**Silver (I) Catalyzed Novel Cascade Cyclization Reactions: Incorporation of Allenes into the Isochromenes**

Nitin T. Patil, Nirmal K. Pahadi and Yoshinori Yamamoto*

*Department of Chemistry, Graduate School of Science, Tohoku University,*

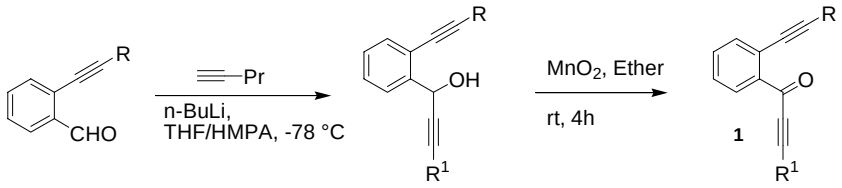
*Sendai 980-8578, Japan*

## Contents of Supporting Information (28 Pages)

**Preparation of starting materials (1a-e).**

The preparation of **1a** is representative. *n*BuLi (1.7 mL, 1.56M in hexanes, 2.669 mmol) was added drop wise to a solution of 1-pentyne (0.26 mL, 2.669 mmol) in THF (15 mL) at –78 °C. The resulting solution was stirred for 10 min at –78 °C and then a solution of *ortho*-alkynylaldehyde (0.5 g, 2.427 mmol) in THF (5 mL) was added. The resulting mixture was warmed to room temperature and stirred for 12 h. Aqueous $NH_4Cl$ was added, the organic layer was separated and the aqueous layer was extracted with $Et_2O$. The combined organic layers were dried ($Na_2SO_4$), filtered, and concentrated under reduced pressure. The residue thus obtained was used directly for further reaction.

$MnO_2$ (400 mol%) was added in portions to a solution of above alcohol (approx. 5 mmol) in $Et_2O$ (20 mL). The resulting suspension was stirred at room temperature for 4h, filtered through celite, and concentrated under reduced pressure. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography to yield the corresponding alkynones **1a** (95%). This procedure was used for the preparation of all substrates **1b-e** by using appropriate *ortho*-alkynylaldehydes and terminal alkynes.




**(1a)**. Yield = 95%; $^1$H NMR ($CDCl_3$, 400 MHz) δ 8.10 (d, *J* = 7.6 Hz, 1H), 7.63-7.56 (m, 3H), 7.50 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.45 (dt, *J* = 7.6,1.6 Hz, 1H), 7.35-7.30 (m, 3H), 2.40 (t, *J* = 6.8 Hz, 2H), 1.62 (sextet, *J* = 6.8 Hz, 2H), 1.01 (t, *J* = 6.8 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 177.5, 138.3, 134.1, 132.1, 131.8, 131.6, 128.5, 128.2, 127.7, 123.2, 122.8, 96.8, 95.0, 88.3, 80.8, 21.4, 21.3, 13.7; HRMS calcd for $C_{20}H_{16}O$ ($M^+$) 272.1201, found 295.1203 (M+Na).

**(1b)**. Yield = 92%; $^1$H NMR ($CDCl_3$, 400 MHz) δ 8.09 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.50-7.46 (m, 3H), 7.38 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.13 (d, *J* = 7.8 Hz, 2H), 2.40 (t, *J* = 7.0 Hz, 2H), 2.35 (s, 3H), 1.62 (sextet, *J* = 7.0 Hz, 2H), 1.01 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 177.5, 138.7, 138.1, 134.0, 132.1, 131.7, 131.6, 128.9, 127.5, 122.9, 120.1,

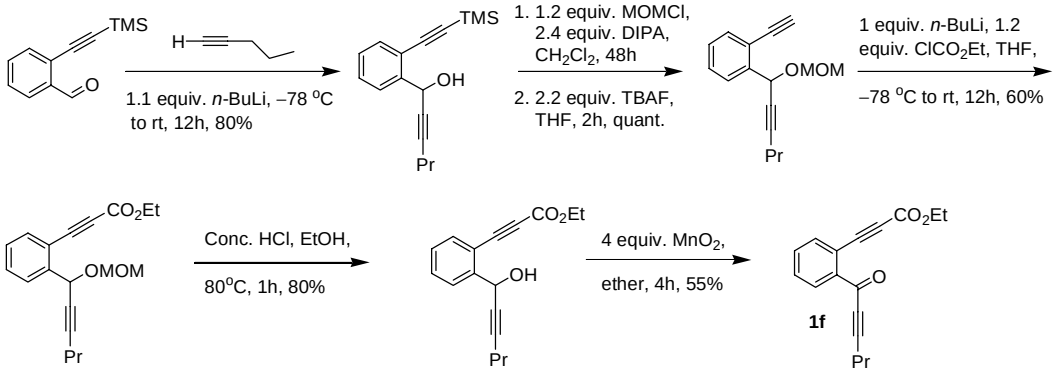
96.7, 95.4, 87.7, 80.9, 21.6, 21.3, 21.3, 13.6; HRMS calcd for $C_{21}H_{18}O$ ($M^+$) 286.1358, found 309.1250 (M+Na).

**(1c)**. Yield = 94%; $^1$H NMR ($CDCl_3$, 400 MHz) δ 8.15 (dt, 7.6, 1.0 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 2H), 7.63 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.52 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.44 (dt, *J* = 7.8, 1.2 Hz, 1H), 2.42 (t, *J* = 7.0 Hz, 2H), 1.64 (sextet, *J* = 7.0 Hz, 2H), 1.03 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 177.2, 138.3, 134.3, 132.3, 131.9, 130.2, 128.3, 127.0, 125.2, 125.0, 122.5, 121.9, 96.9, 93.1, 90.6, 80.6, 21.4, 21.3, 13.6; HRMS calcd for $C_{21}H_{15}F_3O$ ($M^+$) 340.1075, found 363.0967 (M+Na).

**(1d)**.[1] Yield = 94%; $^1$H NMR ($CDCl_3$, 400 MHz) δ 7.96 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.41 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.34 (dt, *J* = 7.8, 1.0 Hz, 1H), 7.25 (dt, *J* = 7.8, 1.0 Hz, 1H), 2.39-2.33 (2q, *J* = 7.0 Hz, 4H), 1.63-1.54 (2 sextet, *J* = 7.0 Hz, 4H), 0.99 (t, *J* = 7.0 Hz, 3H), 0.97 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 177.7, 138.3, 134.4, 131.9, 131.4, 126.9, 123.5, 96.6, 96.3, 80.8, 79.3, 22.1, 21.9, 21.4, 21.3, 13.6; HRMS calcd for $C_{17}H_{18}O$ ($M^+$) 238.1358, found 261.1250 (M+Na).

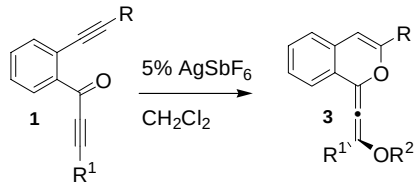
**(1e)**.[2] Yield = 93%, $^1$H NMR ($CDCl_3$, 400 MHz) δ 8.19 (d, *J* = 7.6 Hz, 1H), 7.68 (d, *J* = 7.6 Hz, 1H), 7.62-7.26 (m, 12H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 177.3, 138.2, 134.2, 132.9, 132.4, 131.8, 131.4, 130.6, 128.5, 128.4, 128.1, 127.9, 123.0, 122.9, 120.0, 95.4, 93.3, 88.0; HRMS calcd for $C_{23}H_{14}O$ ($M^+$) 306.1045, found 329.0937 (M+Na).

**Preparation of Substrate 1f.**



**(1f)**. $^{1}$H NMR ($CDCl_3$, 400 MHz) δ 8.12 (m, 1H), 7.66 (m, 1H), 7.55-7.49 (m, 2H), 4.27 (q, *J* = 7.6 Hz, 2H), 2.45 (t, *J* = 6.8 Hz. 2H), 1.70-1.61 (m, 2H), 1.33 (t, *J* = 7.6 Hz, 3H), 1.03 (t, *J* = 7.6 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 176.3, 153.8, 139.2, 135.5, 132.3, 131.7, 129.9, 119.2, 98.0, 85.2, 84.2, 80.3, 62.1, 21.3, 14.1, 13.6; HRMS calcd for $C_{17}H_{16}O_3$ ($M^+$) 268.1099, found 291.0992 (M+Na).

**General Procedure for Ag(I) catalyzed cyclization.**




The preparation of **3a** is representative. **1a** (0.050 g, 0.1838 mmol), MeOH (0.012 g, 0.3676 mmol), $AgSbF_6$ (5 mol%), and $CH_2Cl_2$ (2 mL) were stirred at 35 °C in a screw capped vial for 16 h. Water (10 mL) was added and the resulting reaction mixture was extracted with ethyl acetate. The extracts were concentrated to give a thick residue. The resulting residue was purified by column chromatography with use of silica gel as a solid phase and 9:1 hexane-ethyl acetate as an eluent to afford pure **3a** (0.053 g, 95%).

**(3a).** $^{1}$H NMR ($CDCl_3$, 400 MHz ) δ 8.16 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.34-7.20 (m, 5H), 6.63 (s, 1H), 3.94 (s, 3H), 2.45 (t, *J* = 7.3 Hz, 2H), 1.53 (sextet, *J* = 7.3 Hz, 2H), 0.76 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 200.0 (=C=), 155.8, 138.3, 138.0, 133.4, 131.8, 129.7, 128.5, 128.2, 127.0, 124.8, 124.7, 123.9, 121.9, 105.0, 55.5, 36.4, 24.5, 14.1; HRMS calcd for $C_{21}H_{20}O_2$ ($M^+$) 304.1463, found 327.1356 (M+Na).

**(3b).** $^{1}$H NMR ($CDCl_3$, 400 MHz ) δ 8.31 (d, *J* = 8.3 H, 1H), 7.83 (d, *J* = 8.3 Hz, 2H), 7.54 (t, *J* = 8.3 Hz, 1H), 7.44-7.32 (m, 5H), 6.74 (s, 1H), 4.20 (t, *J* = 6.4 Hz, 2H), 2.55 (t, *J* = 7.6 Hz, 2H), 1.98-1.91 (m, 2H), 1.69-1.59 (m, 4H), 1.06 (t, *J* = 7.3 Hz, 3H), 0.87 (t, *J* = 7.2 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 200.1 (=C=), 155.3, 138.1, 133.3, 131.8, 129.6, 128.5, 127.9, 126.9, 124.8,

124.5, 123.9, 122.0, 105.8, 67.9, 36.4, 31.4, 24.5, 19.5, 14.1, 13.9; HRMS calcd for $C_{24}H_{26}O_2$ ($M^+$) 346.1933, found 369.1825 (M+Na).

**(3c).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.19 (d, *J* = 8.6 Hz, 1H), 7.71 (d, *J* = 7.3 Hz, 2H), 7.44-7.19 (m, 6H), 5.76 (dddd, *J* = 17.0, 13.4, 10.2, 6.8 Hz, 1H), 4.99-4.88 (m, 2H), 4.08 (t, *J* = 6.3 Hz, 1H), 2.43 (t, *J* = 7.56 Hz, 2H), 2.09 (q, *J* = 7.0 Hz, 2H), 1.90-1.83 (m, 2H), 1.64-1.47 (m, 4H), 0.75 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 200.1 (=C=), 155.2, 138.4, 138.3, 138.0, 133.3, 131.8, 129.6, 128.5, 127.9, 126.9, 124.8, 124.6, 123.9, 122.0, 114.7, 105.8, 67.9, 36.4, 33.5, 28.7, 25.5, 24.5, 14.1; HRMS calcd for $C_{26}H_{28}O_2$ ($M^+$) 372.2089, found 395.1982 (M+Na).

**(3d).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.23 (d, *J* = 8.3 Hz, 1H), 7.79 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.42-7.23 (m, 10 H), 6.92 (s, 1H), 5.10 (d, *J* = 9.5 Hz, 2H), 2.53 (t, *J* = 7.3 Hz, 2H), 1.65-1.55 (sextet, *J* = 7.3 Hz, 2H), 0.81 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 199.9 (=C=), 153.8, 138.2, 137.7, 133.4, 131.9, 131.7, 129.6, 128.9, 128.6, 128.5, 128.2, 127.0, 124.8, 123.9, 122.0, 106.9, 87.6, 83.6, 56.9, 36.2, 24.4, 14.1; HRMS calcd for $C_{29}H_{24}O_2$ ($M^+$) 404.1776, found 427.1669 (M+Na).

**(3e).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.20 (d, *J* = 8.3 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.45 (t, J = 7.8 Hz, 1H), 7.33-7.20 (m, 5H), 6.65 (s, 1H), 4.73 (hept, *J* = 6.0 Hz, 1H), 2.44 (t, *J* = 7.6 Hz, 2H), 1.52 (sextet, *J* = 7.6 Hz, 2H), 1.40 (d, *J* = 6.0 Hz, 6H), 0.77 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 200.2 (=C=), 154.0, 138.4, 138.0, 133.4, 132.0, 129.7, 128.5, 128.2, 127.9, 126.9, 124.8, 124.7, 124.5, 122.3, 107.4, 70.3, 36.4, 24.6, 22.1, 14.0; HRMS calcd for $C_{23}H_{24}O_2$ ($M^+$) 332.1776, found 355.1669 (M+Na).

**(3g).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.38 (d, *J* = 8.2 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 2H), 7.55 (t, *J* = 7.8 Hz, 1H), 7.47-7.33 (m, 10 H), 6.84 (s, 1H), 5.32 (s, 2H), 2.56 (t, *J* = 7.0 Hz, 2H), 1.67 (m, 2H), 0.85 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 200.0 (=C=), 154.8, 138.3, 137.9, 136.7, 133.4, 131.9, 129.7, 128.5, 127.9, 127.3, 127.0, 124.9, 124.8, 124.0, 122.2, 106.5, 70.2, 36.3, 24.4, 14.0; HRMS calcd for $C_{27}H_{24}O_2$ ($M^+$) 380.1776, found 403.1669 (M+Na).

