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Supporting Information

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Enantioselective Organocatalytic Hydrophosphination of α,β-Unsaturated Aldehydes

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General. Chemicals and solvents were either purchased *puriss p.A.* from commercial suppliers or purified by standard techniques. Catalysts **11** and **12** were synthesized according to literature procedures.[1] For thin-layer chromatography (TLC), silica gel plates Merck 60 F254 were used and compounds were visualized by irradiation with UV light and/or by treatment with a solution of phosphomolybdic acid (25 g), $Ce(SO_4)_2 \cdot H_2O$ (10 g), conc. H_2SO_4 (60 mL), and H_2O (940 mL) followed by heating or by treatment with a solution of *p*-anisaldehyde (23 mL), conc. H_2SO_4 (35 mL), acetic acid (10 mL), and ethanol (900 mL) followed by heating. Flash chromatography was performed using silica gel Merck 60 (particle size 0.040-0.063 mm), ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded on Varian AS 400. Chemical shifts are given in δ relative to tetramethylsilane (TMS) and to external standard 85% H_3PO_4 (^{31}P NMR), the coupling constants *J* are given in Hz. The spectra were recorded in $CDCl_3$ as solvent at room temperature, TMS served as internal standard (δ = 0 ppm) for ^{1}H NMR, and $CDCl_3$ was used as internal standard (δ = 77.0 ppm) for ^{13}C NMR. HPLC was carried out using a Waters 2690 Millennium with photodiode array detector. Optical rotations were recorded on a Perkin Elemer 241 Polarimeter (λ = 589 nm, 1 dm cell). High-resolution mass spectra were recorded on a Bruker MicrOTOF spectrometer.

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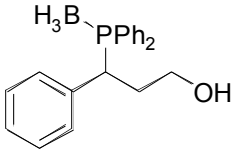
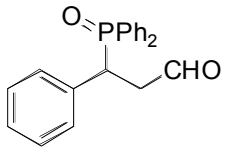
1. a) M. Marigo, T. C. Wabnitz, D. Fielenbach, K. A. Jørgensen, *Angew. Chem. Int. Ed.* **2005**, *44*, 794. b) M. Marigo, D. Fielenbach, A. Braunton, A. Kjaersgaard, K. A. Jørgensen, *Angew. Chem. Int. Ed.* **2005**, *44*, 3703. c) J. Franzén, M. Marigo, D. Fielenbach, T. C. Wabnitz, A. Kjaersgaard, K. A. Jørgensen, *J. Am. Chem. Soc.* **2005**, *127*, 18296. d) Y. Hayashi, H. Gotoh, T. Hayashi, M. Shoji, *Angew. Chem. Int. Ed.* **2005**, *44*, 4212. e) M. Marigo, J. Franzén, T. B. Poulsen, W. Zhuang, K. A. Jørgensen, *J. Am. Chem. Soc.* **2005**, *127*, 6964. g) H. Sundén, I. Ibrahem, A. Córdova, *Tetrahedron Lett.* **2006**, 47, 99. h) I. Ibrahem, A. Córdova, *Chem. Commun.* **2006**, 1760.

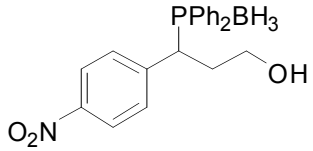
Typical experimental procedure for the organocatalytic AHP reactions. To a stirred solution of catalyst (20 mol%) and 2-fluorobenzoic acid (10 mol%) in $CHCl_3$ (1.0 mL) at 4 °C, was added 0.25 mmol (1.0 equiv) of α,β-unsaturated aldehyde **1**. The reaction was then flushed with Ar, and 0.3 mmol (1.2 equiv) of diphenylphosphine was added. The reaction was stirred at 4 °C for the time reported in Table 2. Next, the product was oxidized or reduced *in situ*. Thus,1.5 equiv of I_2 in 2%H_2O in pyridine (1.2 mL) was added. The crude mixture was purified by column chromatography to afford the pure phosphineoxide aldehydes **5**. In order to determine the ee, the crude was reduced "in situ" by $NaBH_4$ in MeOH, and the crude was purifed by column chromatography to afford the corresponding phosphine alcohol derivatives **4**.



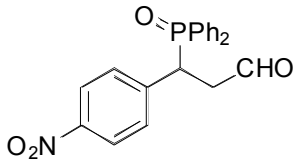
4a: Colorless oil. IR (KBr): 3226, 3080, 3029, 2970, 1593, 1495, 1454, 1437, 1178, 1048, 752, 697 cm^{-1} ^{31}P (162 MHz, $CDCl_3$): 24.5 (m). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.00-7.93 (m, 2H), 7.56-7.48 (m, 3H), 7.36-7.29 (m, 3H), 7.22-7.18 (m, 2H), 7.17-7.12 (m, 5H), 4.10-3.99 (m, 1H), 3.62-3.56 (m, 1H), 3.39-3.31 (m, 1H), 2.34-2.26 (m, 1H), 2.16-2.06 (m, 1H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 135.3, 133.4, 133.3, 132.9, 132.8, 131.7, 131.6, 131.0, 130.9, 130.3, 130.2, 129.2, 129.1, 128.3, 128.2, 127.5, 127.4, 125.8, 60.1 (d, J_{C-P} =12.9Hz), 39.2 (d, J_{C-P} =31.8Hz), 33.0 (d, J_{C-P}=3.7Hz), $[\alpha]_D$ = -13.2 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+Na]^+$($C_{21}H_{24}BOP$) requires m/z 357.1550, found 357.1565. The enantiomeric excess (83%) was determined by HPLC with an AD column. (*n*-hexane: *i*-PrOH = 95:5, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 29.77 min, minor enantiomer 25.3 min.



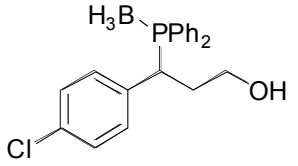
5a: Colorless solid. IR (KBr): 3080, 3029, 2970, 1966, 1723, 1593, 1495, 1454, 1437, 1178, 1048, 752, 697 cm^{-1} ^{31}P (162 MHz, $CDCl_3$): 34.5 (s). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.60 (t, *J*=1.47Hz, 1H), 7.92-7.87 (m, 2H), 7.55-7.13 (m, 13H), 4.23-4.16 (m, 1H), 3.38-3.28 (m, 1H), 3.01-2.92 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 199.1 (d, J_{C-P}=13.7Hz), 135.4, 135.3, 132.4, 132.3, 131.8, 131.7, 131.5, 131.4, 131.3, 131.2, 129.9, 129.8, 129.2, 129.1, 128.6, 128.5, 127.6, 127.5, 125.9, 44.0, 40.2 (d, J_{C-P}=68.3Hz). Enantiomeric excess: 83%. $[\alpha]_D$ = -44.2 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+(C_{21}H_{20}O_2P)$ requires m/z 335.1195, found 335.1189.



4b: Colorless oil. IR (KBr): 3397, 2928, 2392, 1722, 1597, 1519, 1346, 1261, 694 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 25.2 (m).^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05-7.95 (m, 4H), 7.60-7.47 (m, 3H), 7.35-7.05 (m, 7H), 4.24 (ddd, J=2.8Hz, 12Hz, 15.2Hz, 1H), 3.66-3.60 (m, 1H), 2.80 (td, J= 3.2Hz, 10Hz, 1H), 2.36-2.26 (m, 1H), 2.23-2.10 (m, 1H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 147.0, 141.5, 133.0, 132.9, 132.5, 132.4, 131.8, 131.8, 131.4, 131.3, 130.9, 130.8, 129.2, 129.1, 128.6, 128.5, 128.1, 127.5, 127.2, 126.7, 123.1, 123.1, 59.4 (d, J_{C-P}=11.4Hz), 39.9 (d, J_{C-P}=31.1Hz) 32.6 (d, J_{C-P}=3.0Hz). $[\alpha]_D$ = -25.7 (c = 1.0, $CHCl_3$)**.** HRMS (ESI): calcd. for $[M+Na]^+(C_{21}H_{23}NPO_3)$ requires m/z 402.1401, found 402.1407. The enantiomeric excess (99%) was determined by HPLC with an ODH column. (*n*-hexane: *i*-PrOH = 90:10, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 14.9 min, minor enantiomer 20.9 min.

