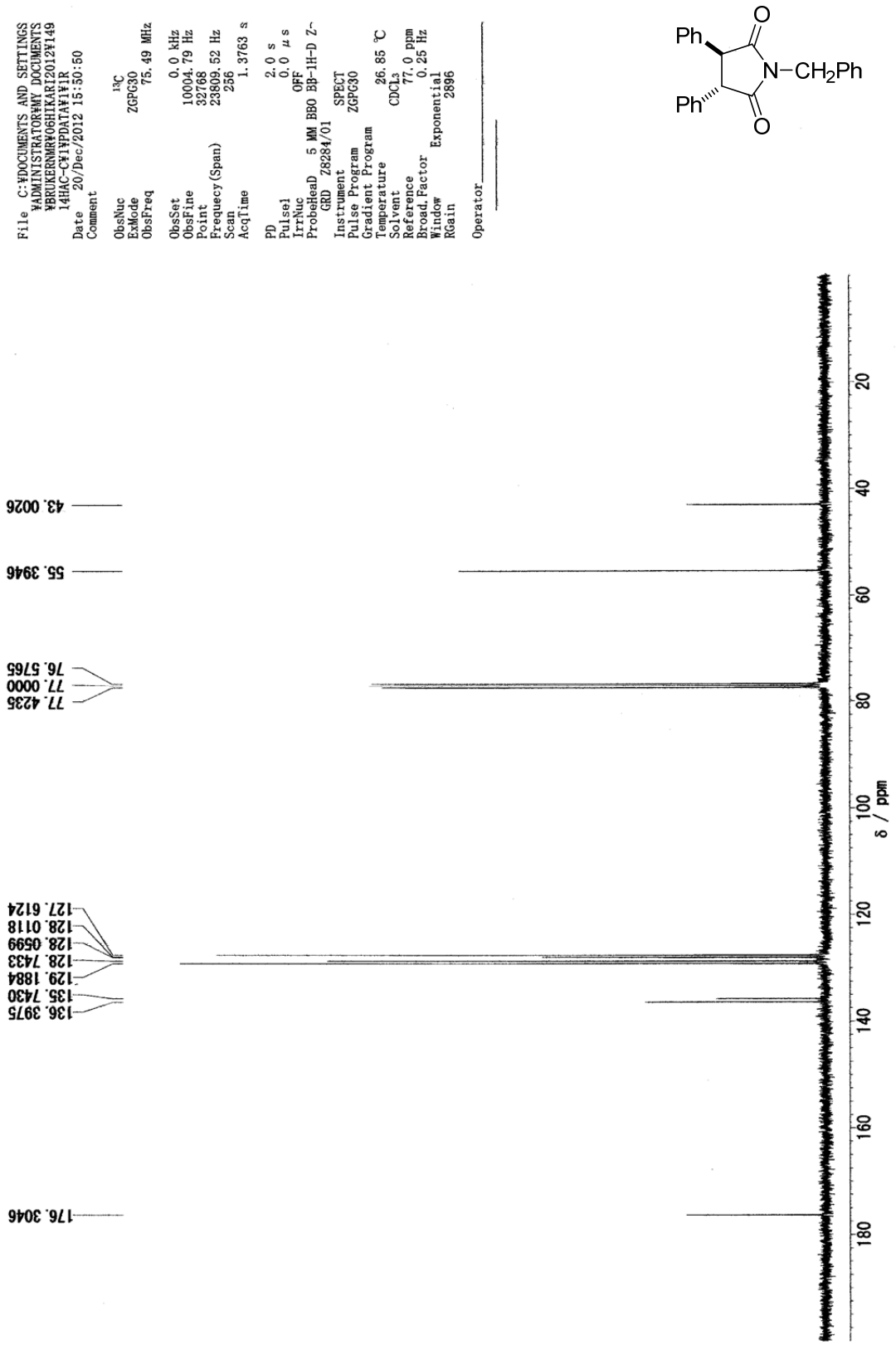

Figure S23. ^{1}H NMR spectrum for *trans*-*N*-phenethyl-3,4-diphenylsuccinimide 3g