**(3h).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.29 (d, *J* = 8.2 Hz, 1H), 7.74 (d, *J* = 7.8 Hz, 2H), 7.46-7.31 (m, 3H), 7.21 (d, *J* = 8.2 Hz, 2H), 6.75 (s, 1H), 4.04 (s, 3H), 2.57 (t, *J* = 7.8 Hz, 2H), 2.40 (s, 3H),

1.66 (sextet, *J* = 7.8 Hz, 2H), 0.89 (t, *J* = 7.8 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 199.7 (=C=), 155.7, 144.3, 137.8, 135.9, 131.8, 129.8, 129.3, 128.5, 126.9, 124.9, 124.6, 123.8, 121.9, 105.1, 55.5, 36.4, 24.5, 21.8, 14.2; HRMS calcd for $C_{22}H_{22}O_2$ ($M^+$) 318.1620, found 341.1512 (M+Na).

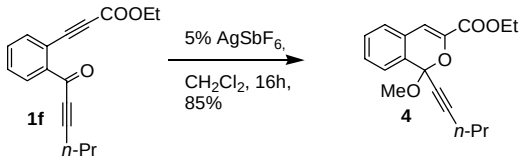
**(3i).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.32 (d, *J* = 8.3 Hz, 1H), 7.76 (m, *J* = 7.3 Hz, 2H), 7.46-7.32 (m, 3H), 7.22 (d, *J* = 8.0 Hz, 2H), 6.79 (s, 1H), 4.86 (hept, *J* = 6.0 Hz, 1H), 2.58 (t, *J* = 7.0 Hz, 2H), 2.42 (s, 3H), 1.66 (sextet, *J* = 7.0 Hz, 2H), 1.52 (d, *J* = 6.0 Hz, 6H), 0.90 (t, *J* = 7.0 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 199.8 (=C=), 153.9, 144.3, 137.8, 136.0, 132.0, 129.9, 129.4, 129.2, 128.8, 128.1, 126.8, 124.9, 124.7, 124.4, 122.3, 107.4, 70.3, 36.3, 24.6, 22.1, 21.9, 21.8, 14.1; HRMS calcd for $C_{24}H_{26}O_2$ ($M^+$) 346.1933, found 369.1825 (M+Na).

**(3j).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.18 (d, *J* = 8.3 Hz, 1H), 7.82 (d, *J* = 8.3 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.34-7.23 (m, 3H); 6.64 (s, 1H), 3.96 (s, 3H), 2.43 (t, *J* = 7.8 Hz, 2H), 1.53 (sextet, *J* = 7.8 Hz, 2H), 0.77 (t, *J* = 7.8 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 198.9 (=C=), 156.2, 140.9, 138.6, 134.3, 129.9, 127.3, 125.7, 125.7, 125.6, 124.9, 124.6, 123.9, 122.2, 105.1, 55.6, 36.4, 24.6, 14.1; HRMS calcd for $C_{22}H_{19}F_3O_2$ ($M^+$) 372.1337, found 395.1229 (M+Na).

**(3k).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.21 (dd, *J* = 8.3, 1.2 Hz, 1H), 7.83 (d, *J* = 8.3 Hz, 2H), 7.56 (d, *J* = 7.3 Hz, 2H), 7.32-7.23 (m, 3H), 6.66 (s, 1H), 4.74 (hept, *J* = 6.0 Hz, 1H), 2.43 (t, *J* = 7.6 Hz, 2H), 1.51 (sextet, *J* = 7.6 Hz, 2H), 1.40 (d, *J* = 6.0 Hz, 6H), 0.77 (t, *J* = 7.6 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 199.0 (=C=), 154.4, 141.0, 138.6, 134.6, 134.3, 131.8, 129.9, 127.2, 126.9, 125.7, 125.6, 124.7, 124.5, 122.2, 107.3, 70.3, 36.4, 24.7, 22.1, 14.1; HRMS calcd for $C_{24}H_{23}F_3O_2$ ($M^+$) 372.1337, found 395.1229 (M+Na).

**(3l).** $^1$H NMR ($CDCl_3$, 400 MHz ) δ 8.14 (d, J = 8.2 Hz, 1H), 7.43-7.31 (m, 3H), 6.54 (s, 1H), 3.90 (s, 3H), 2.75 (t, *J* = 7.3 Hz, 2H), 2.51 (t, *J* = 7.3 Hz, 2H), 1.76-1.59 (m, 4H), 0.95 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 210.2 (=C=), 155.5, 135.9, 131.3, 130.5, 127.1, 124.7, 123.9, 123.8, 122.1, 104.9, 55.5, 48.2, 36.1, 24.9, 17.3, 14.2, 13.9; HRMS calcd for $C_{18}H_{22}O_2$ ($M^+$) 270.1620, found 293.1512 (M+Na).

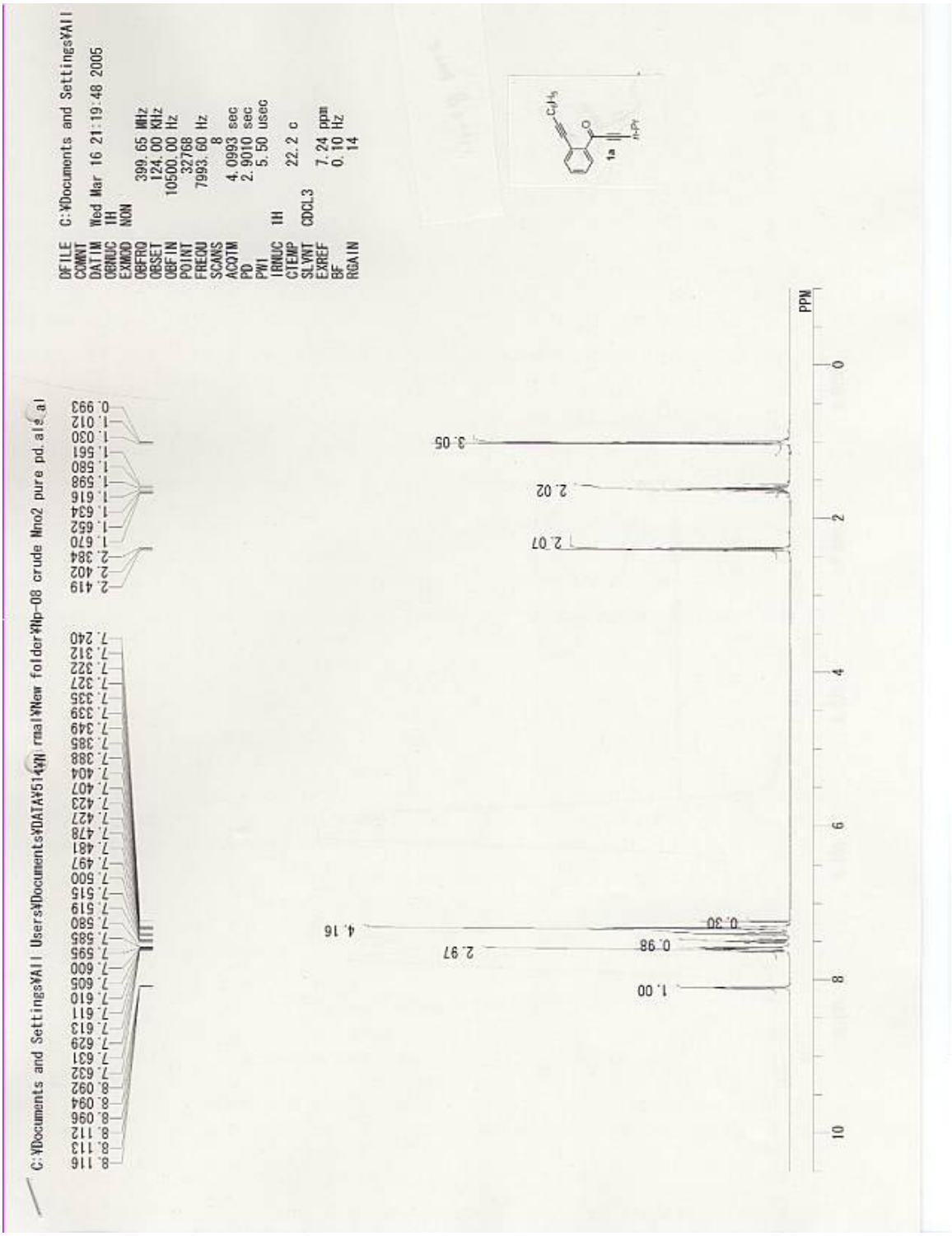
**(3m).** $^{1}$H NMR ($CDCl_3$, 400 MHz ) δ 8.38 (d, *J* = 8.3 Hz, 1H), 7.75 (d, *J* = 8.3 Hz, 1H), 7.63 (d, J = 8.0 Hz, 2H), 7.55-7.18 (m, 10H), 6.89 (s, 1H), 4.1 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 199.5 (=C=), 155.9, 140.7, 138.5, 138.4, 132.8, 131.9, 129.5, 129.3, 128.3, 128.1, 128.0, 127.0, 127.3, 125.5, 125.2, 124.5, 122.1, 105.5, 55.6; HRMS calcd for $C_{24}H_{18}O_2$ ($M^+$) 338.1307, found 361.1199 (M+Na).




**(4).** $^{1}$H NMR ($CDCl_3$, 400 MHz) δ 7.52-7.43 (m, 4H), 7.24 (s, 1H), 4.21 (m, 2H), 3.57 (s, 3H), 2.24 (t, *J* = 6.4 Hz, 2H), 1.57-1.51 (m, 2H), 1.30 (t, *J* = 6.4 Hz, 3H), 0.96 (t, *J* = 7.2 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ 165.4 (C=O/O-C-O), 162.9 (C=O/O-C-O), 141.9, 132.8, 131.7, 130.2, 122.7, 121.0, 106.6, 90.0, 88.4, 74.2, 59.7, 53.1, 21.6, 20.8, 14.5, 13.6; HRMS calcd for $C_{18}H_{20}O_4$ ($M^+$) 300.1362, found 323.1361 (M+Na).