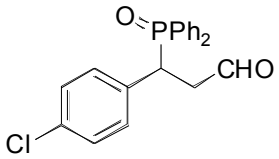


5b: white solid. IR (KBr): 3079, 2970, 2618, 1966, 1723, 1601, 1491, 1485, 1260, 1048, 751 cm^{-1}.^{31}P (162 MHz, $CDCl_3$): 34.7 (s). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.60 (m, 1H), 8.02 (d, *J*=8.75Hz, 2H), 7.72-7.61 (m, 2H), 7.46-7.43 (m, 3H), 7.32-7.28 (m, 3H), 7.19-7.15 (m, 4H), 4.30-4.21 (m, 1H), 3.17-3.10 (m, 1H), 2.87-2.80 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 199.3 (d, J_{C-P}=12.2Hz), 148.7, 148.6, 134.9, 134.8, 134.1, 134.0, 133.9, 133.5, 133.4, 130.3, 130.0, 129.8, 129.6, 129.3, 129.2, 128.6, 128.5, 47.2 (d, J_{C-P}=19.7Hz), 38.8 (d, J_{C-P}=15.0Hz). Enantiomeric Excess: 99%. $[\alpha]_D$ = -176.5 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+(C_{21}H_{19}NO_4P)$ requires m/z 380.1046, found 380.1039.

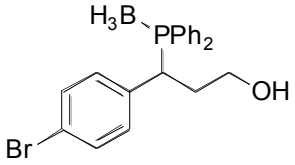


4c: Colorless oil. IR (KBr): 3217 (bs), 2928, 2616, 1965, 1732, 1682, 1591, 1488, 1411, 1216, 1150, 842 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 24.8 (m). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.98-7.92 (m, 2H), 7.57-7.50 (m, 3H), 7.37-7.30 (m, 3H), 7.24-7.20 (m, 2H), 7.13 (d, *J*=8.4Hz, 2H), 7.06 (d, *J*=8.4Hz, 2H), 4.08-3.99 (m, 1H), 3.64-3.58 (m, 1H), 3.36-3.29 (m, 1H), 2.29-2.20 (m, 1H), 2.14-2.06 (m, 1H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 134.1, 134.0, 133.4, 133.3, 133.0, 132.8, 132.0, 131.8, 131.6, 131.5, 131.3, 131.2, 129.3, 129.2, 129.0, 128.9, 128.8, 128.6, 128.5, 128.4, 127.9, 59.9 (d, J_{C-P}=12.2Hz), 38.5 (d, J_{C-P}=32.7Hz), 32.3 (d, J_{C-P}=4.6Hz). $[\alpha]_D$ = -56.9 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+Na]^+(C_{21}H_{23}BClOP)$ requires m/z 391.1160, found 391.1174. The enantiomeric excess (92%) was determined by HPLC with an ODH column. (*n*-

hexane: *i*-PrOH = 93:7, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 13.3 min, minor enantiomer 18.0 min.

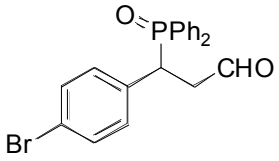


5c: Colorless solid. IR (KBr): 3061, 3029, 2020, 2970, 1965, 1847, 1723, 1601, 1592, 1436, 1259, 1118, 1047 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 34.9 (s). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.59 (m, 1H), 7.93-7.88 (m, 2H), 7.58-7.35 (m, 8H), 7.24 (d, *J*=8.4Hz, 2H), 7.14 (d, *J*=8.4Hz, 2H), 4.21-4.15 (m, 1H), 3.36-3.26 (m, 1H), 3.03-2.93 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 198.7 (d, J_{C-P}=13.6Hz), 134.2, 134.1, 133.5, 133.4, 132.6, 132.5, 132.1, 132.0, 131.5, 131.4, 131.3, 131.2, 131.1, 131.0, 129.3, 129.2, 128.9, 128.8, 128.6, 128.5, 44.2, 39.8 (d, J_{C-P}=68.3Hz). Enantiomeric Excess = 92%. $[\alpha]_D$ = -56.8 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+$($C_{21}H_{19}ClO_2P$) requires m/z 369.0806, found 369.0812.

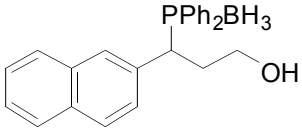


4d: Colorless oil. IR (KBr): 3302, 3080, 2958, 2932, 2382, 1725, 1663, 1628, 1488, 1437, 1063, 738, 693 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 24.8 (m). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.00-7.92 (m, 2H), 7.56-7.52 (m, 3H), 7.37-7.33 (m, 3H), 7.28 (d, *J*=8.4Hz, 2H), 7.24-7.22 (m, 2H), 7.01 (d, *J*=8.4Hz, 2H), 4.07-3.98 (m, 1H), 3.64-3.57 (m, 1H), 3.36-3.28 (m, 1H), 2.30-2.19 (m, 1H), 2.15-2.06 (m, 1H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 134.5, 134.4, 133.4, 133.3, 132.9, 132.8, 132.0, 131.9, 131.8, 131.7, 131.5, 131.4, 131.3, 131.2, 129.3, 129.2, 128.6, 128.5, 121.6, 121.5, 59.9 (d, J_{C-P}=12.1Hz), 38.6

(d, $J_{C\text{-}P}$=32.7Hz), 32.9 (d, $J_{C\text{-}P}$=4.6Hz). $[\alpha]_D$ = -44.8 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+Na]^+(C_{21}H_{23}BBr^{79}OP)$ requires m/z 435.0655, found 435.0676. The enantiomeric excess (90%) was determined by HPLC with an AD column. (*n*-hexane: *i*-PrOH = 92:8, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 16.7 min, minor enantiomer 18.3 min.

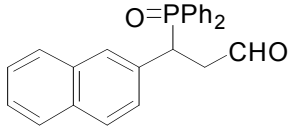


5d: Colorless solid. IR (KBr): 3063, 3028, 2967, 1966, 1843, 1726, 1602, 1587, 1422, 1259, 1115, 1047, 738 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 34.5 (s). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.58 (m, 1H), 7.92-7.86 (m, 2H), 7.60-7.36 (m, 8H), 7.29 (d, *J*=8.4Hz, 2H), 7.17 (d, *J*=8.4Hz, 2H), 4.20-4.14 (m, 1H), 3.36-3.26 (m, 1H), 3.03-2.93 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 198.6 (d, $J_{C\text{-}P}$=13.6 Hz), 135.0, 134.7, 133.0, 132.8, 132.5, 132.4, 132.1, 131.6, 131.5, 131.4, 131.3, 131.2, 131.1, 131.0, 130.9, 129.3, 129.2, 128.6, 128.5, 44.1, 39.5 (d, $J_{C\text{-}P}$=67Hz). Enantiomeric Excess = 90%. $[\alpha]_D$ = -67.0 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+(C_{21}H_{19}Br^{81}O_2P)$ requires m/z 415.0283, found 415.0264.

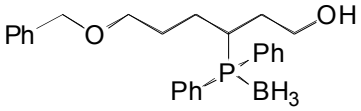


4e: Colorless oil. IR (KBr): 3365, 3060, 2935, 2880, 2390, 1600, 1486, 1436, 1217, 1062, 738, 600 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 24.6 (m)**.** ^1H NMR (400 MHz, $CDCl_3$): δ = 8.14-8.12 (m, 1H), 8.04-7.98 (m, 2H), 7.84-7.80 (m, 2H), 7.78-7.74 (m, 1H), 7.64-7.60 (m, 2H), 7.54-7.24 (m, 7H), 7.18-7.14 (m, 2H), 4.82 (bs, 1H), 4.25-4.16 (m, 1H), 3.65-3.60 (m, 1H), 3.40-3.33 (m, 1H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 133.6, 133.2, 133.1, 133.0, 132.7, 132.6, 131.5, 131.4, 130.8, 130.2, 129.3, 129.2, 129.0, 128.9, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127,7, 127.6, 126.2, 126.0, 59.9 (d, $J_{C\text{-}P}$=12.2Hz), 39.1 (d, $J_{C\text{-}P}$=31.9Hz), 32.8 (d, $J_{C\text{-}P}$=3.8Hz). $[\alpha]_D$ = -15.2 (c = 0.5, $CHCl_3$).

HRMS (ESI): calcd. for $[M+Na]^+(C_{25}H_{26}BOP)$ requires m/z 407.1707, found 407.1712. The enantiomeric excess (98%) was determined by HPLC with an AD column. (*n*-hexane: *i*-PrOH = 95:5, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 41.6 min, minor enantiomer 70.2 min.