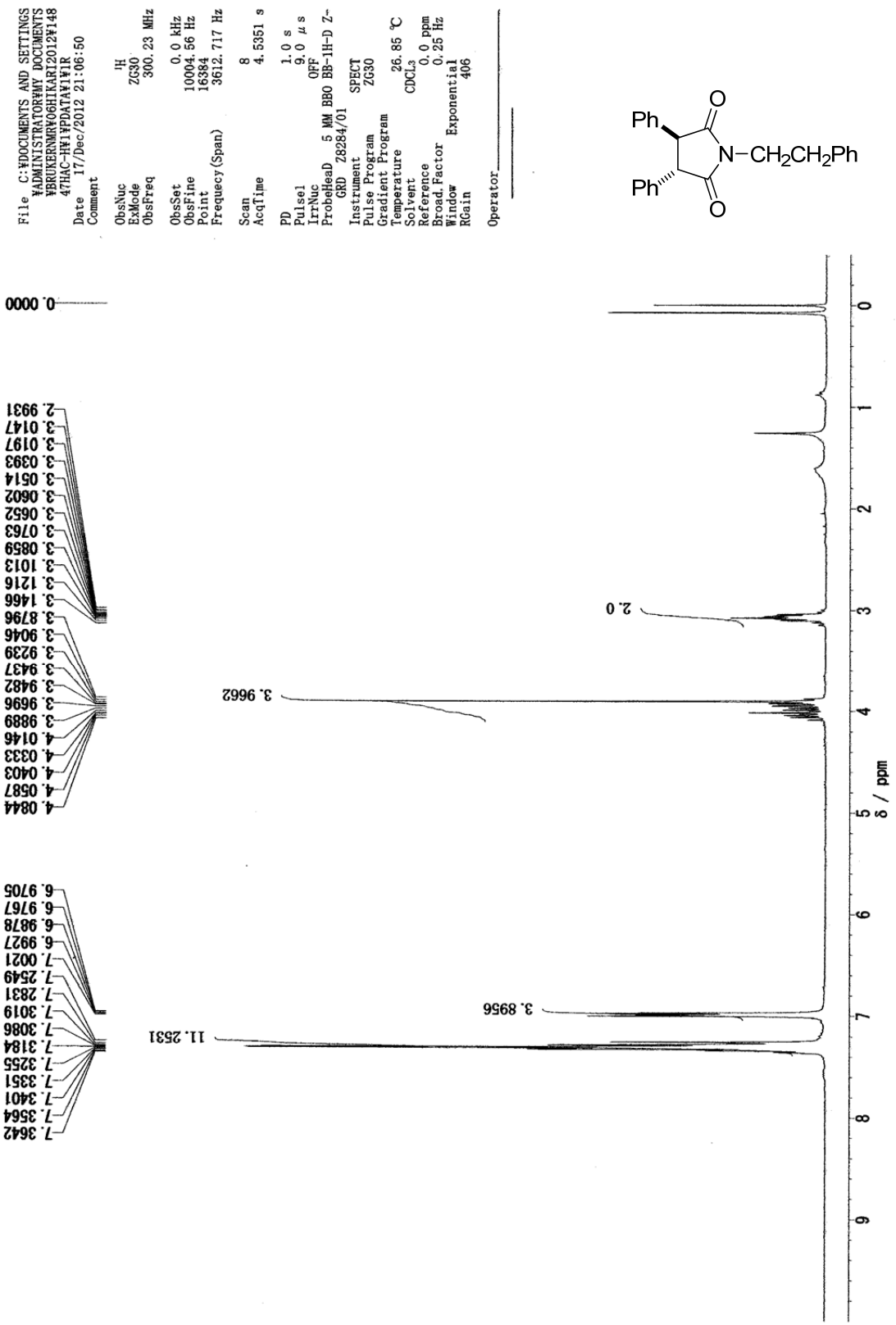

Figure S24. ^{13}C NMR spectrum for *trans*-*N*-phenyl-3,4-diphenylsuccinimide 3g