**References:**


1. Pena, D.; Perez, D.; Guitian, E.; Castedo, L. *Eur. J. Org. Chem.* **2003**, 1238-1243.
2. Rodriguez, D.; Navarro, A.; Castedo, L.; Dominguez, D.; Saa, C. *Org. Lett.* **2003**, *2*, 1497-1500.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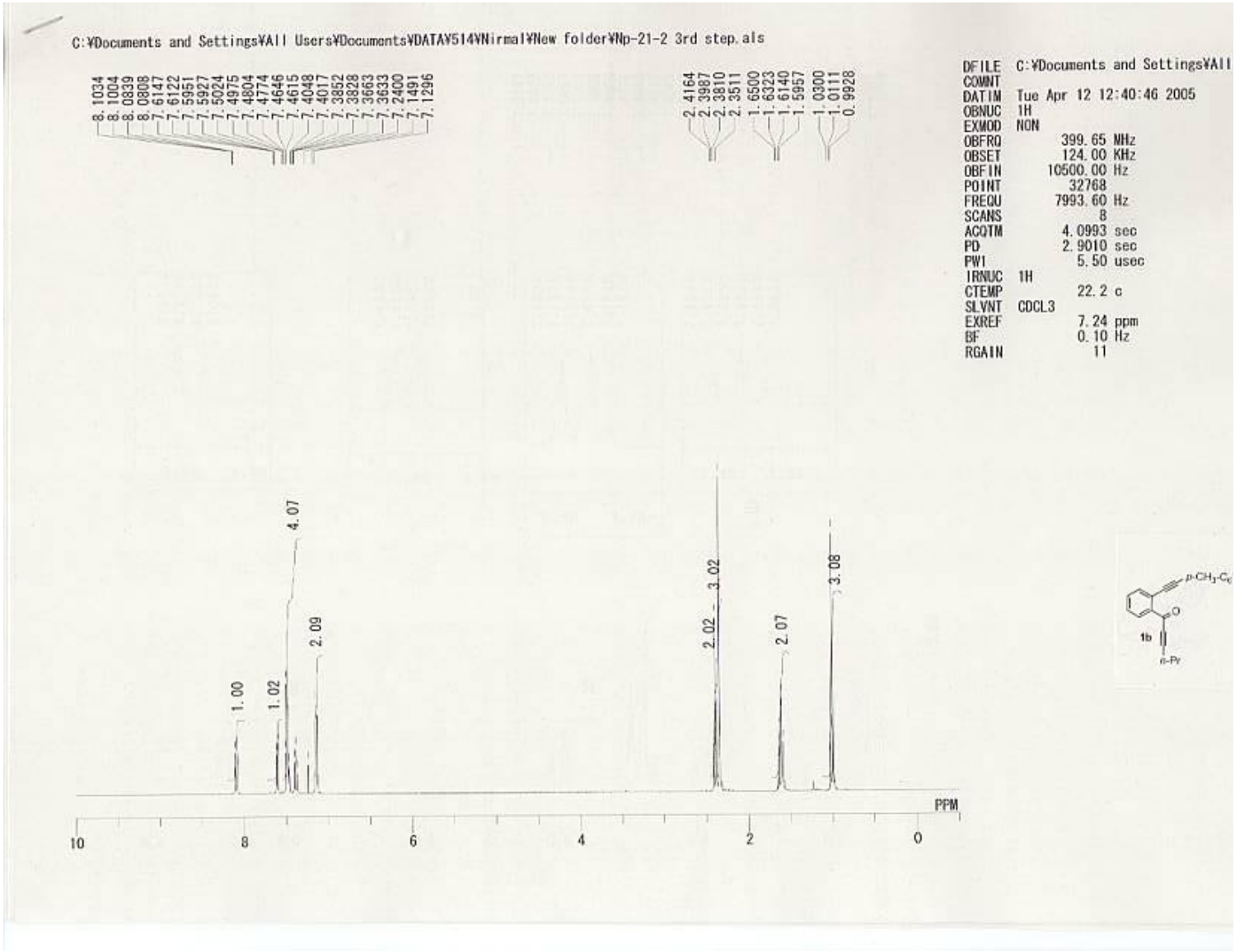
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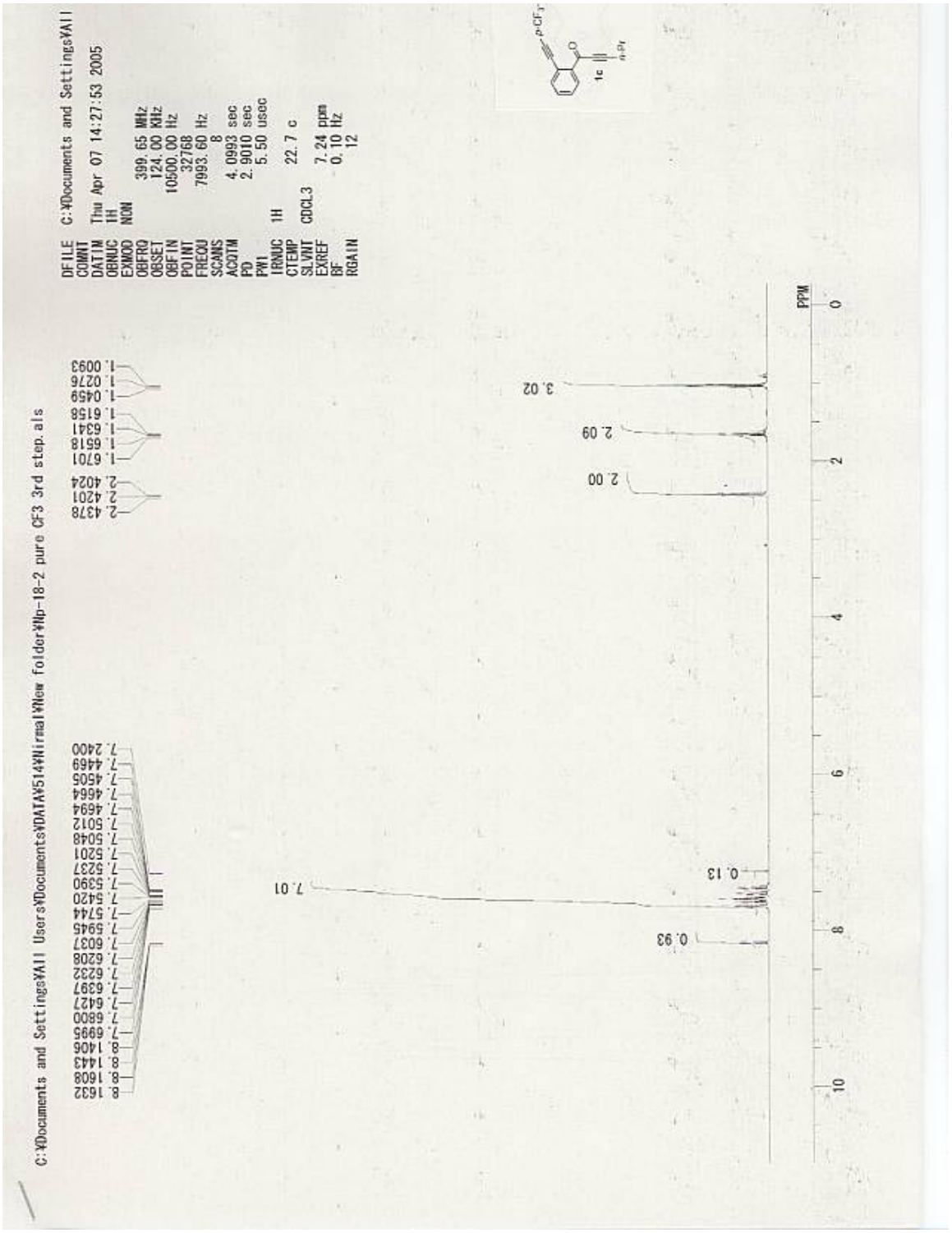
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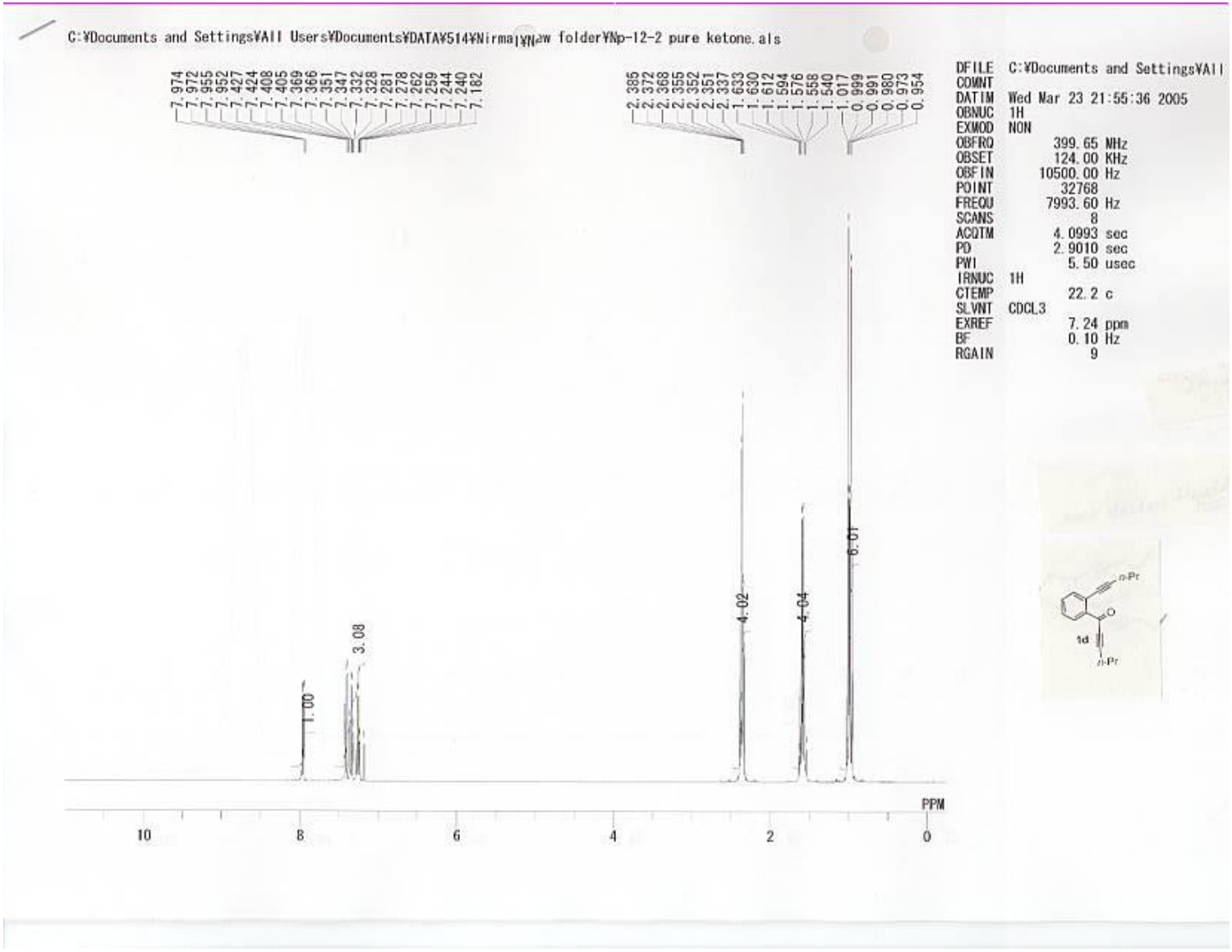
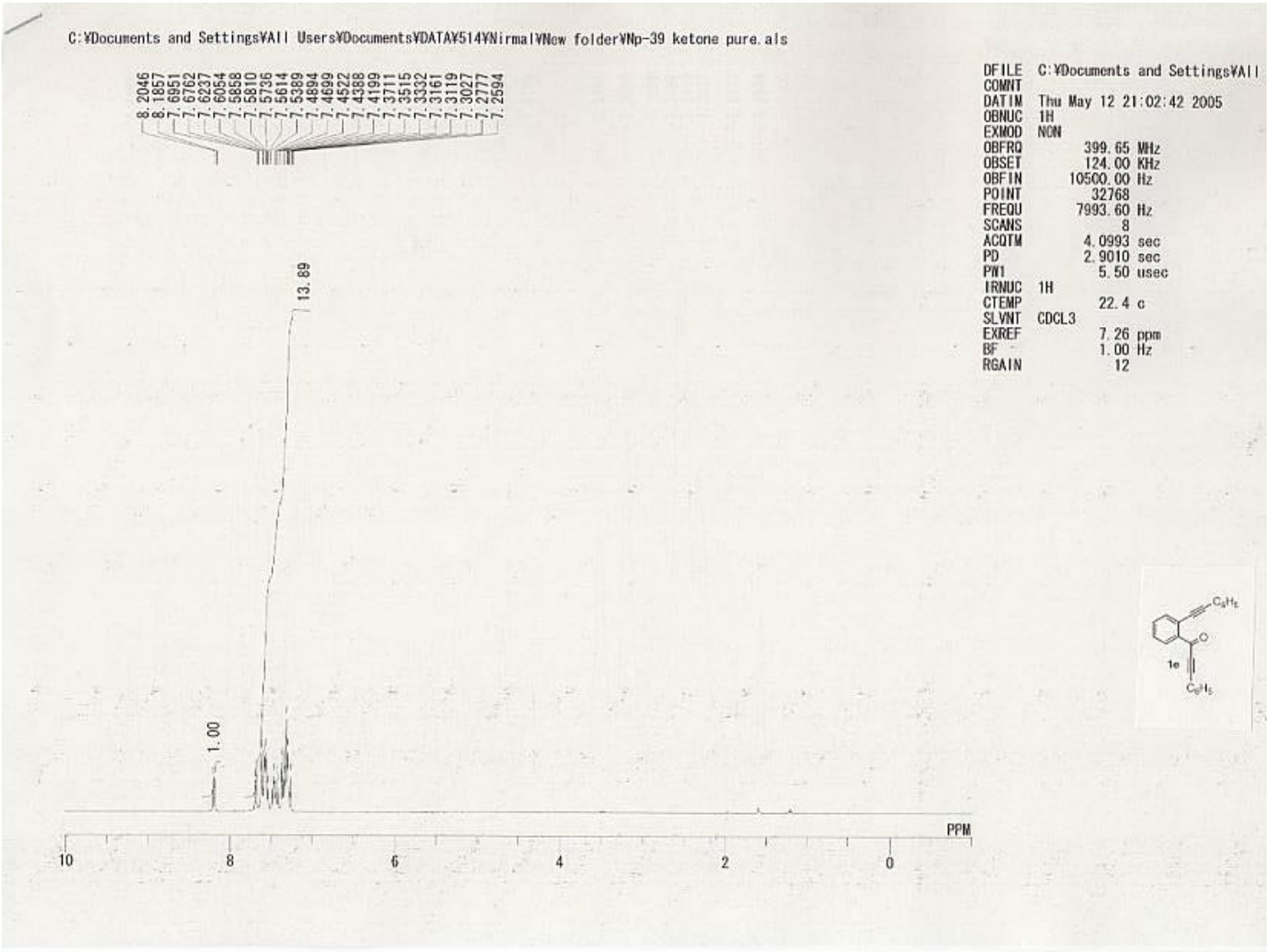
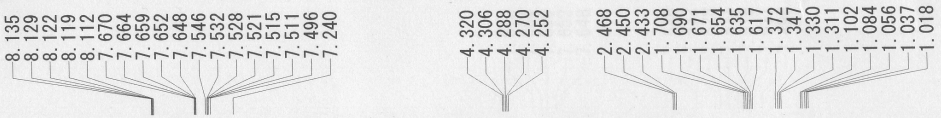
CTEMP 22.7 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 12
8.1632
8.1608
8.1443
8.1406
7.6995
7.6800
7.6427
7.6397
7.6232
7.6208
7.6037
7.5945
7.5744
7.5420
7.5390
7.5237
7.5201
7.5048
7.5012
7.4694
7.4664
7.4505
7.4469
7.2400
7.01
0.93
0.13
3.02
2.09
2.00
2.4378
2.4201
2.4024
1.6701
1.6618
1.6341
1.6158
1.0459
1.0276
1.0093
PPM
0
2
4
6
8
10
p-CF3
1c
n-Pr