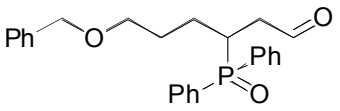


5e: Yellow solid. IR (KBr): 2919, 2851, 1437, 1788, 1721, 1626, 1599, 1437, 1354, 913, 744 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 34.1 (s). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.61 (m, 1H), 8.00-7.14 (m, 17H), 4.40-4.34 (m, 1H), 3.50-3.40 (m, 1H), 3.12-2.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 198.8 (d, J_{C-P}=13.5), 132.5, 132.2, 132.1, 131.6, 131.6, 131.4, 131.3, 131.2, 131.1, 131.0, 129.1, 129.0, 128.9, 128.8, 128.7, 128.2, 128.1, 128.1, 127.9, 127.6, 127.6, 127.2, 127.5, 126.1, 126.0, 44.1, 40.1 (d, J_{C-P}=68.0Hz). Enantiomeric Excess: 98%. $[\alpha]_D$ = -45.7 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+(C_{25}H_{21}O_2P)$ requires m/z 385.1352, found 385.1357.

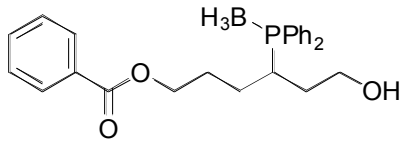


4f: Colorless oil: IR (KBr): 3056, 2920, 2857, 1718, 1663, 1589, 1437, 1357, 1278, 1161, 1118, 913, 747 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 24.0 (m). $[\alpha]_D$= -11.0 (c=1.0). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.79-7.73 (m, 3H), 7.48-7.28 (m, 12H), 4.44 (q, *J*=7.6Hz, 11.6Hz, 2H), 3.59-3.47 (m, 2H), 3.44-3.33 (m, 2H), 2.86-2.76 (m, 1H), 1.96-1.56 (m, 6H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 138.3, 132.8, 132.8, 131.3, 129.4, 128.9, 128.8, 128.6, 127.9, 127.8, 73.1, 70.3, 60.8 (d, J_{C-P}=10.0Hz), 32.8 (d, J_{C-P}=3.0Hz), 29.2 (d, J_{C-P}=34.9Hz), 28.4 (d, J_{C-P}=9.2Hz), 27.0 (d, J_{C-P}=3.0Hz). HRMS (ESI): calcd. for $[M+Na]^+(C_{25}H_{32}BNO_2P)$ requires m/z 429.2125, found 429.2134. The enantiomeric

excess (91%) was determined by HPLC with an ODH column. (*n*-hexane: *i*-PrOH = 97:3, λ =210nm), 1.0 mL/min t_R = major enantiomer 60.3 min, minor enantiomer 66.3 min.

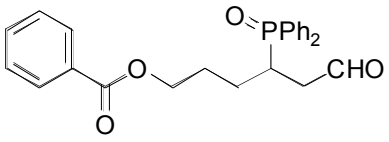


5f: Colorless oil: IR (KBr): 3391, 2925, 2860, 2380, 1719, 1437, 1279, 1105, 1064, 739 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 36.7 (s). Enantiomeric Excess = 91%. $[\alpha]_D$= -33.1 (c=1.0). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.67 (s, 1H), 7.84-7.75 (m, 3H), 7.52-7.43 (m, 6H), 7.33-7.23 (m, 6H), 4.41 (d, *J*=11.2Hz, 1H), 4.39 (d, *J*=11.2Hz, 1H), 3.42-3.33 (m, 2H), 3.21-3.12 (m, 1H), 2.83 (dt, *J*=3.6Hz, 16.4Hz, 1H), 2.68 (dt, *J*=4.0Hz, 17.2Hz, 1H), 1.87-1.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): 199.5 (d, J_{C-P} =9.9Hz), 138.7, 132.1, 132.0, 131.3, 131.2, 131.1, 129.1, 129.0, 128.9, 128.8, 128.7, 128.5, 127.7, 73.0, 69.8, 42.4, 30.8 (d, J_{C-P}=72.0Hz), 27.9 (d, J_{C-P}=11.3Hz), 25.6. HRMS (ESI): calcd. for $[M+H]^+$($C_{25}H_{27}NO_3P$) requires m/z 407.1771, found 407.1790.



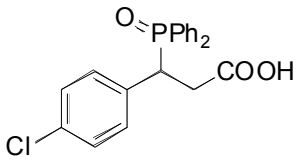
4g: Colorless oil. IR (KBr): 3307 (bs), 2924, 2381, 1752, 1714, 1698, 1606, 1457, 1276, 1194, 913, 744 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 24.7 (m). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.84 (m, 2H), 7.82-7.72 (m, 4H), 7.60-7.56 (m, 1H), 7.50-7.30 (m, 8H), 4.22-4.18 (m, 2H), 3.64-3.56 (m, 2H), 2.86-2.78 (m, 1H), 2.00-1.50 (m, 6H), 1.7-0.9 (bs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 166.5, 132.9, 132.7 (d, J_{C-P} =4.3Hz), 132.5 (d, J_{C-P} =3.9Hz), 131.3, 131.2 (J_{C-P} =2.3Hz), 129.5, 128.9 (d, J_{C-P} =17.6Hz), 128.7, 128.5 (d, J_{C-P} =23.3Hz), 64.2, 60.4, 32.5 (d, J_{C-P} =3.3Hz), 28.9 (d, J_{C-P} =35.1Hz), 27.6 (d, J_{C-P} =8.7Hz), 26.2 (d, J_{C-P} =3.1Hz). $[\alpha]_D$ = -11.4 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+Na]^+$($C_{25}H_{30}BO_3P$) requires m/z 443.1918, found 443.1922. The enantiomeric excess

(95%) was determined by HPLC with an AD column. (*n*-hexane: *i*-PrOH = 92:8, λ =250 nm), 1.0 mL/min; t_R = major enantiomer 16.7 min, minor enantiomer 18.3 min.



5g: Colorless oil. IR (KBr): 3057, 2920, 1717, 1438, 1275, 1117, 713 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 37.7 (s). ^1H NMR (400 MHz, $CDCl_3$): δ = 9.71 (m, 1H), 7.94-7.80 (m, 5H), 7.60-7.30 (m, 10H), 4.23-4.15 (m, 2H), 3.22-3.18 (m, 1H), 2.94-2.60 (m, 2H), 1.90-1.80 (m, 2H), 1.70-1.60 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 199.1 (d, J_{C-P}=10.6 Hz), 166.4, 132.8, 132.0, 131.9, 131.0, 130.9, 129.5, 128.9, 128.9, 128.8, 128.7, 128.3, 64.0, 42.3, 30.4 (d, J_{C-P}=72.1Hz), 26.8 (d, J_{C-P}=10.7Hz), 25.1(d, J_{C-P}=1.5Hz). Enantiomeric Excess = 95%. $[\alpha]_D$ = -34.2 (c = 1.0, $CHCl_3$). HRMS (ESI): calcd. for $[M+H]^+$($C_{25}H_{25}O_4P$) requires m/z 421.1563, found 421.1575.

Typical experimental procedure for the direct enantioselective catalytic hydrophosphination of α,β unsaturated aldehydes and *in situ* oxidation to acid. To a stirred solution of catalyst (20 mol%) and 2-fluorobenzoic acid (10 mol%) in $CHCl_3$ (1.0 mL) at 4 °C, was added 0.25 mmol (1.0 equiv) of α,β-unsaturated aldehyde. The reaction was then flushed with Ar, and 0.3 mmol (1.2 equiv) of diphenylphosphine was added. The reaction was stirred at 4 °C for the time reported in Table 2. Next, isobutene (0.1 mL), *tert*-butanol (0.4 mL), H_2O (0.2 mL) KH_2PO_4 (54.4 mg, 4 mmol), and $NaClO_2$ (36 mg, 4mmol) were added sequentially. The reaction was allowed to reach room temperature and was stirred overnight. The crude was purified by column chromatography (pentane/Ethyl Acetate mixtures) to afford the desired acid.



13c: Colorless solid. IR (KBr): 3057, 3025, 2969, 2916, 2533, 2227, 1717, 1486, 1438, 1164, 840, 726 cm^{-1}. ^{31}P (162 MHz, $CDCl_3$): 35.9 (s). $[\alpha]_D$= -35.2 (c=1.0). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.65 (s, 1H), 7.90-7.85 (m, 2H), 7.60-7.40 (m, 2H), 7.17 (d, *J*=8.5Hz, 2H), 7.06 (d, *J*=8.5Hz, 2H), 4.16-4.10 (m, 1H), 3.12-3.02 (m, 1H), 2.90-2.81 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 172.9, 172.7, 134.0, 133.9, 133.3, 133.2, 133.0, 132.9, 132.5, 132.0, 131.7, 131.5, 131.4, 131.3, 131.2, 131.1, 131.0, 130.3, 130.1, 129.3, 129.2, 128.6, 128.5, 42.6, 42.0, 35.1. HRMS (ESI): calcd. for $[M+H]^+$($C_{21}H_{18}ClO_3P$) requires m/z 407.0580, found 407.0583.