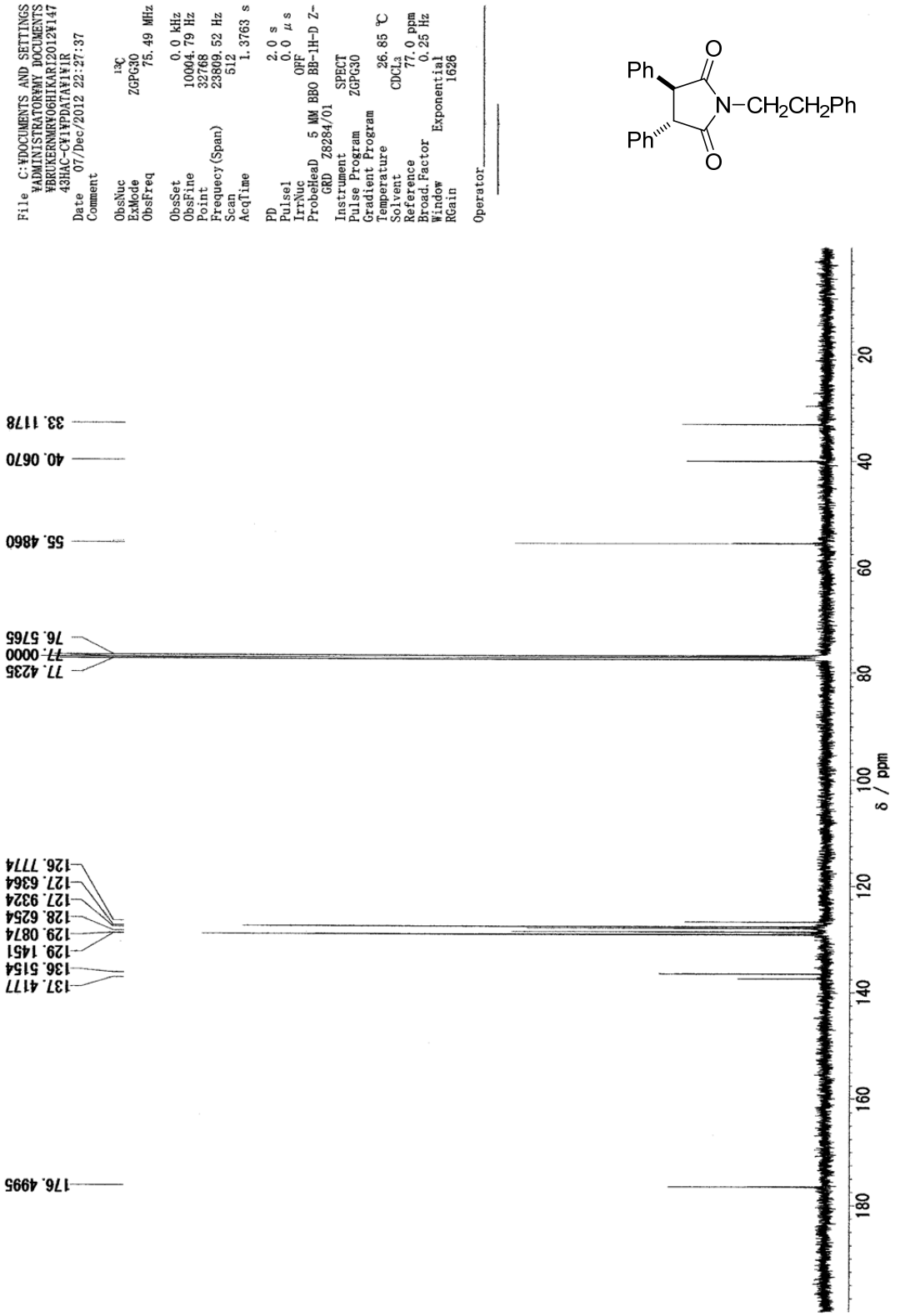

Figure S25. ^{1}H NMR spectrum for *trans*-*N*-phenyl-3,4-diphenylsuccinimide 3h

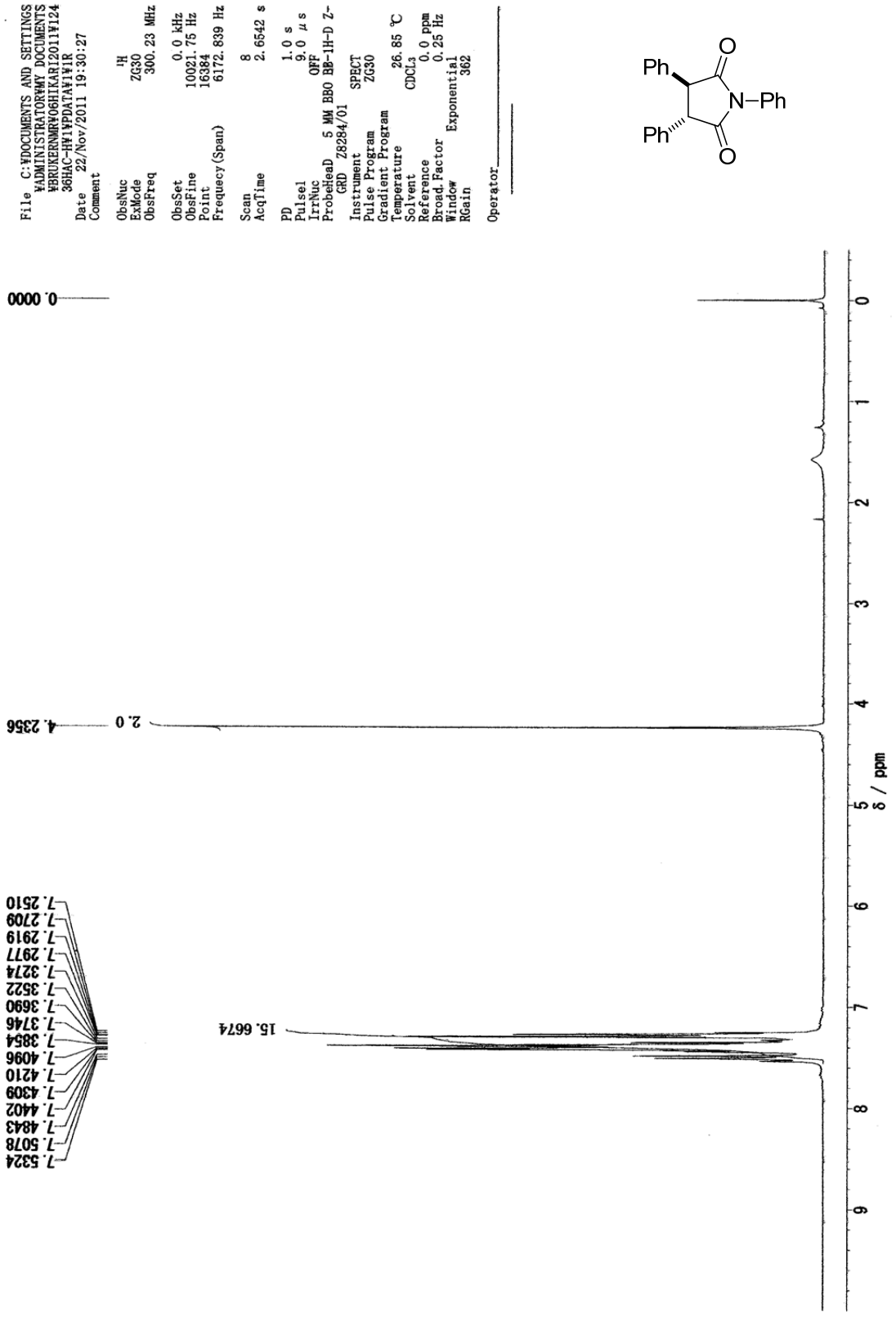

Figure S26. ^{13}C NMR spectrum for *trans*-*N*-phenyl-3,4-diphenylsuccinimide 3h