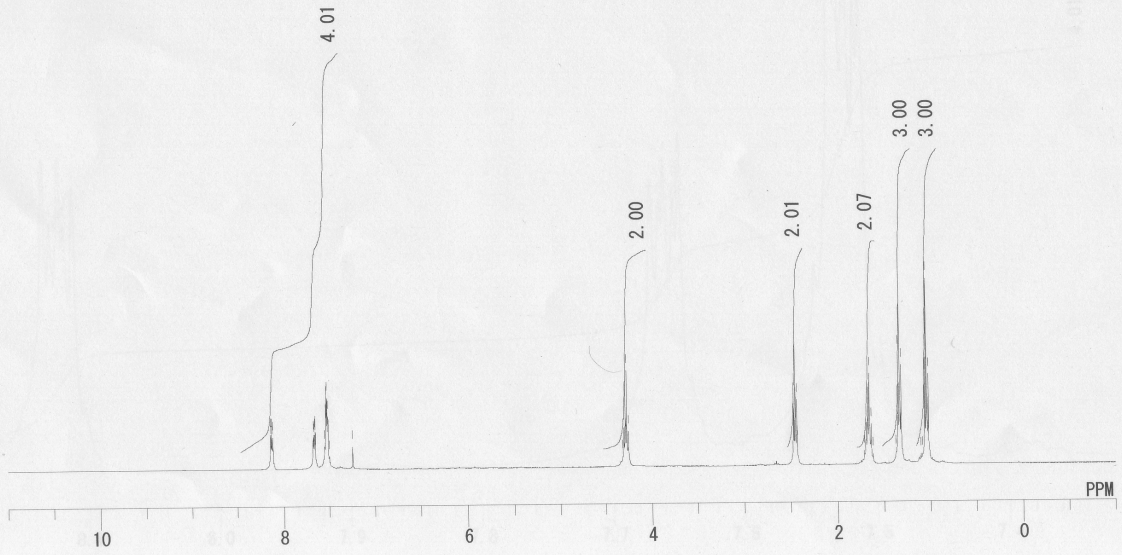

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirma¦¥New folder¥Np-12-2 pure ketone.als
7.974
7.972
7.955
7.952
7.427
7.424
7.408
7.405
7.369
7.366
7.351
7.347
7.332
7.328
7.281
7.278
7.262
7.259
7.244
7.240
7.182
2.385
2.372
2.368
2.355
2.352
2.351
2.337
1.633
1.630
1.612
1.594
1.576
1.558
1.540
1.017
0.999
0.991
0.980
0.973
0.954
DFILE C:¥Documents and Settings¥All
COMNT
DATIM Wed Mar 23 21:55:36 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 9
1.00
3.08
4.02
4.04
6.01
PPM
10
8
6
4
2
0
n-Pr
O
1d
n-Pr

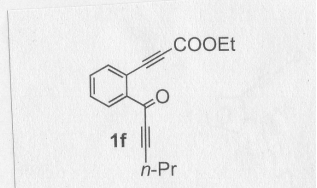

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-39 ketone pure.als
8.2046
8.1857
7.6951
7.6762
7.6237
7.6054
7.5858
7.5810
7.5736
7.5614
7.5389
7.4894
7.4699
7.4522
7.4388
7.4199
7.3711
7.3515
7.3332
7.3161
7.3119
7.3027
7.2777
7.2594
DFILE C:¥Documents and Settings¥All
COMNT
DATIM Thu May 12 21:02:42 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.00 Hz
RGAIN 12
13.89
1.00
PPM
10
8
6
4
2
0
1e

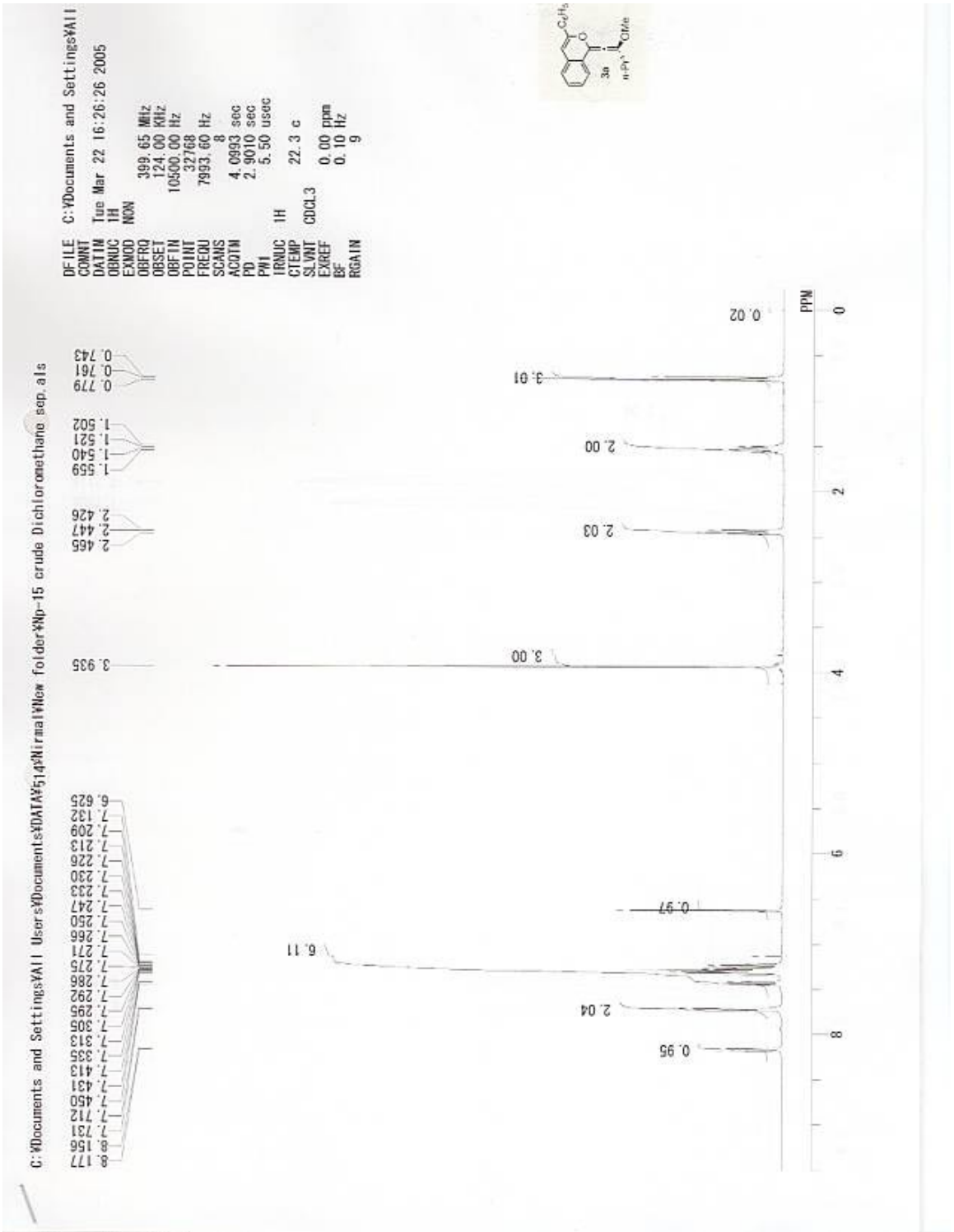
C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥Naya Kame¥Np-104 final pure pd.als

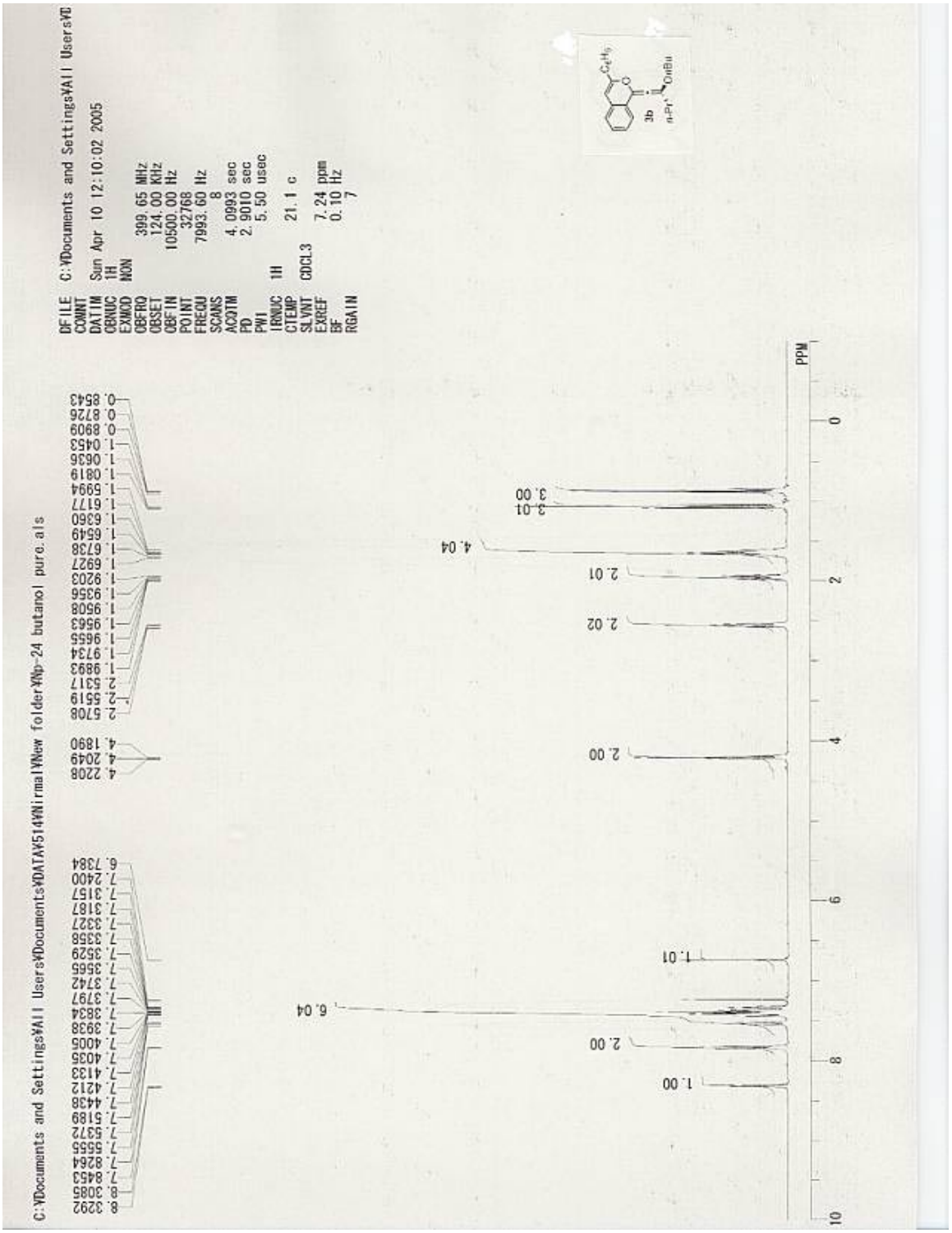


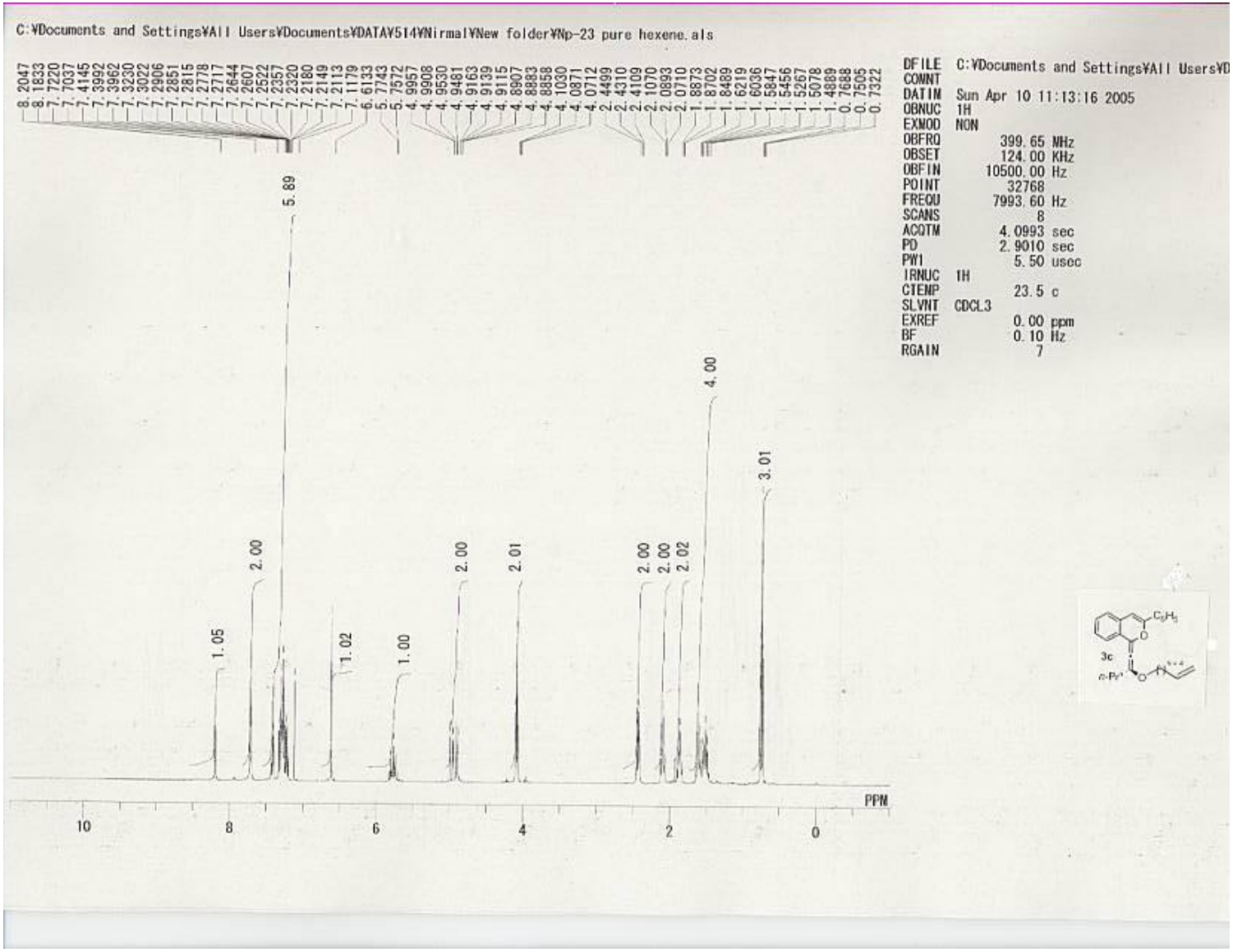

| | |
|---|---|
| DFILE | C:¥Documents and Settings¥All Users¥D |
| COMNT | |
| DATIM | Sat Oct 01 17:17:03 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 399.65 MHz |
| OBSET | 124.00 KHz |
| OBFIN | 10500.00 Hz |
| POINT | 32768 |
| FREQU | 7993.60 Hz |
| SCANS | 8 |
| ACQTM | 4.0993 sec |
| PD | 2.9010 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 24.7 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.20 Hz |
| RGAIN | 13 |