Cartesian coordinates of optimized transition states:

TS-*S*

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TS-*R*

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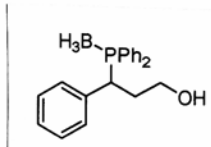
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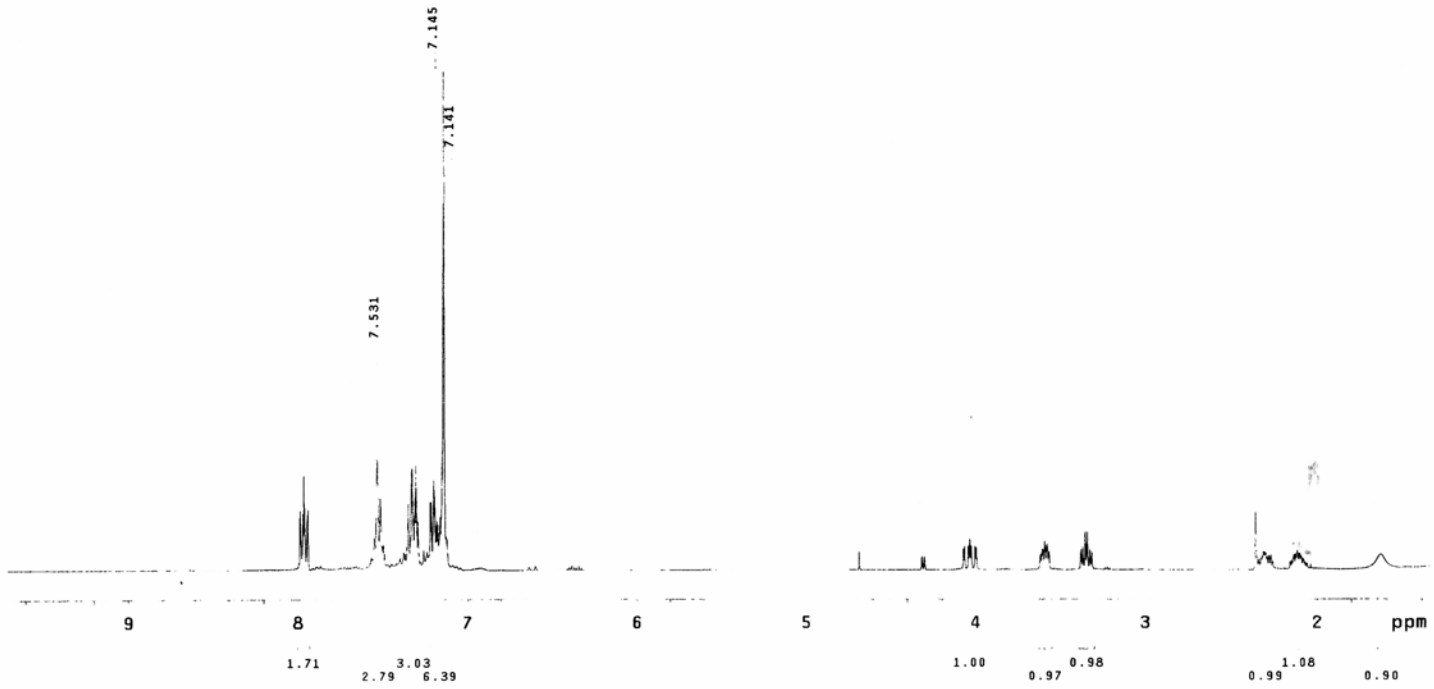
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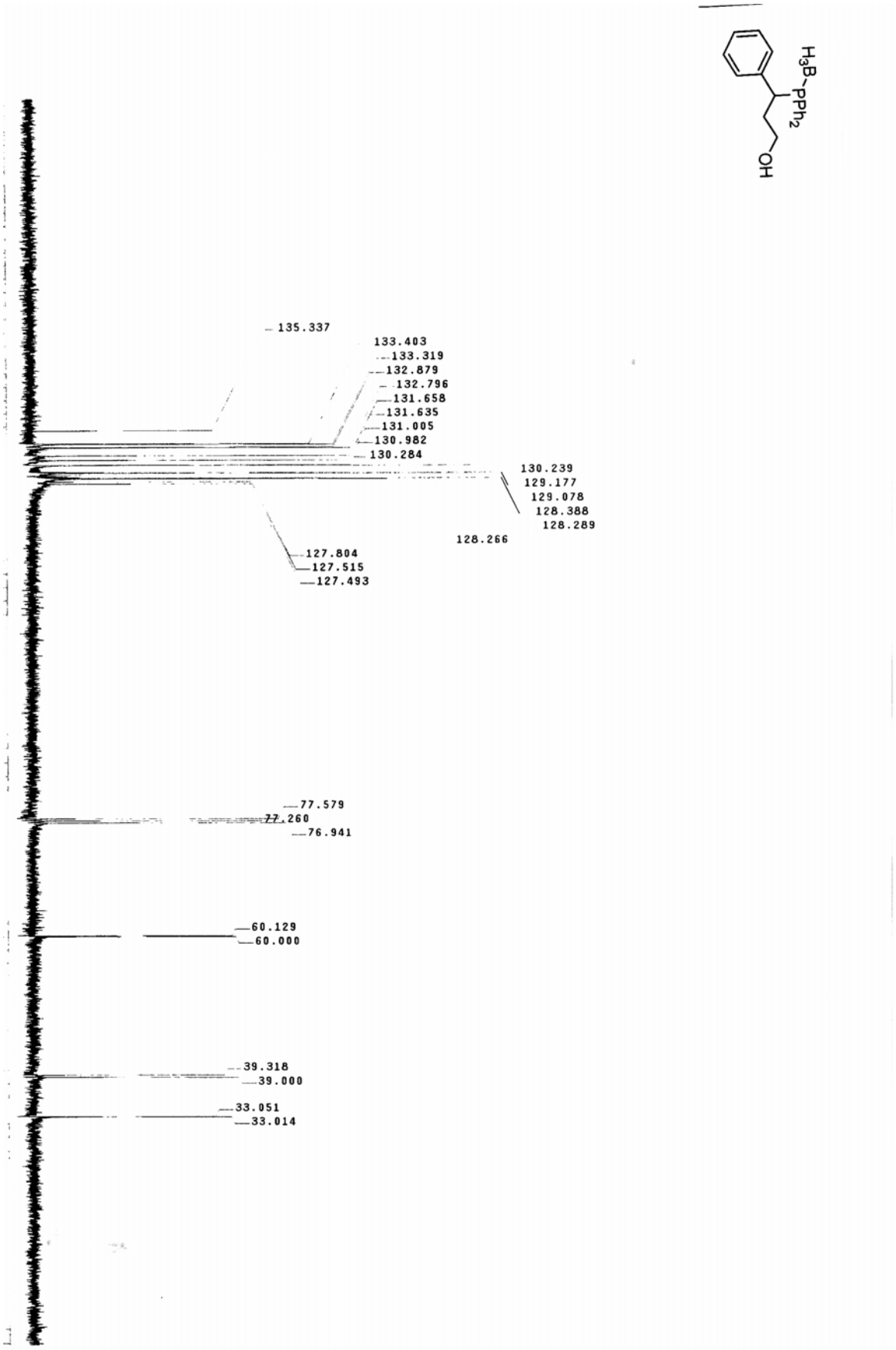
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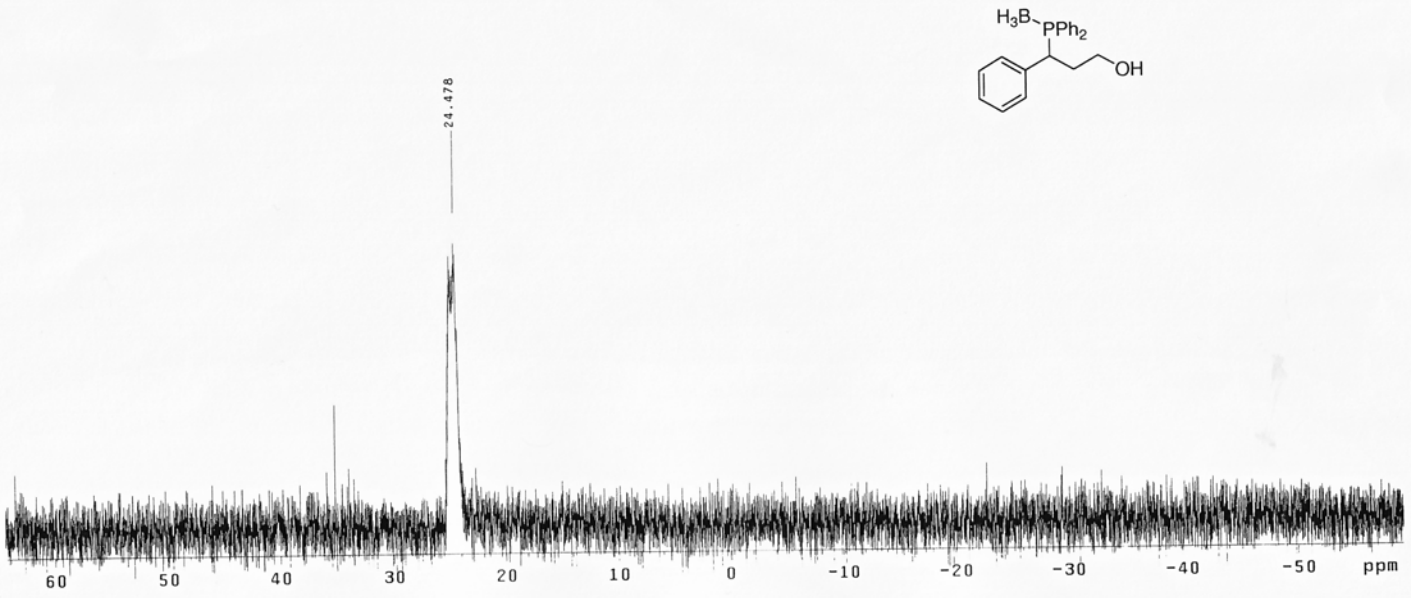