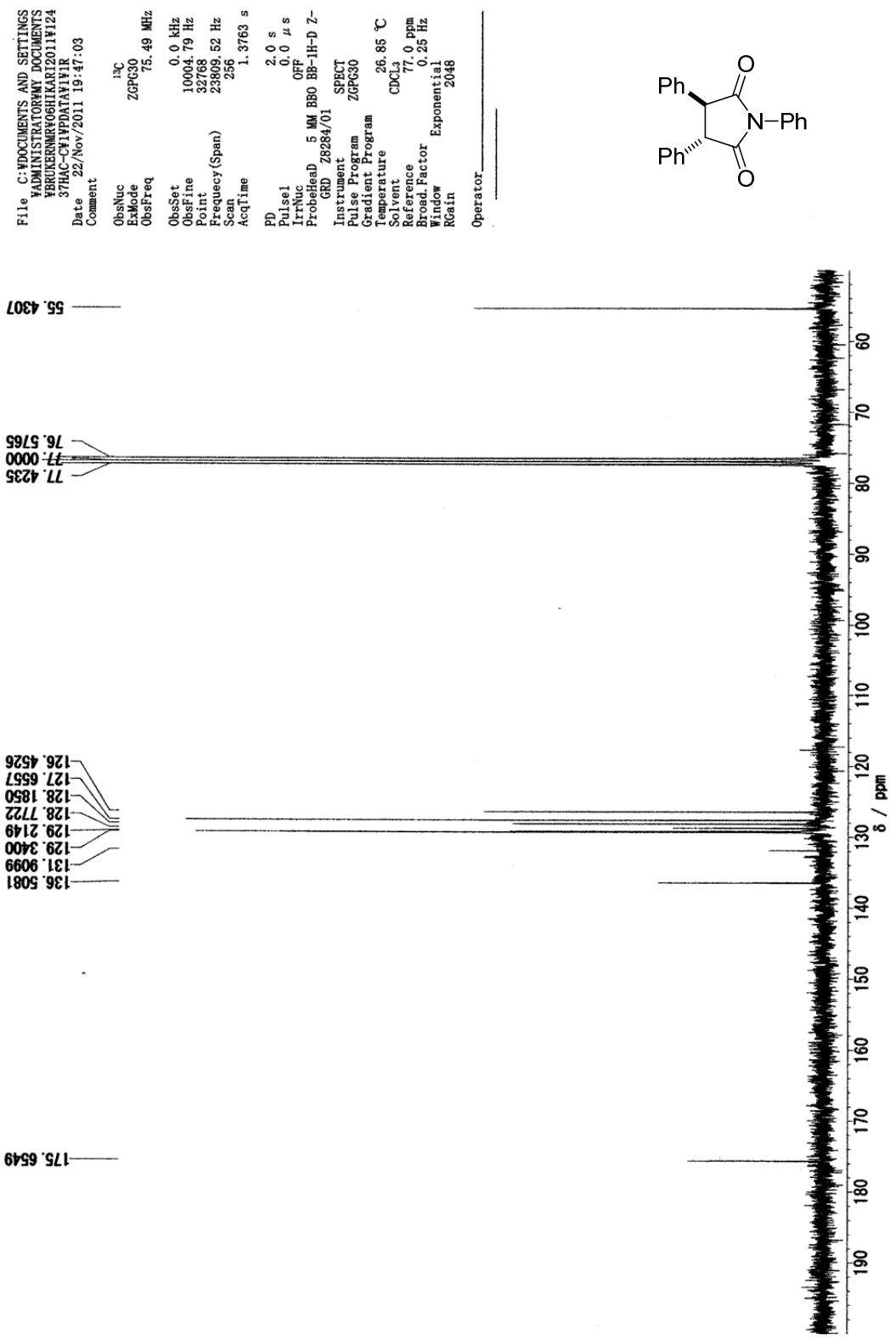

Figure S27. ^{1}H NMR spectrum for *trans*-*N*-(2-methylphenyl)-3,4-diphenylsuccinimide 3i

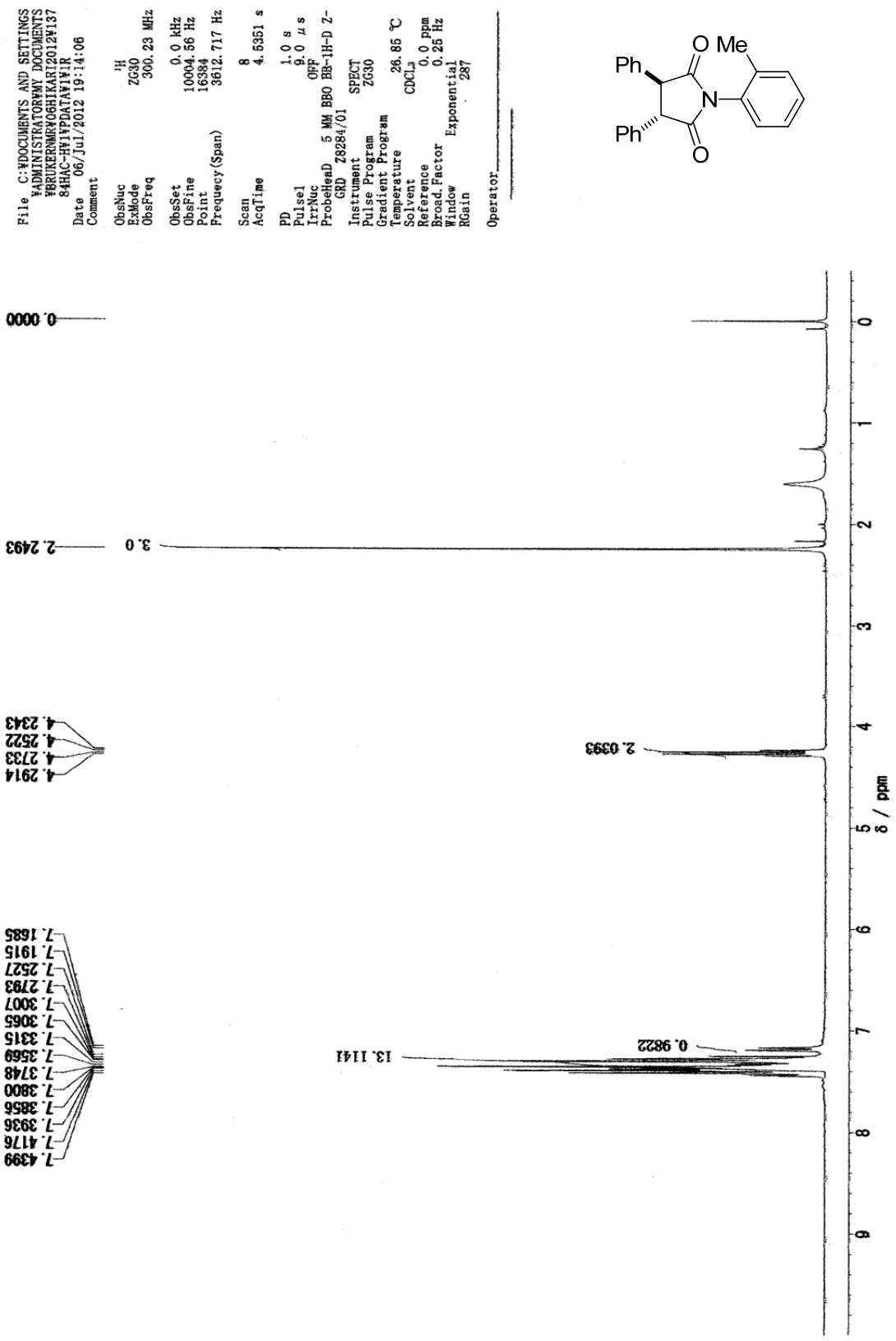

Figure S28. ^{13}C NMR spectrum for *trans*-*N*-(2-methylphenyl)-3,4-diphenylsuccinimide 3i