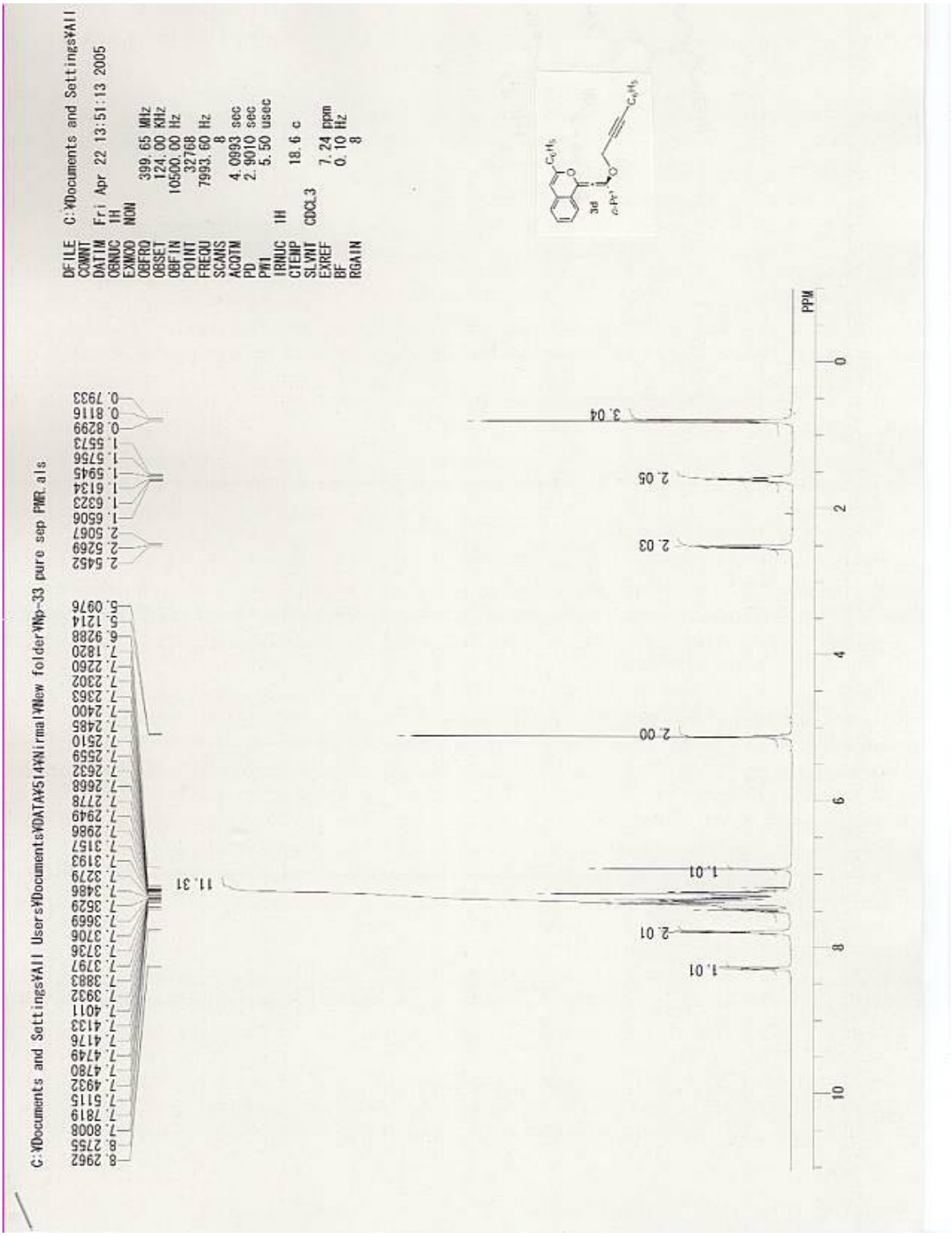


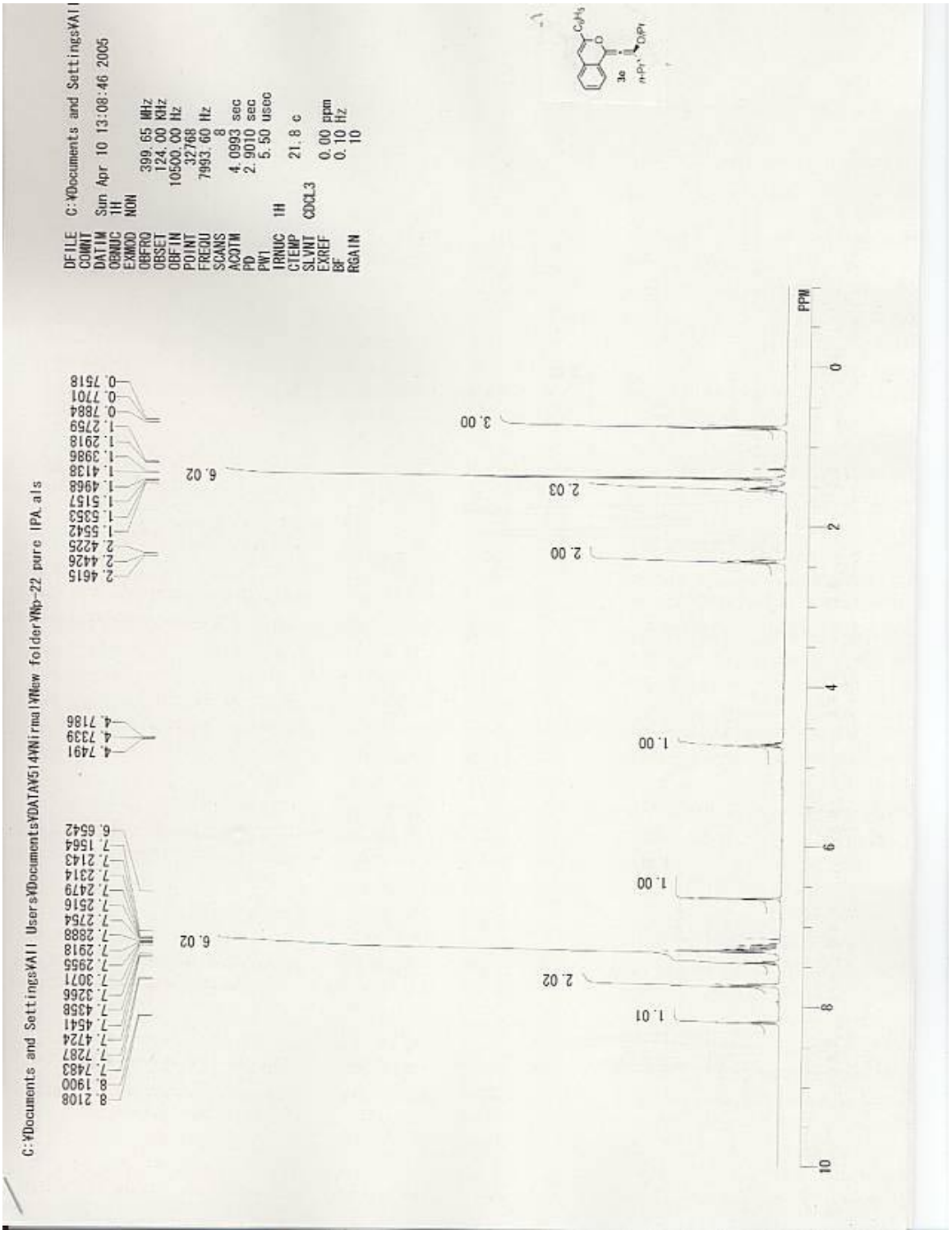



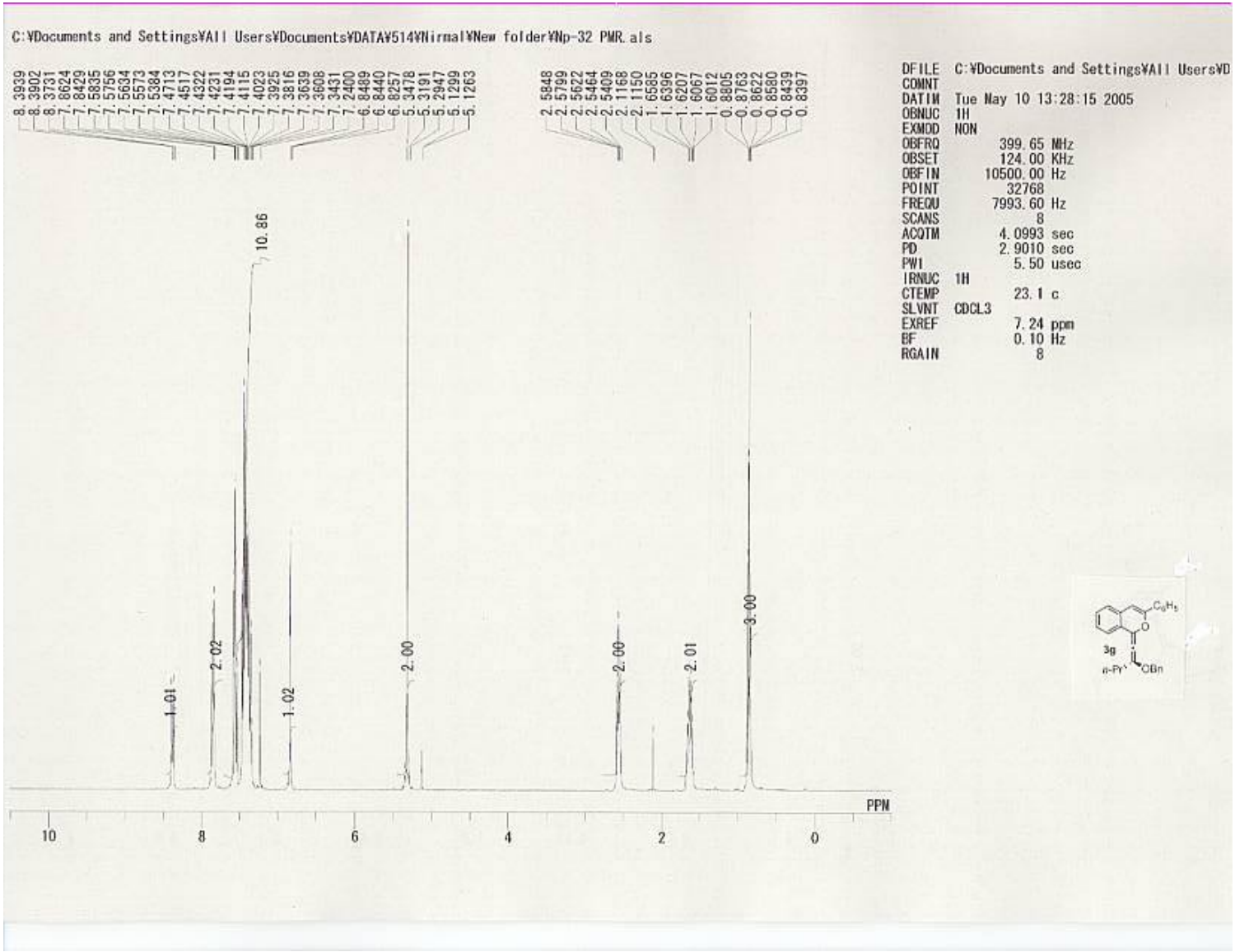

C:¥Documents and Settings¥All User s¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-15 crude Dichloromethane sep. als
DFILE C:¥Documents and Settings¥All
COMNT
DATIM Tue Mar 22 16:26:26 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 9
3a
n-Pr
Me
PPM
0.02
3.01
2.00
2.03
3.00
0.97
6.11
2.04
0.95
8.177
8.156
7.731
7.712
7.450
7.431
7.413
7.335
7.313
7.305
7.295
7.292
7.286
7.275
7.271
7.266
7.260
7.247
7.233
7.230
7.226
7.213
7.209
7.132
6.625
3.935
2.466
2.447
2.426
1.559
1.540
1.521
1.502
0.779
0.761
0.743