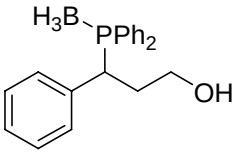
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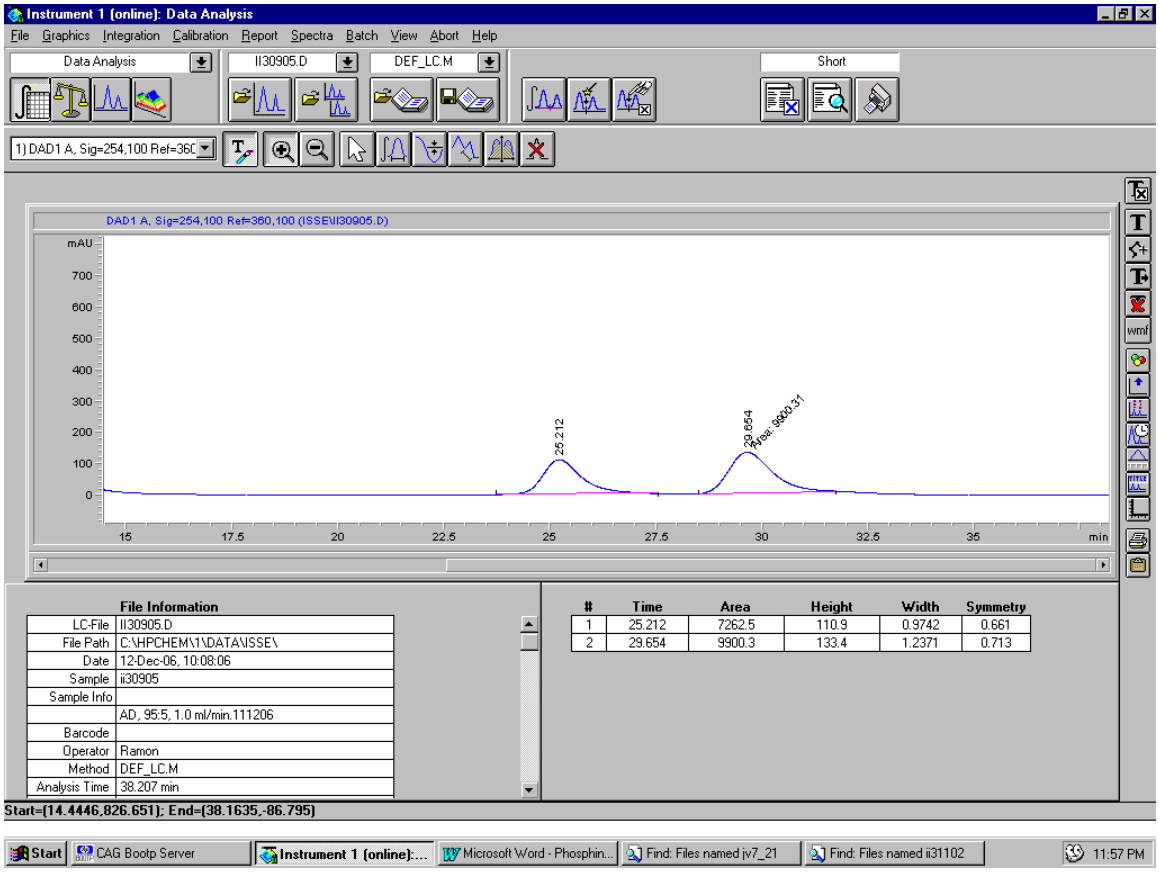
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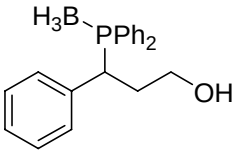
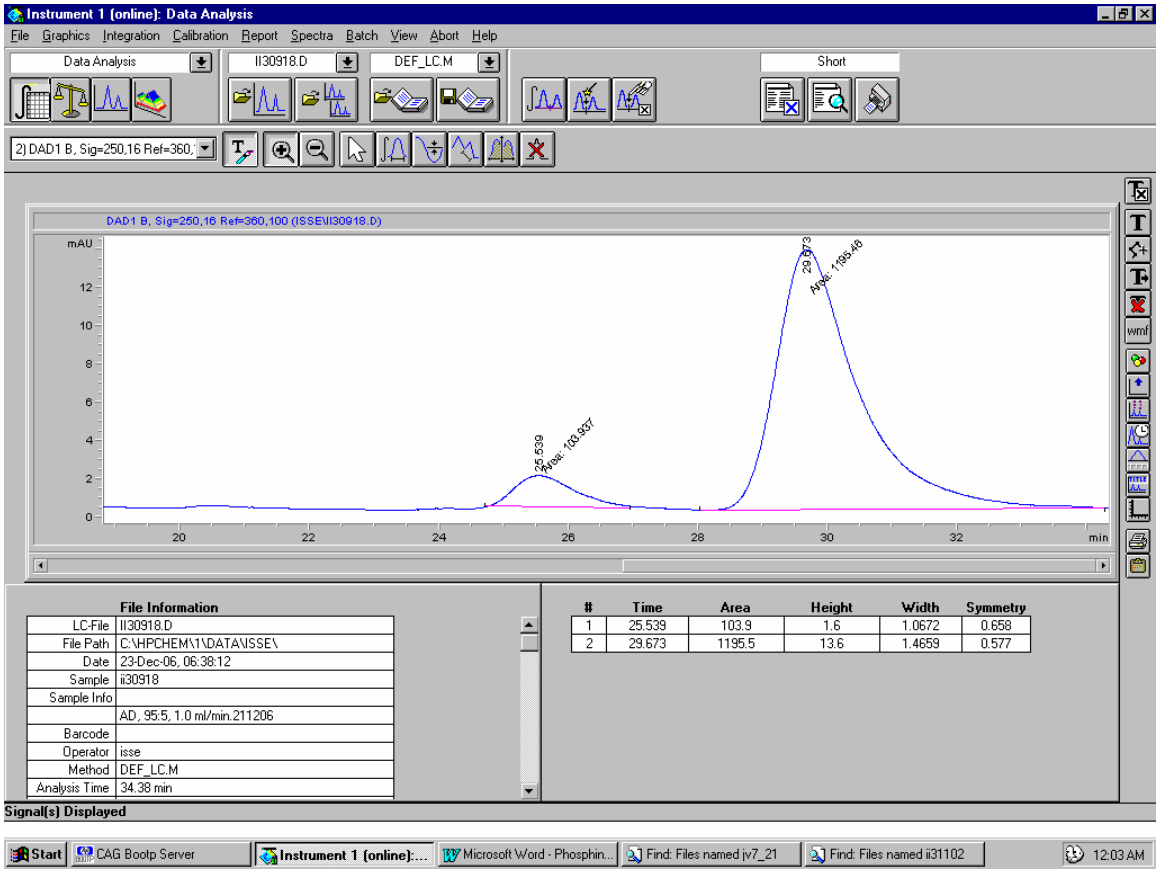
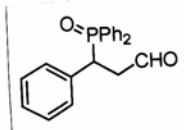
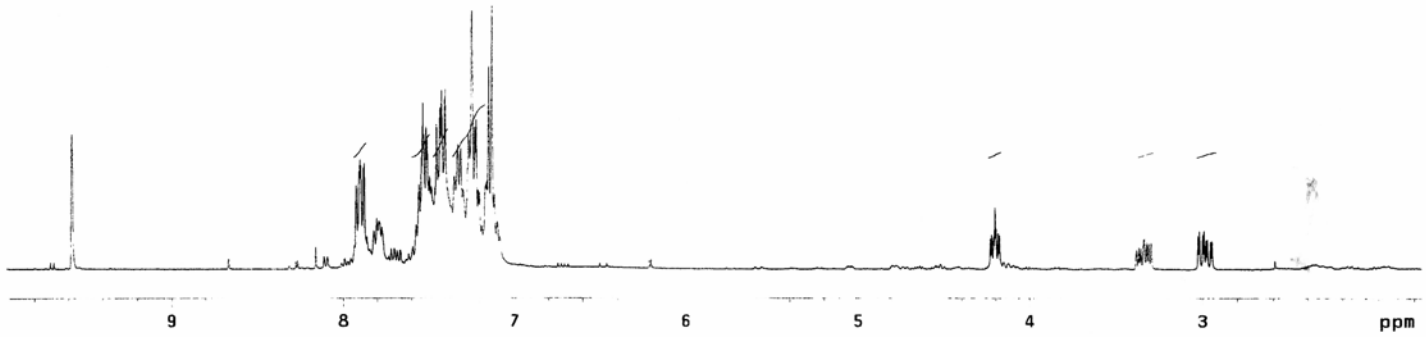
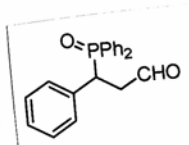
Find: Files named ii31102
11:57 PM