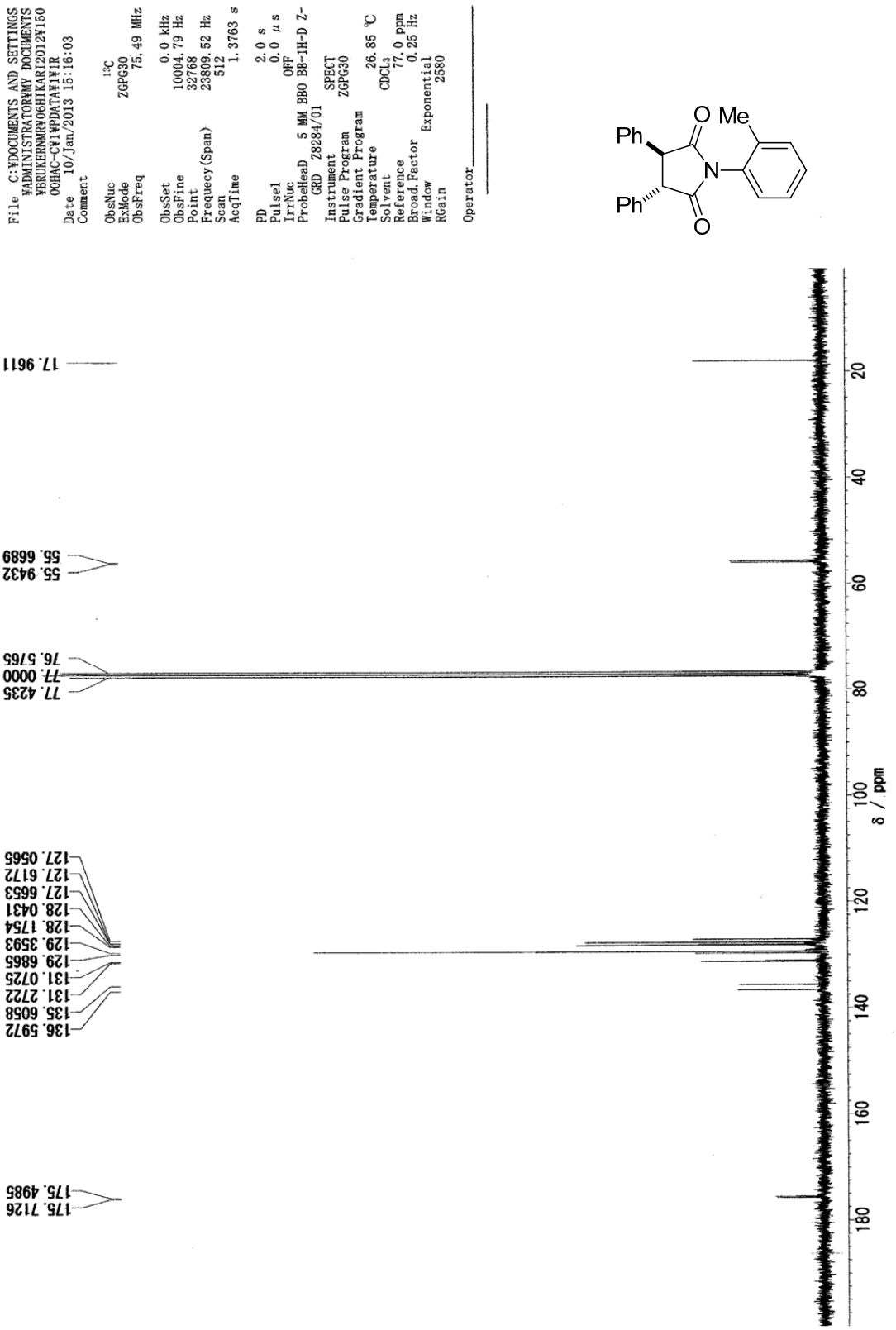

Figure S29. ^{1}H NMR spectrum for *trans*-*N*-(3-methylphenyl)-3,4-diphenylsuccinimide 3j