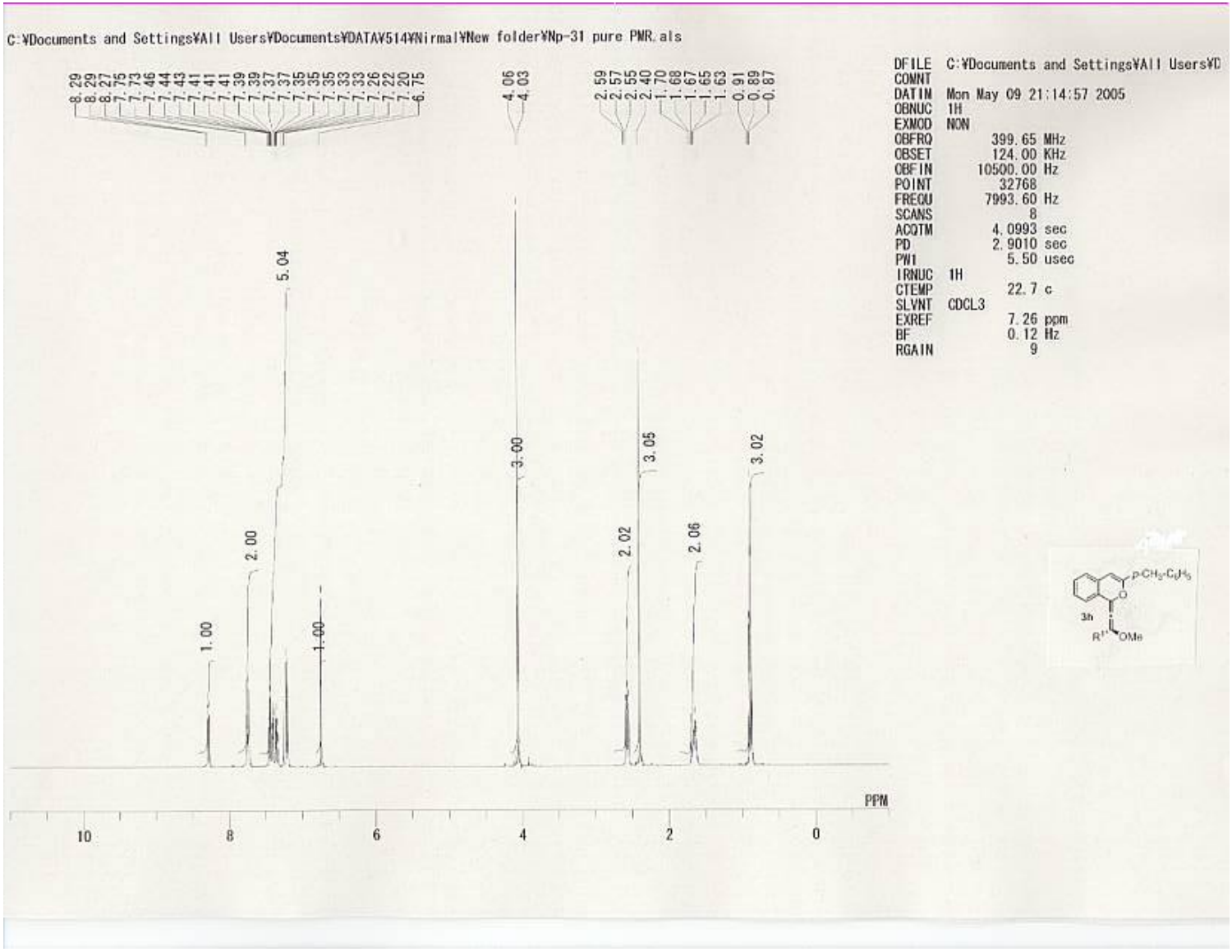

C:¥Documents and Settings¥All Users¥DATA¥514¥Nirmal¥New folder¥Np-24 butanol pure.als
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Sun Apr 10 12:10:02 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 7
8.3292
8.3085
7.8453
7.8264
7.5655
7.5372
7.5189
7.4438
7.4212
7.4133
7.4035
7.4005
7.3938
7.3834
7.3797
7.3742
7.3565
7.3529
7.3358
7.3327
7.3187
7.3157
7.2400
6.7384
4.2208
4.2049
4.1890
2.5708
2.5519
2.5317
1.9893
1.9734
1.9655
1.9563
1.9508
1.9356
1.9203
1.8927
1.6738
1.6549
1.6360
1.6177
1.5994
1.0819
1.0636
1.0453
0.8909
0.8726
0.8543
6.04
2.00
1.00
1.01
2.00
4.04
2.01
2.02
3.01
3.00
PPM
0
2
4
6
8
10
3b
n-Pr
OnBu

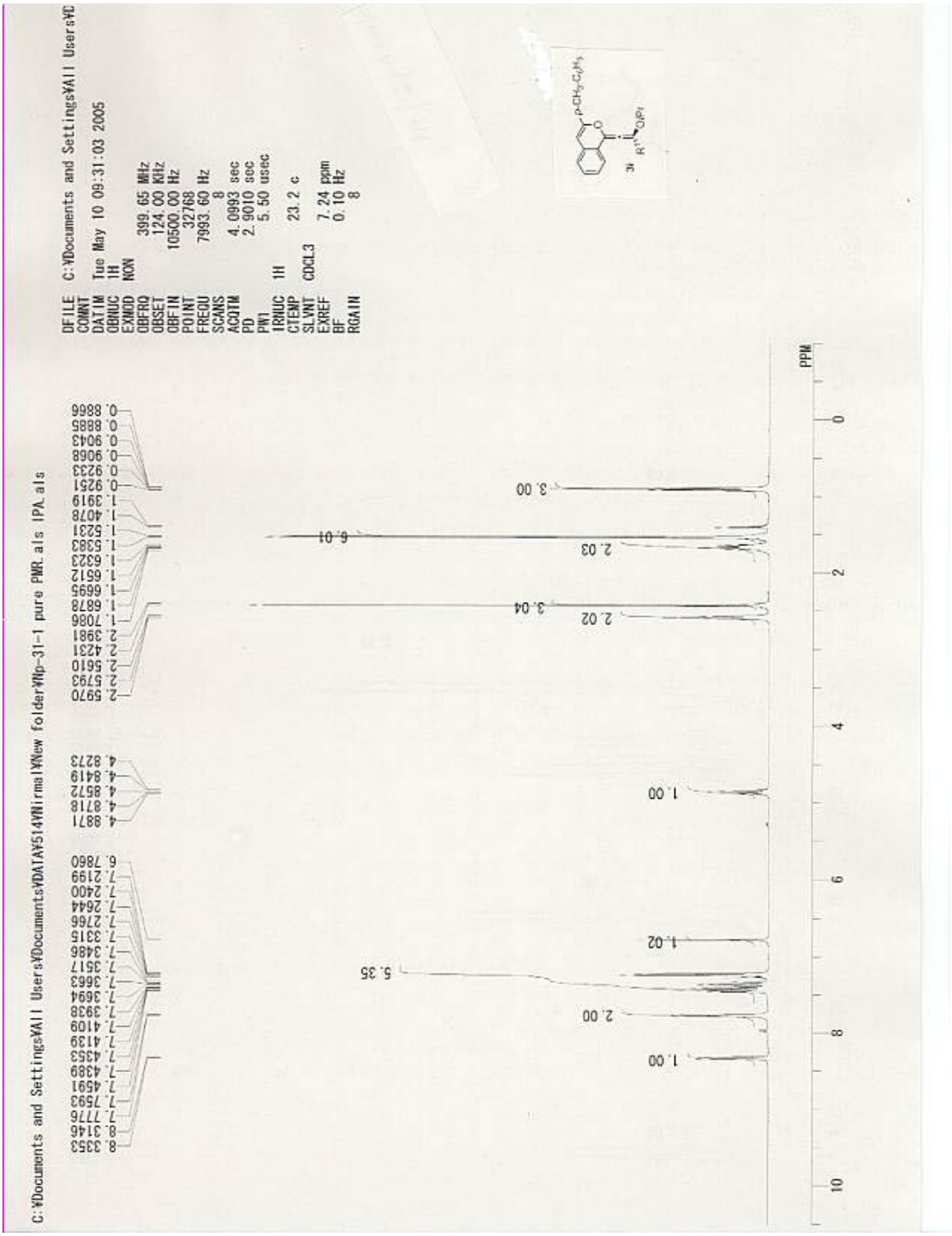

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-23 pure hexene.als
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Sun Apr 10 11:13:16 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 7
1.05
2.00
5.89
1.02
1.00
2.00
2.01
2.00
2.00
2.02
4.00
3.01
PPM
10
8
6
4
2
0

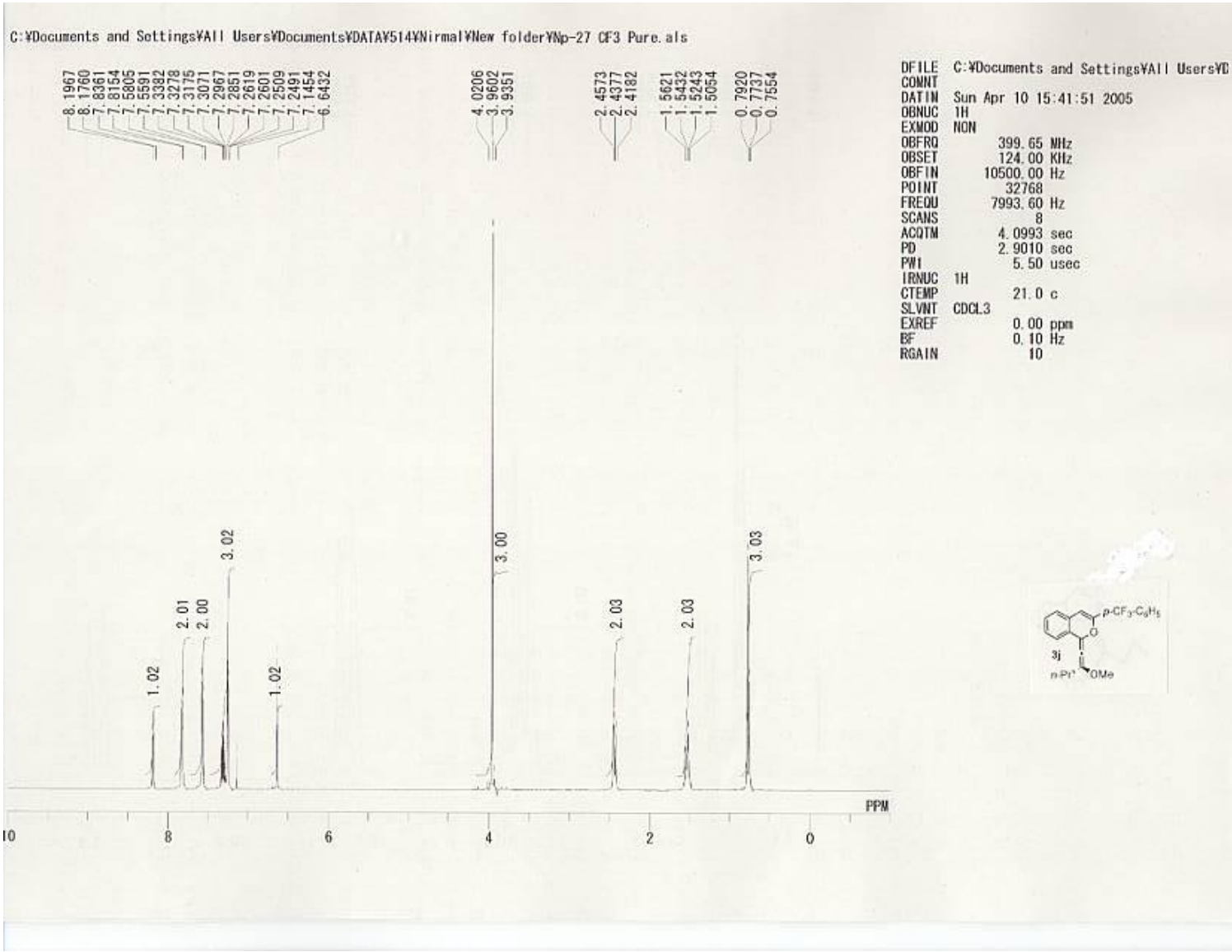

C:¥Documents and Settings¥All Users¥Documents¥DATA¥5144¥Nirmal¥New folder¥Np-33 pure sep PMR.als
DFILE C:¥Documents and Settings¥All
COMNT
DATIM Fri Apr 22 13:51:13 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 105500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 8
PPM
11.31
3.04
2.05
2.03
2.00
1.01
2.01
1.01

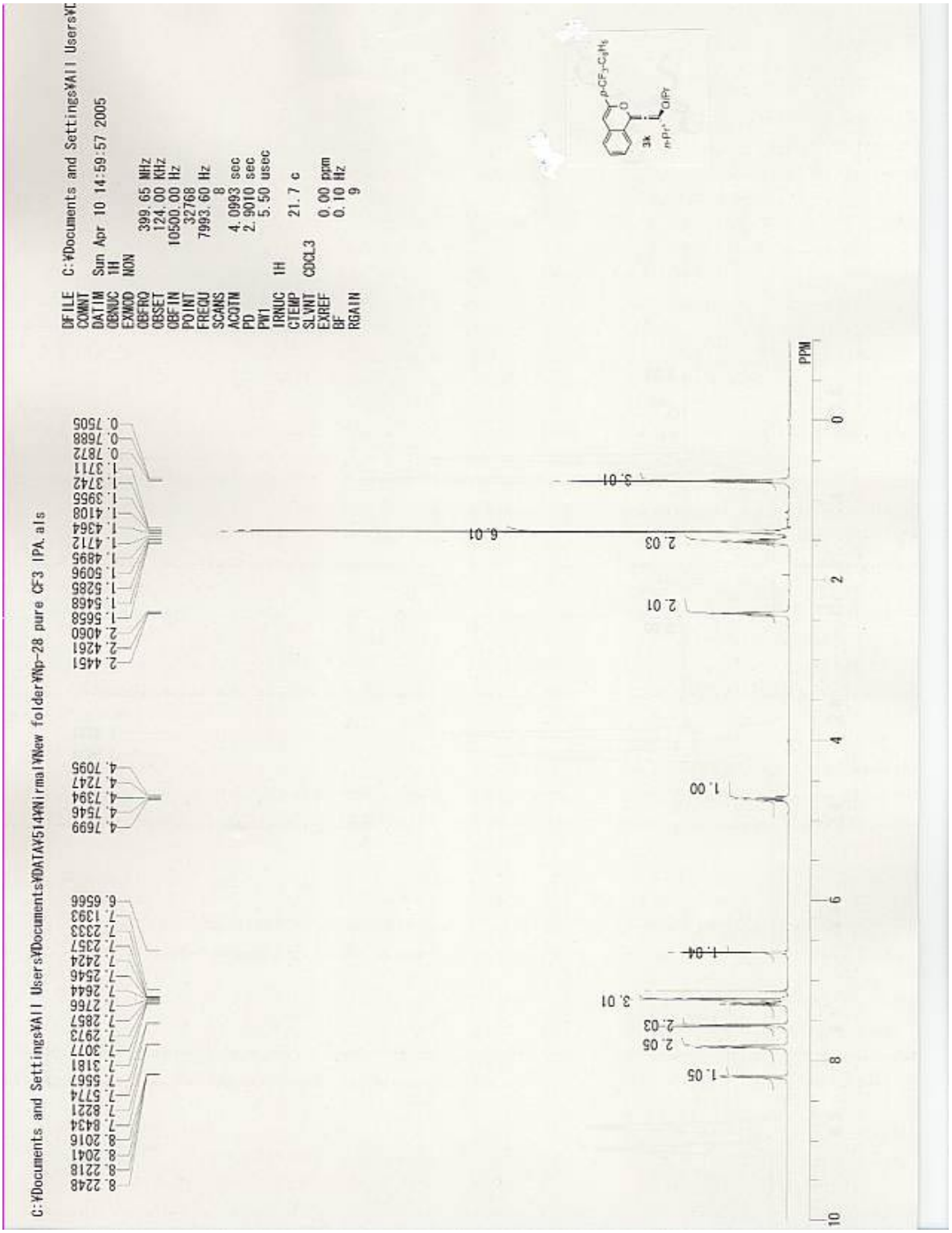

C:¥Documents and Settings¥All Users¥Documents¥DATA¥5514¥Nirmal¥New folder¥Np-22 pure IPA.als
DFILE C:¥Documents and Settings¥All
COMNT
DATIM Sun Apr 10 13:08:46 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 10
PPM
0
2
4
6
8
10
3.00
6.02
2.03
2.00
1.00
1.00
6.02
2.02
1.01
8.2108
8.1900
7.7483
7.7287
7.4724
7.4541
7.4358
7.3266
7.3071
7.2955
7.2918
7.2888
7.2754
7.2516
7.2479
7.2314
7.2143
7.1564
6.6542
4.7491
4.7339
4.7186
2.4615
2.4426
2.4225
1.5542
1.5353
1.5157
1.4968
1.4138
1.3986
1.2918
1.2759
0.7884
0.7701
0.7518