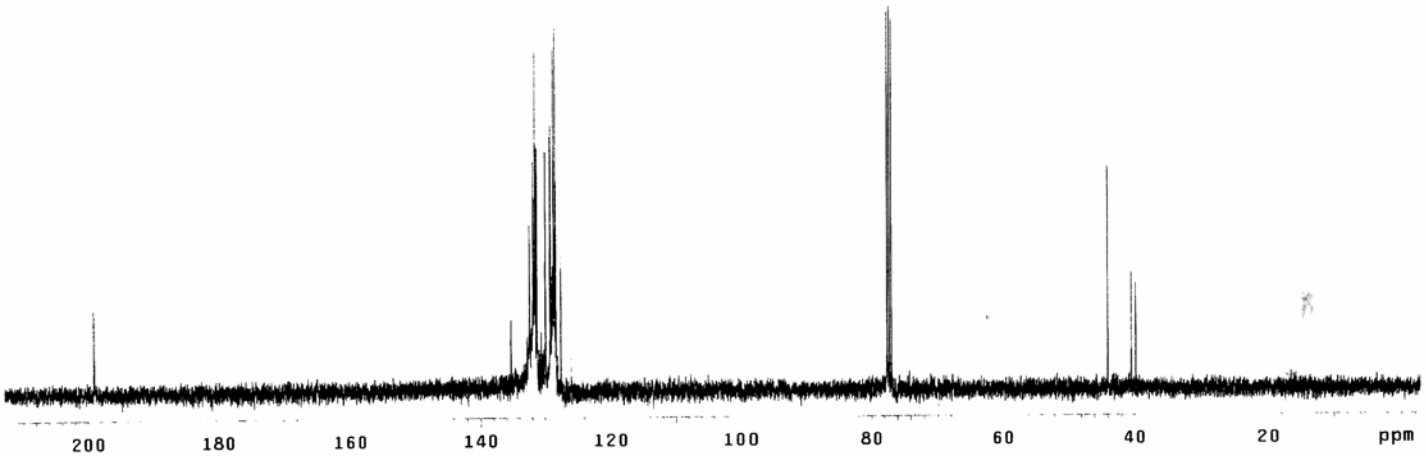

H3B
PPh2
OH


Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II30918.D
DEF_LC.M
Short
2) DAD1 B, Sig=250,16 Ref=360,
DAD1 B, Sig=250,16 Ref=360,100 (ISSE\II30918.D)
mAU
min
25.539
Area: 103.937
29.673
Area: 1195.48
File Information
LC-File II30918.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 23-Dec-06, 06:38:12
Sample ii30918
Sample Info
AD, 95:5, 1.0 ml/min.211206
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 34.38 min
Time Area Height Width Symmetry
1 25.539 103.9 1.6 1.0672 0.658
2 29.673 1195.5 13.6 1.4659 0.577
Signal(s) Displayed
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
Find: Files named ii31102
12:03 AM

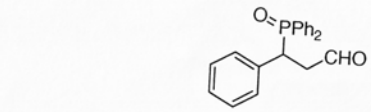

O=PPh2
CHO

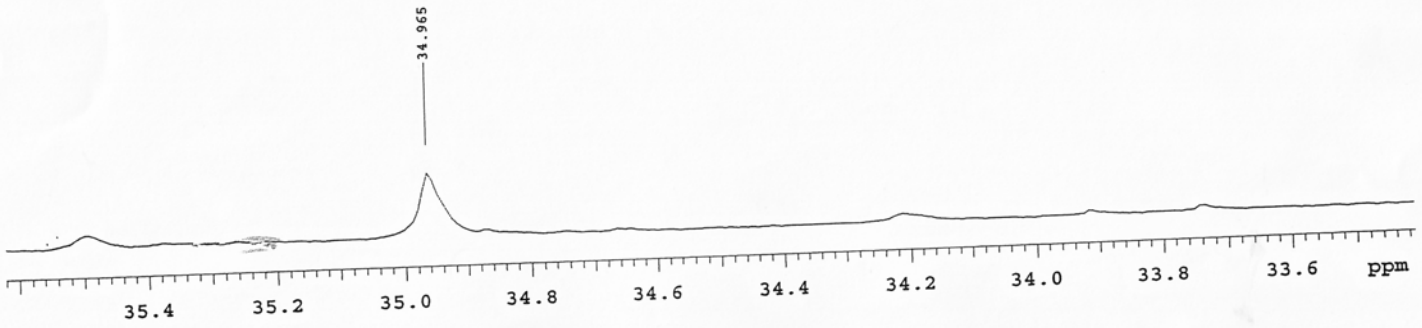

9
8
7
6
5
4
3
ppm


O=PPh2
CHO


200
180
160
140
120
100
80
60
40
20
ppm

INDEX	FREQUENCY	PPM	HEIGHT
1	5660.718	34.965	11.9

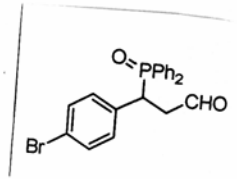


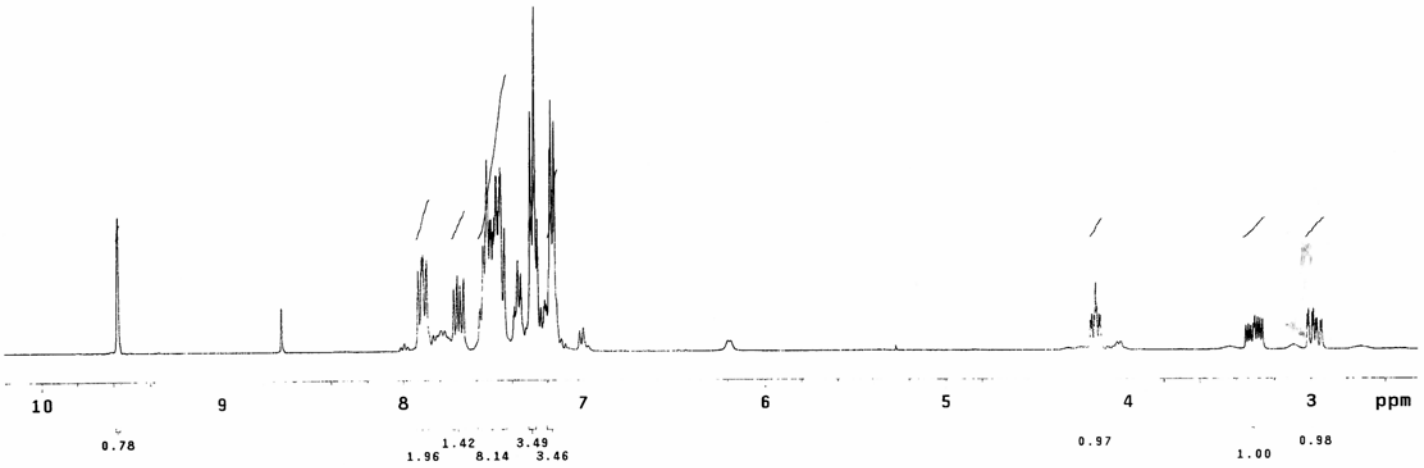


ii31105_flash_EtOAc

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.733 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 2 sec