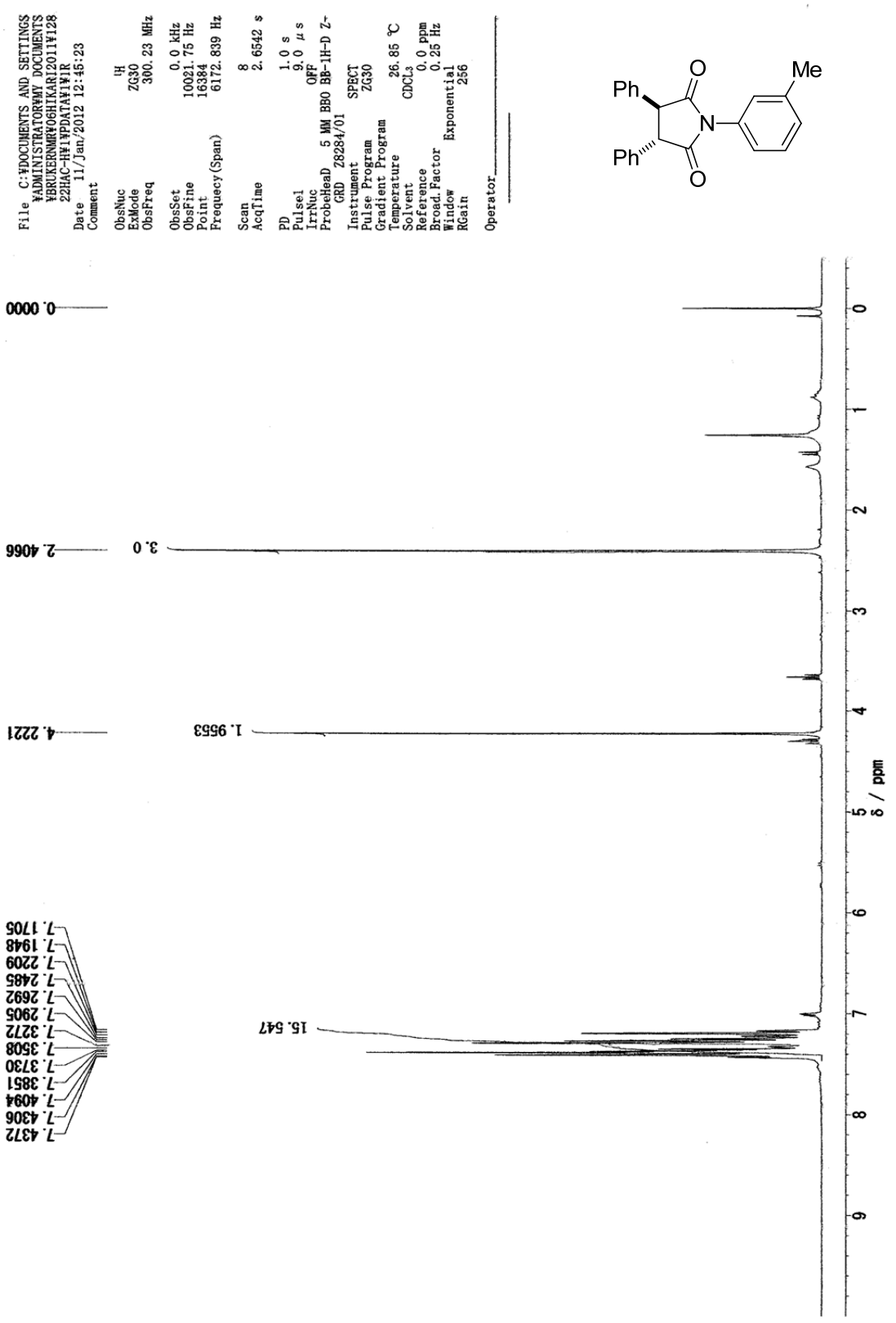

Figure S30. ^{13}C NMR spectrum for *trans*-*N*-(3-methylphenyl)-3,4-diphenylsuccinimide 3j