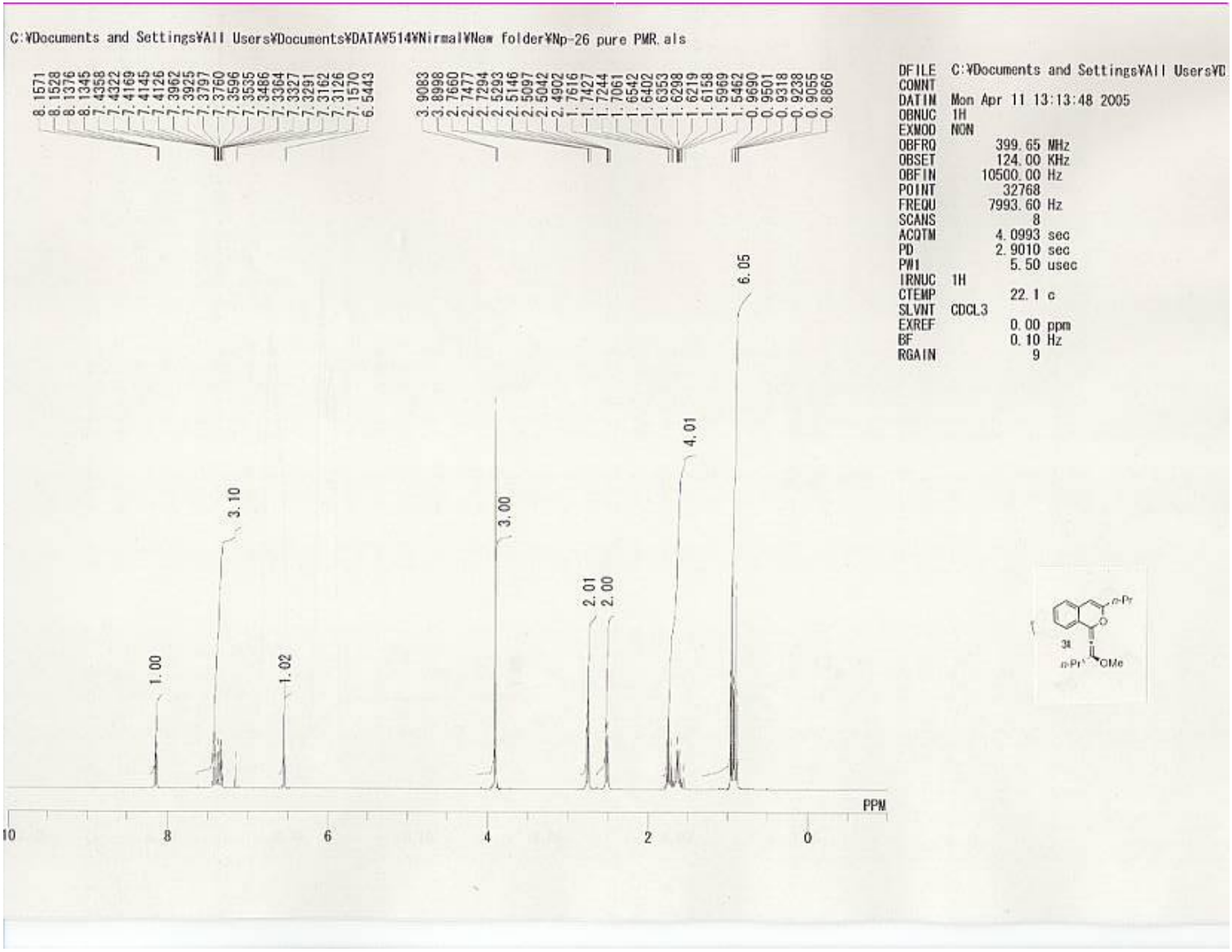

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-32 PMR.als
8.3939
8.3902
8.3731
7.8624
7.8429
7.5835
7.5756
7.5634
7.5573
7.5384
7.4713
7.4517
7.4322
7.4231
7.4194
7.4115
7.4023
7.3925
7.3816
7.3639
7.3608
7.3431
7.2400
6.8489
6.8440
6.8257
5.3478
5.3191
5.2947
5.1299
5.1263
2.5848
2.5799
2.5622
2.5464
2.5409
2.1168
2.1150
1.6585
1.6396
1.6207
1.6067
1.6012
0.8805
0.8763
0.8622
0.8580
0.8439
0.8397
10.86
1.01
2.02
1.02
2.00
2.00
2.01
3.00
PPM
10
8
6
4
2
0
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Tue May 10 13:28:15 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 8
3g
OBn

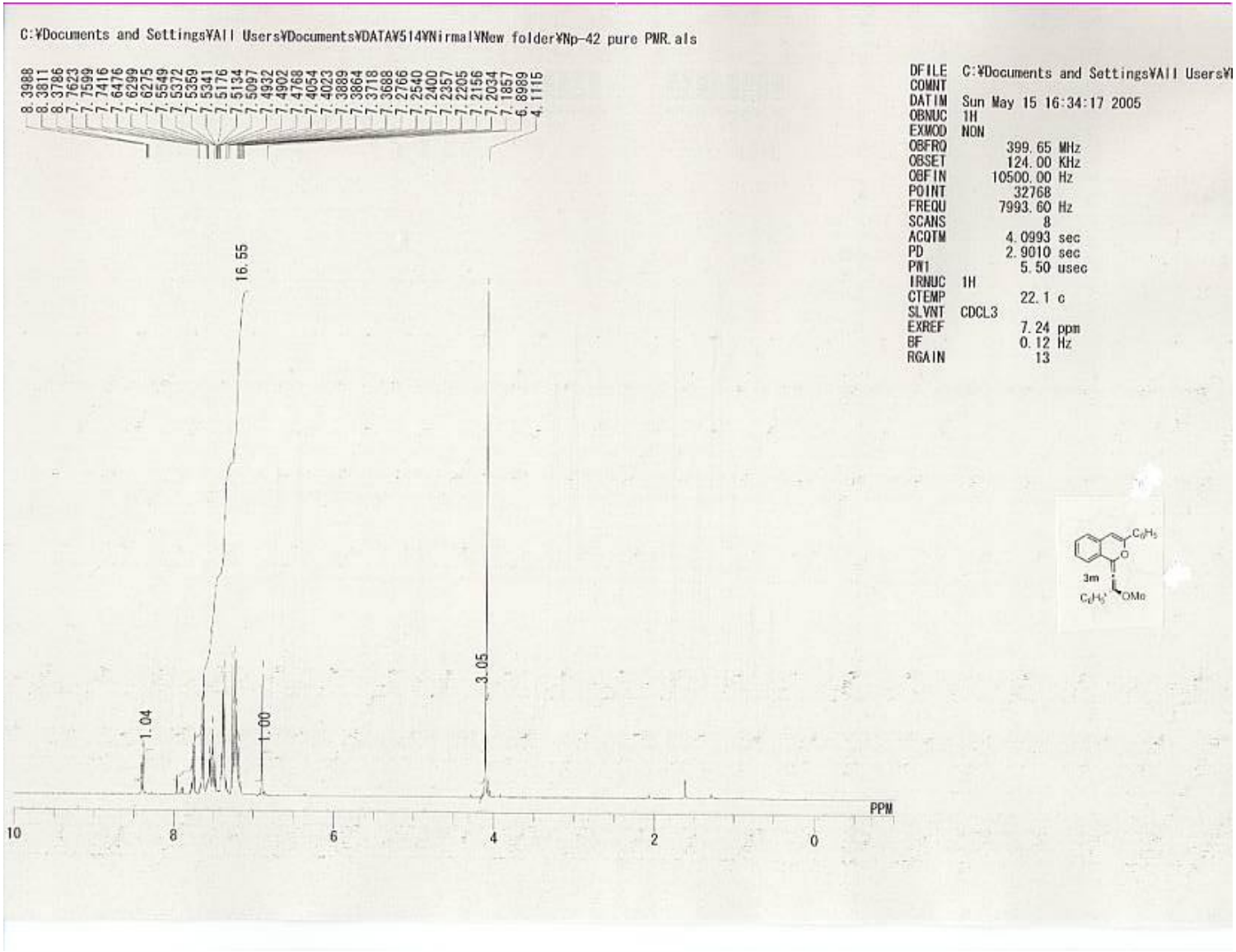

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-31 pure PMR.als
8.29 8.29 8.27 7.75 7.73 7.46 7.44 7.43 7.41 7.41 7.41 7.39 7.39 7.37 7.37 7.35 7.35 7.35 7.33 7.33 7.26 7.22 7.20 6.75
4.06 4.03
2.59 2.57 2.55 2.40 1.70 1.68 1.67 1.65 1.63 0.91 0.89 0.87
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Mon May 09 21:14:57 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 9
1.00
2.00
5.04
1.00
3.00
2.02
3.05
2.06
3.02
PPM
10
8
6
4
2
0
p-CH3-C6H5
3h
R1
OMe

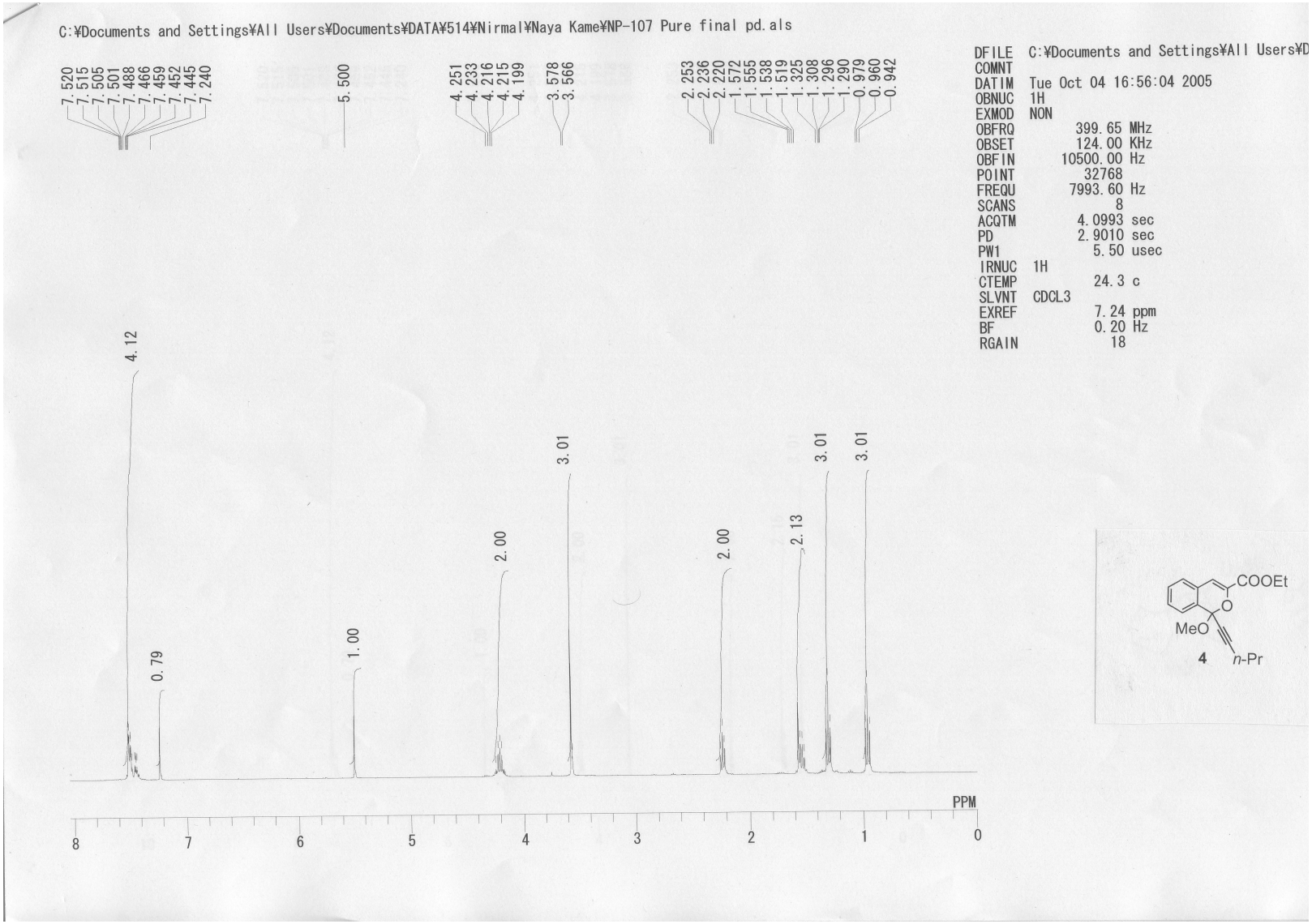

C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-31-1 pure PMR.als IPA.als
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Tue May 10 09:31:03 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 8
8.3353
8.3146
7.7776
7.7593
7.4591
7.4389
7.4353
7.4139
7.4109
7.3938
7.3694
7.3663
7.3517
7.3486
7.3315
7.2766
7.2644
7.2400
7.2199
6.7860
4.8273
4.8419
4.8572
4.8718
4.8871
2.5970
2.5793
2.5610
2.4231
2.3981
1.7086
1.6878
1.6695
1.6512
1.6323
1.5383
1.5231
1.4078
1.3919
0.9251
0.9233
0.9068
0.9043
0.8885
0.8866
3.00
6.01
2.03
3.04
2.02
1.00
1.02
5.35
2.00
1.00
PPM
10
8
6
4
2
0