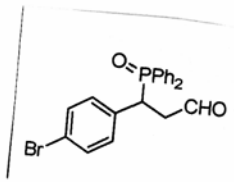


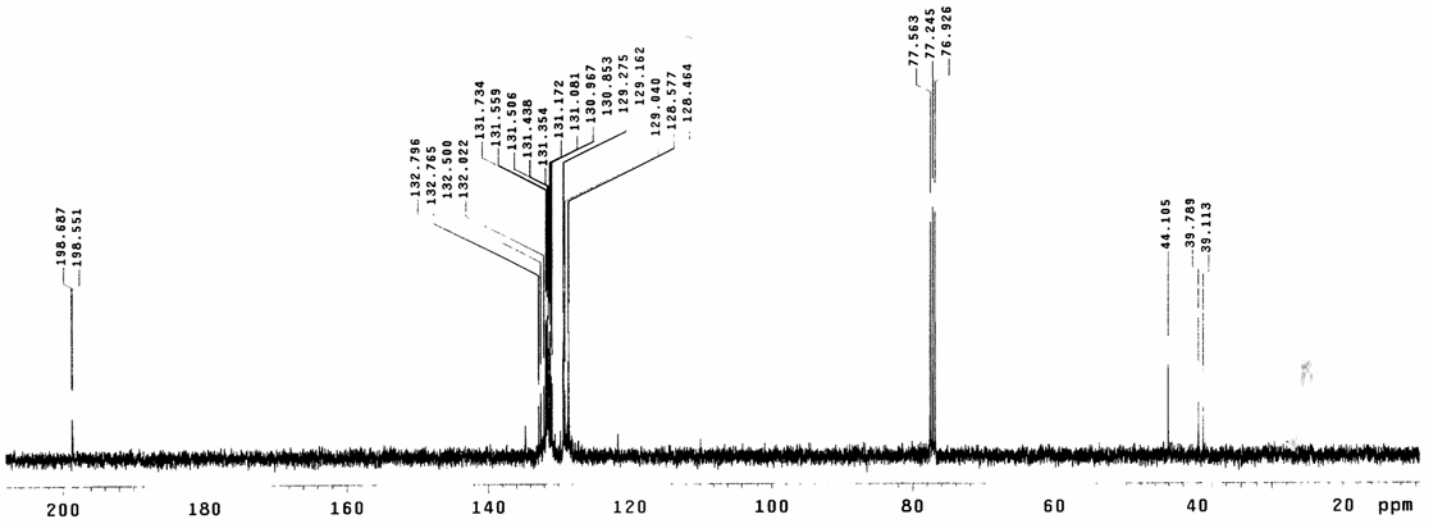


ii31209_flash_13C

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 48.9 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
128 repetitions
OBSERVE C13, 100.5638793 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 137 hr, 1 min, 31 sec