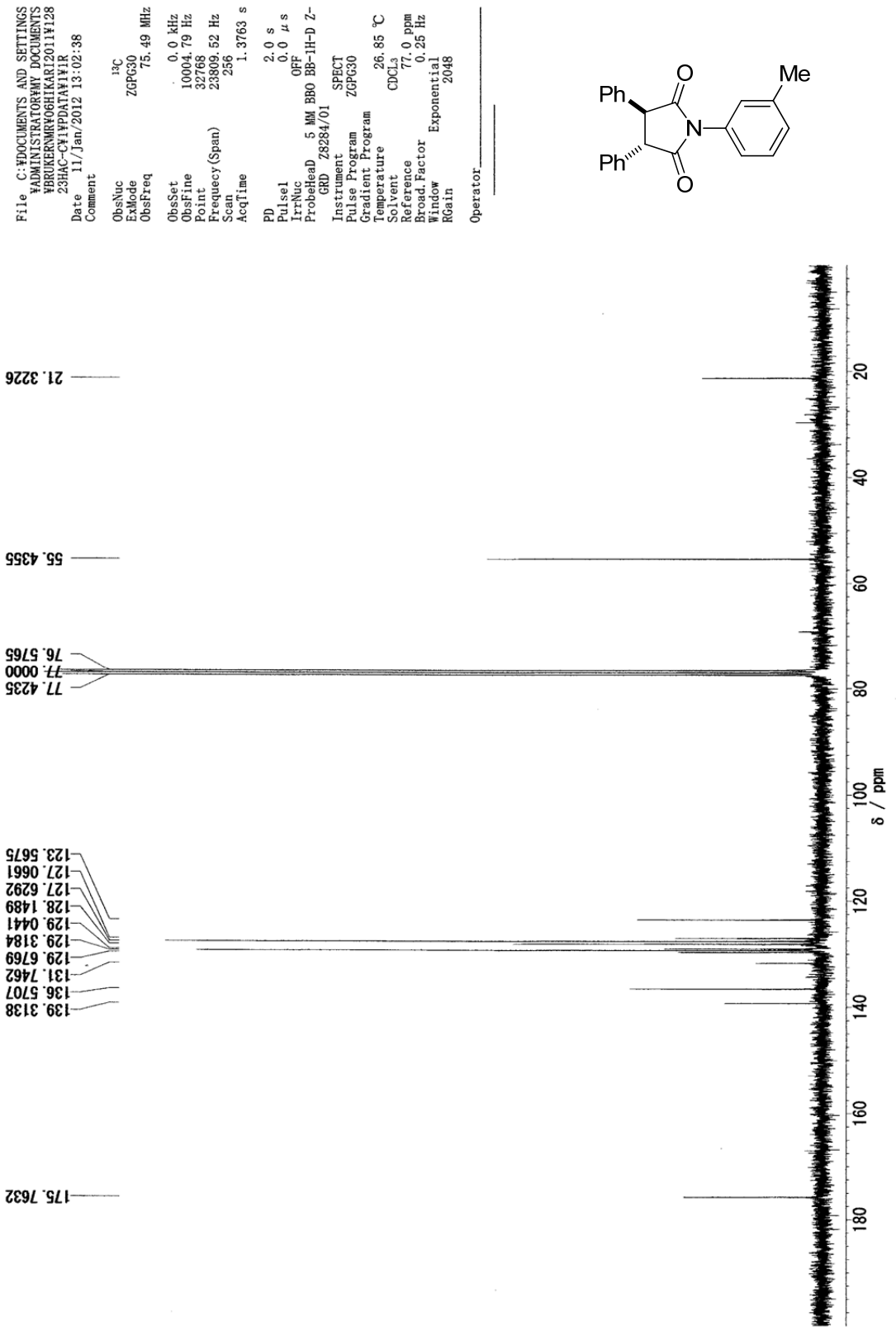

Figure S31. ^{1}H NMR spectrum for *trans*-*N*-(4-methylphenyl)-3,4-diphenylsuccinimide 3k

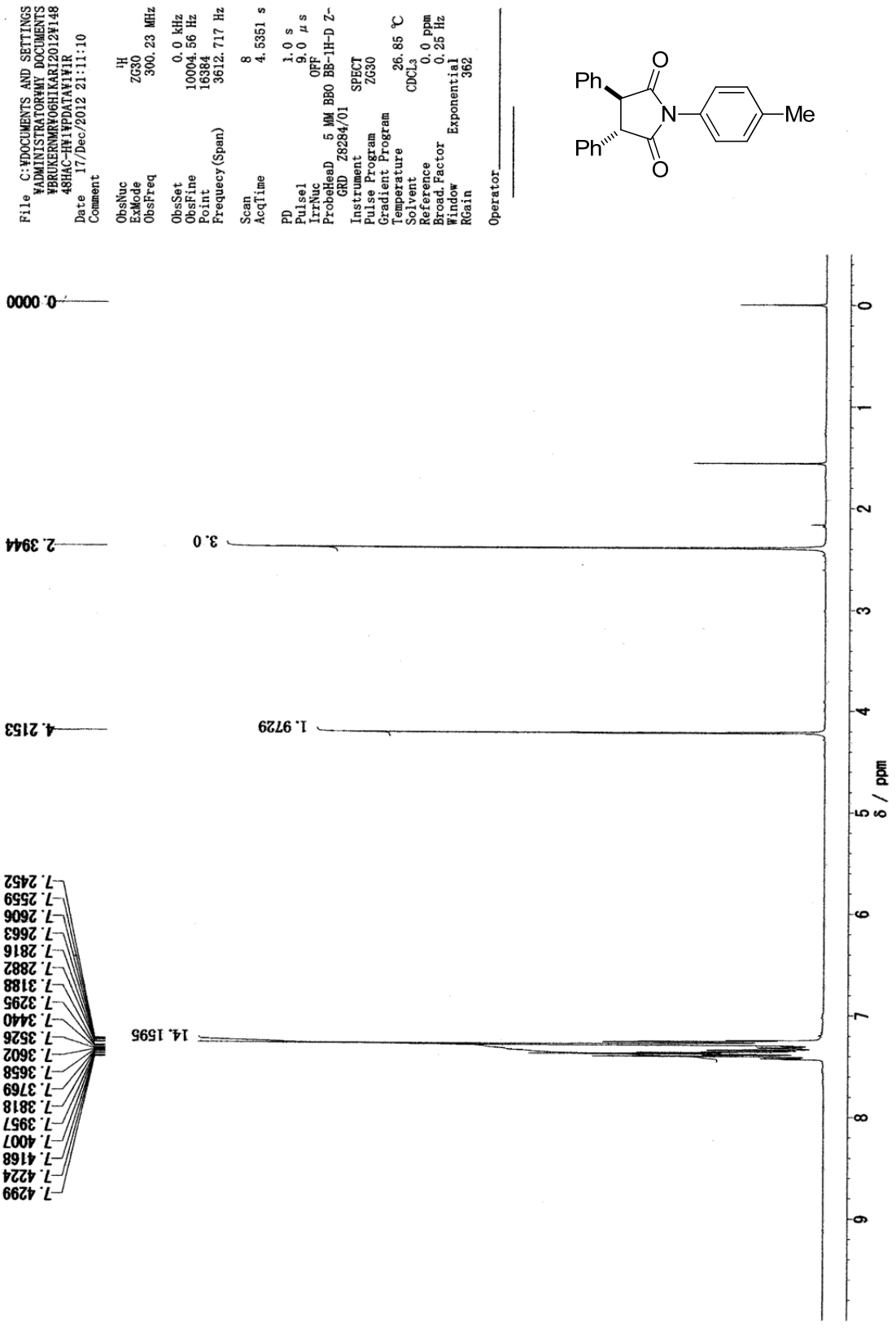

Figure S32. ^{13}C NMR spectrum for *trans*-*N*-(4-methylphenyl)-3,4-diphenylsuccinimide 3k