C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-27 CF3 Pure.als
8.1967
8.1760
7.8361
7.8154
7.5805
7.5591
7.3382
7.3278
7.3175
7.3071
7.2967
7.2851
7.2619
7.2601
7.2509
7.2491
7.1454
6.6432
4.0206
3.9602
3.9351
2.4573
2.4377
2.4182
1.5621
1.5432
1.5243
1.5054
0.7920
0.7737
0.7554
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIN Sun Apr 10 15:41:51 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 10
1.02
2.01
2.00
3.02
1.02
3.00
2.03
2.03
3.03
PPM
10
8
6
4
2
0
p-CF3-C6H5
3j
nPr
OMe


C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-28 pure CF3 IPA.als
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Sun Apr 10 14:59:57 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 9
8.2248
8.2218
8.2041
8.2016
7.8434
7.8221
7.5774
7.5667
7.3181
7.3077
7.2973
7.2857
7.2766
7.2644
7.2546
7.2424
7.2357
7.2333
7.1393
6.6566
4.7699
4.7546
4.7394
4.7247
4.7095
2.4451
2.4261
2.4060
1.5658
1.5468
1.5285
1.5096
1.4895
1.4712
1.4364
1.4108
1.3965
1.3742
1.3711
0.7872
0.7688
0.7505
6.01
5.01
2.03
2.01
1.00
1.04
3.01
2.05
2.03
1.05
PPM
0
2
4
6
8
10


C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-26 pure PMR.als
8.1571
8.1528
8.1376
8.1345
7.4358
7.4322
7.4169
7.4145
7.4126
7.3962
7.3925
7.3797
7.3760
7.3596
7.3535
7.3486
7.3364
7.3327
7.3291
7.3162
7.3126
7.1570
6.5443
3.9083
3.8998
2.7660
2.7477
2.7294
2.5293
2.5146
2.5097
2.5042
2.4902
2.7616
1.7427
1.7244
1.7061
1.6542
1.6402
1.6353
1.6298
1.6219
1.6158
1.5969
1.5462
0.9690
0.9501
0.9318
0.9238
0.9055
0.8866
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Mon Apr 11 13:13:48 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 9
1.00
3.10
1.02
3.00
2.01
2.00
4.01
6.05
PPM
10
8
6
4
2
0
n-Pr
3H
n-Pr
OMe


C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥Np-42 pure PMR.als
8.3988
8.3811
8.3786
7.7623
7.7599
7.7416
7.6476
7.6299
7.6275
7.5549
7.5372
7.5359
7.5341
7.5176
7.5134
7.5097
7.4932
7.4902
7.4768
7.4054
7.4023
7.3889
7.3864
7.3718
7.3688
7.2766
7.2540
7.2400
7.2357
7.2205
7.2156
7.2034
7.1857
6.8989
4.1115
DFILE C:¥Documents and Settings¥All Users¥
COMNT
DATIM Sun May 15 16:34:17 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 13
16.55
3.05
1.04
1.00
PPM
10
8
6
4
2
0
3m
OMe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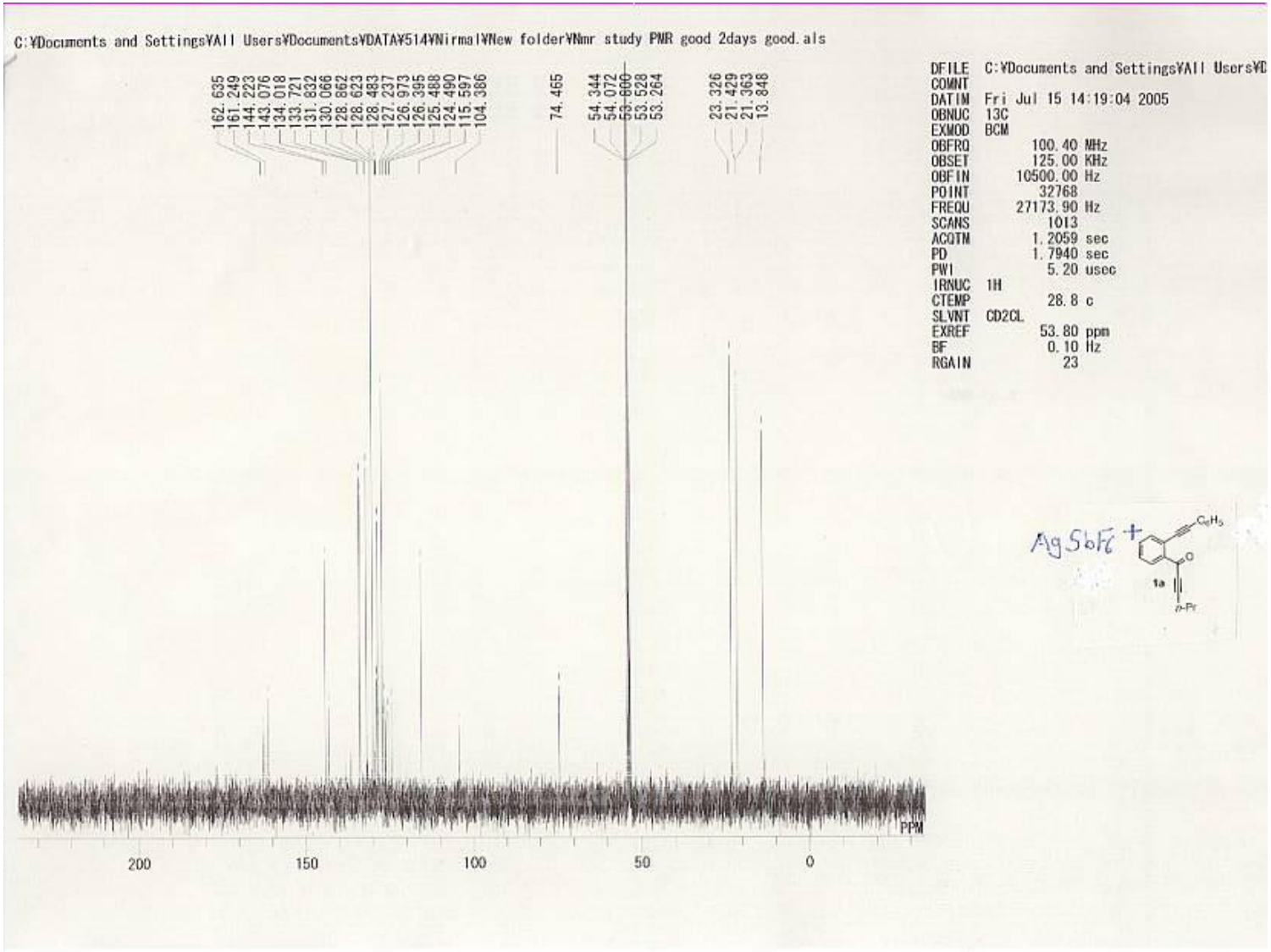
7.520
7.515
7.505
7.501
7.488
7.466
7.459
7.452
7.445
7.240
5.500
4.251
4.233
4.216
4.215
4.199
3.578
3.566
2.253
2.236
2.220
1.572
1.555
1.538
1.519
1.325
1.308
1.296
1.290
0.979
0.960
0.942
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Tue Oct 04 16:56:04 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.20 Hz
RGAIN 18
4.12
0.79
1.00
2.00
3.01
2.00
2.13
3.01
3.01
PPM
8
7
6
5
4
3
2
1
0
COOEt
O
MeO
4
n-Pr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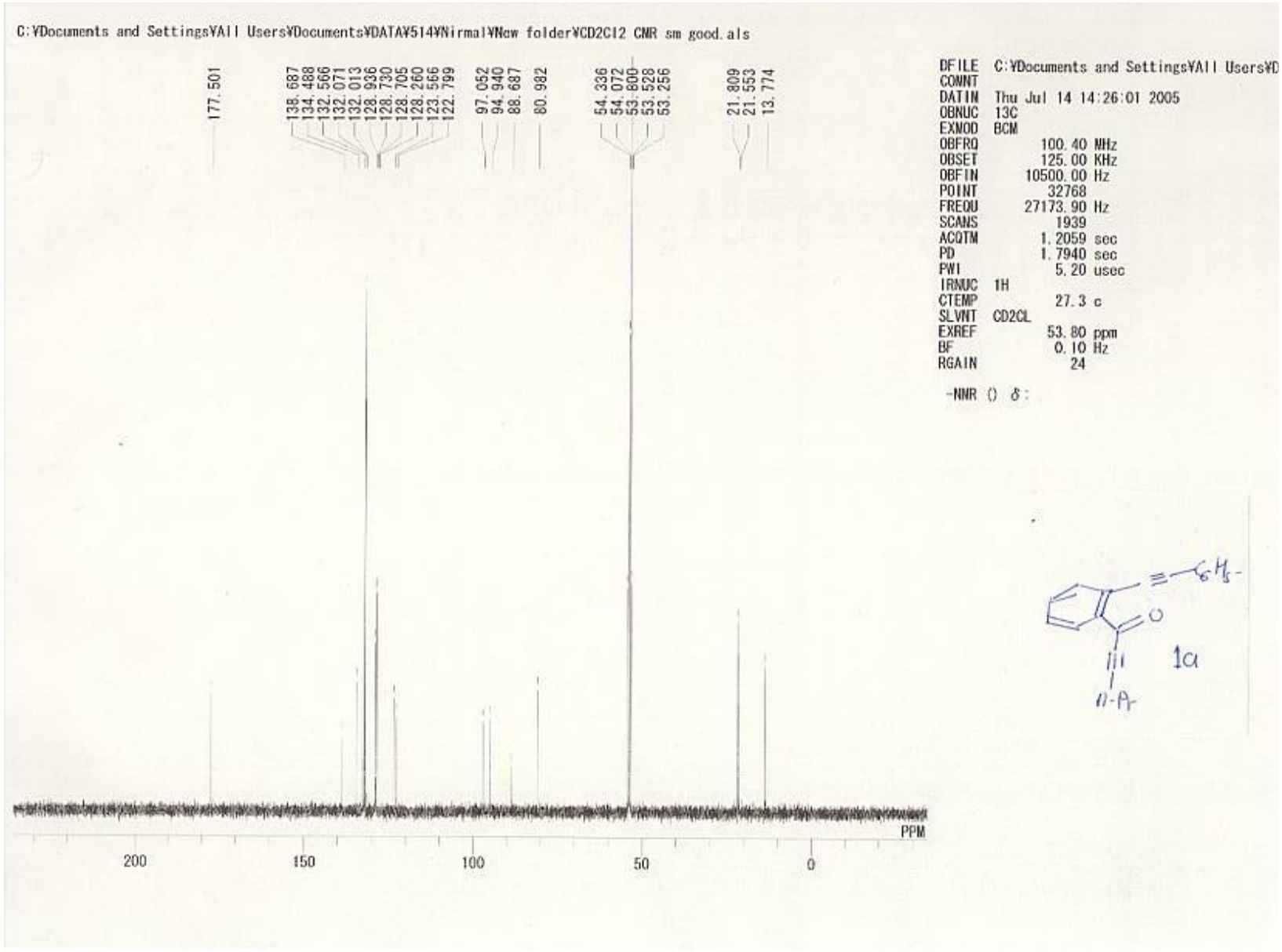
162.635
161.249
144.223
143.076
134.018
133.721
131.832
130.066
128.862
128.623
128.483
127.237
126.973
126.395
125.488
124.490
115.597
104.386
74.465
54.344
54.072
53.800
53.528
53.264
23.326
21.429
21.363
13.848
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Fri Jul 15 14:19:04 2005
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27173.90 Hz
SCANS 1013
ACQTM 1.2059 sec
PD 1.7940 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 28.8 c
SLVNT CD2CL
EXREF 53.80 ppm
BF 0.10 Hz
RGAIN 23
AgSbF6
1a
n-Pr
PPM
200
150
100
50
0


C:¥Documents and Settings¥All Users¥Documents¥DATA¥514¥Nirmal¥New folder¥CD2Cl2 CNR sm good.als
177.501
138.687
134.488
132.566
132.071
132.013
128.936
128.730
128.705
128.260
123.566
122.799
97.052
94.940
88.687
80.982
54.336
54.072
53.800
53.528
53.256
21.809
21.553
13.774
DFILE C:¥Documents and Settings¥All Users¥D
COMNT
DATIM Thu Jul 14 14:26:01 2005
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27173.90 Hz
SCANS 1939
ACQTM 1.2059 sec
PD 1.7940 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 27.3 c
SLVNT CD2CL
EXREF 53.80 ppm
BF 0.10 Hz
RGAIN 24
-NNR () δ:
1a
PPM
200
150
100
50
0