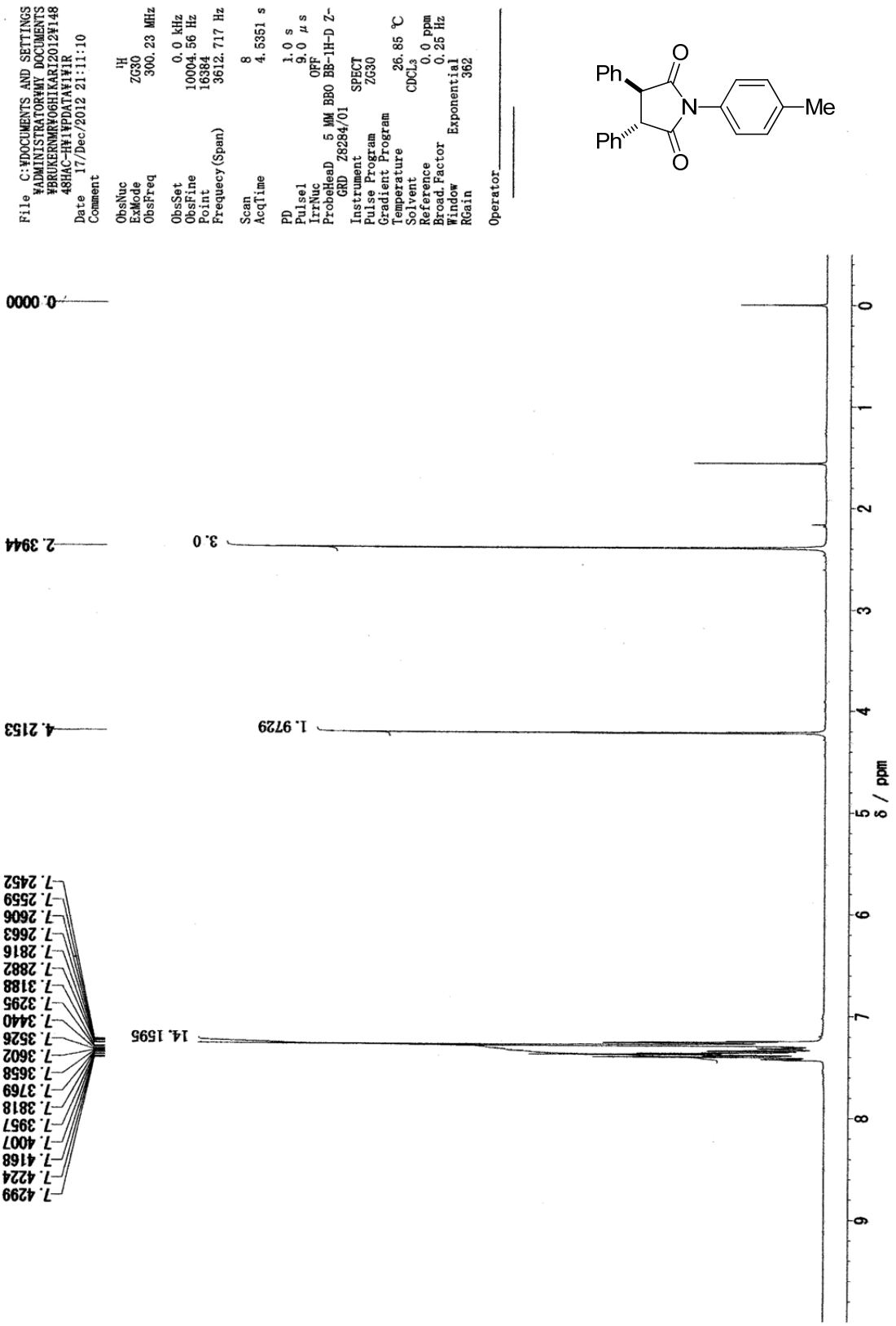

Figure S33. HPLC analysis of **3d**, (a) racemic, (b) 90% ee. Ee value was determined using a chiral column (Daicel Ind. CHIRALPAK AD-H), flow rate 0.7 mL/min, solvent, Hexane : EtOH = 95 : 5.

(a) (b)

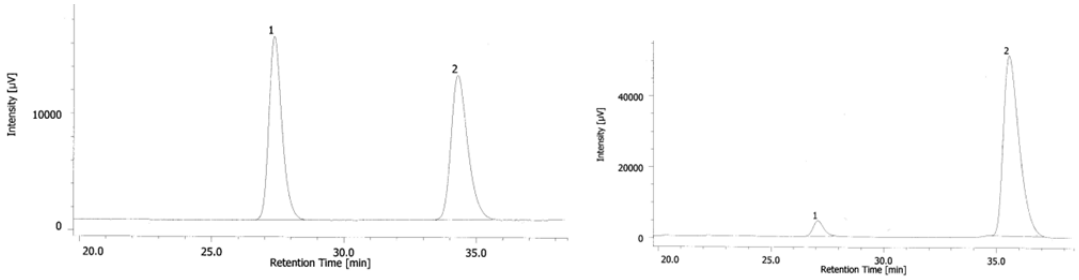

Figure S34. HPLC analysis of **3e**, (a) racemic, (b) 97% ee. Ee value was determined using a chiral column (Daicel Ind. CHIRALPAK IA-3), flow rate 0.5 mL/min, solvent, Hexane : EtOH = 98 : 2.

(a) (b)

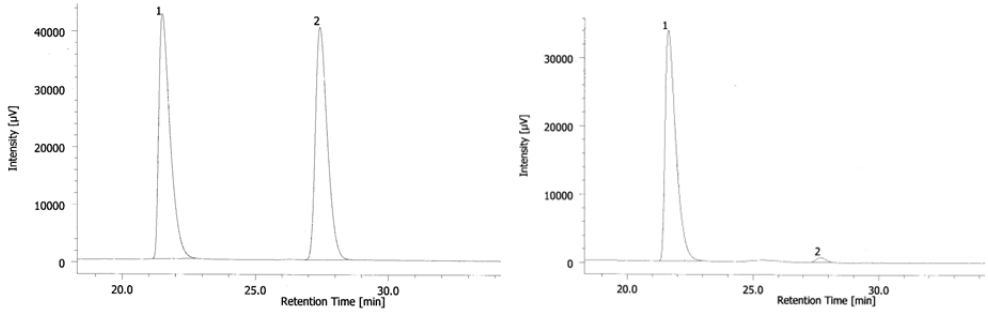

Figure S35. HPLC analysis of **3i**, (a) racemic, (b) 98% ee. Ee value was determined using a chiral column (Daicel Ind. CHIRALPAK IA), flow rate 0.7 mL/min, solvent, Hexane : EtOH = 90 : 10.

(b) (b)