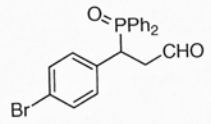


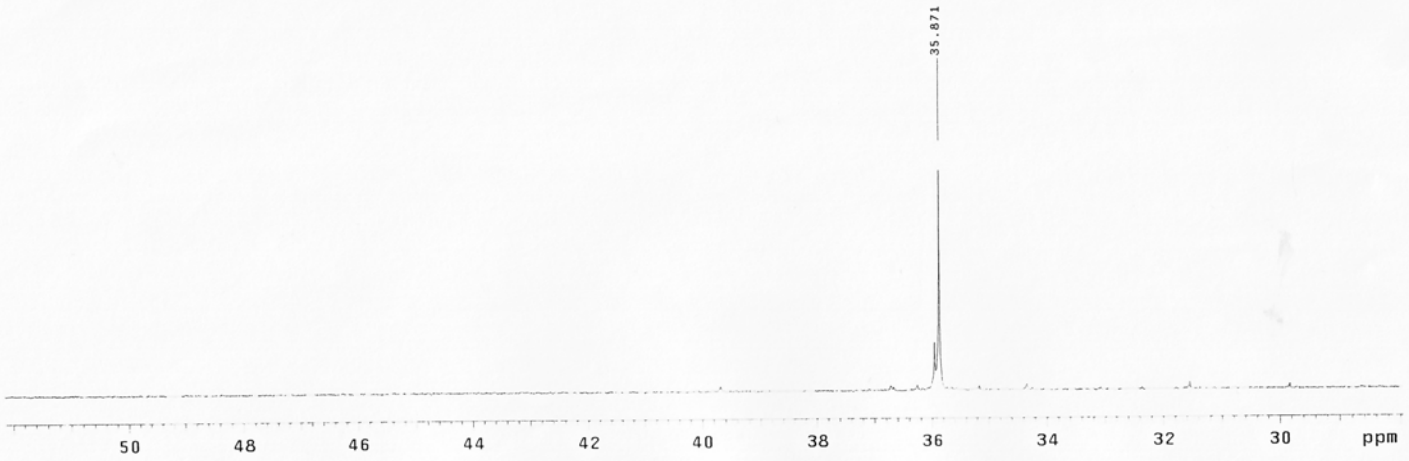


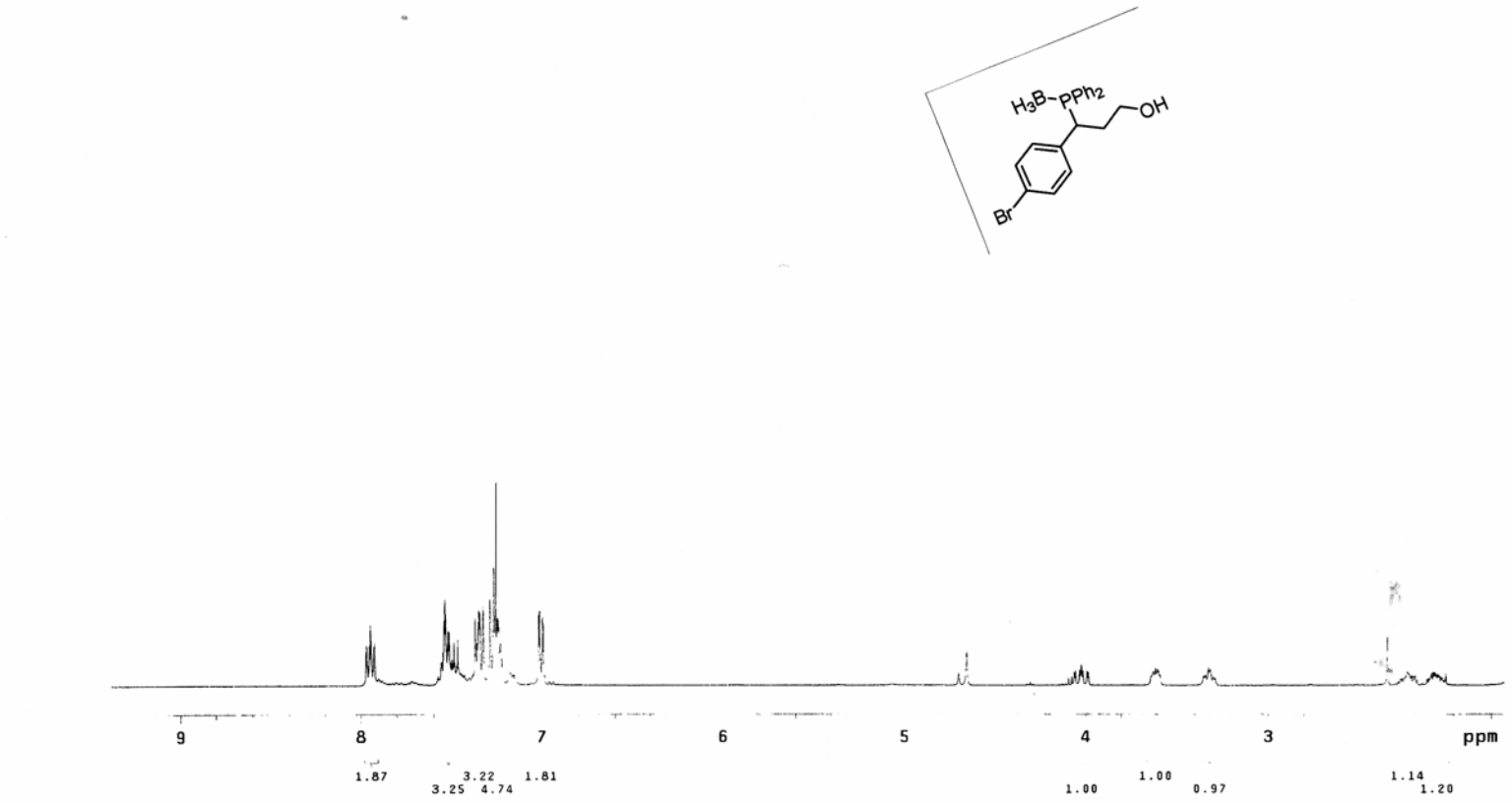
ii31106_flash_31P

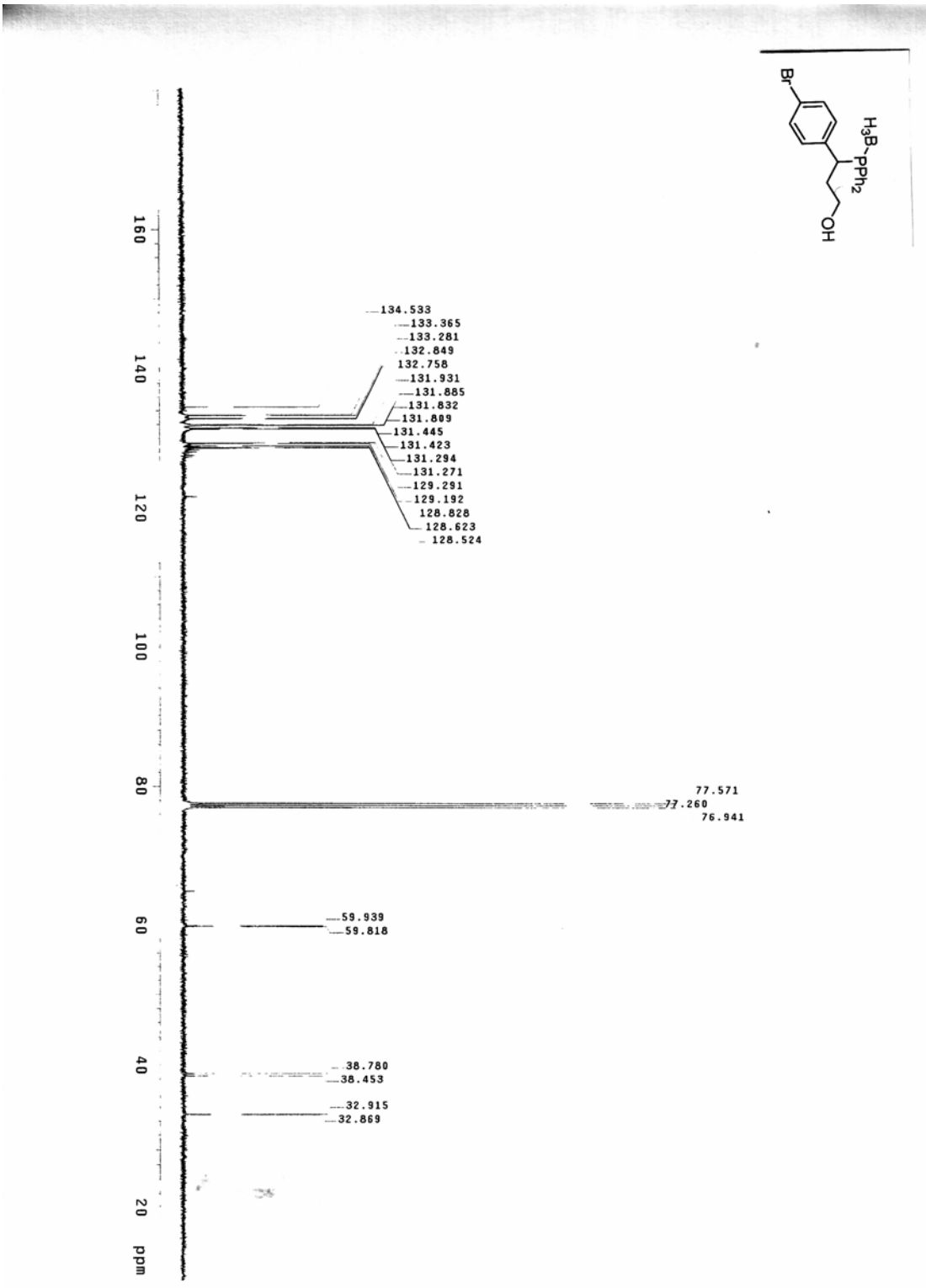
Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 25.0 C / 298.1 K
File: ii31106_flash_31P
Mercury-400BB "nmr4"

 Pulse 27.6 degrees
 Acq. time 0.800 sec
 Width 20000.0 Hz
 16 repetitions
OBSERVE P31, 161.8967379 MHz
DECOUPLE H1, 399.9376154 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 15 sec





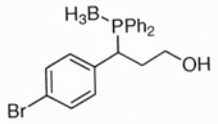

H3B-PPh2
OH
Br
9
8
7
6
5
4
3
ppm
1.87
3.25
3.22
4.74
1.81
1.00
1.00
0.97
1.14
1.20

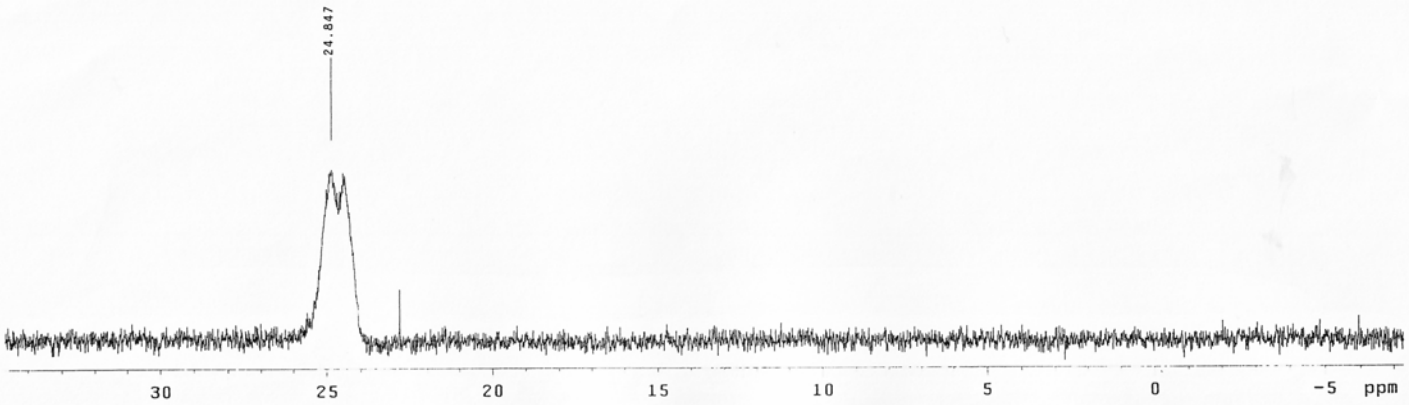

Br
H3B
PPh2
OH
134.533
133.365
133.281
132.849
132.758
131.931
131.885
131.832
131.809
131.445
131.423
131.294
131.271
129.291
129.192
128.828
128.623
128.524
77.571
77.260
76.941
59.939
59.818
38.780
38.453
32.915
32.869
160
140
120
100
80
60
40
20
ppm

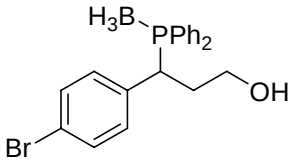
Pulse Sequence: s2pul

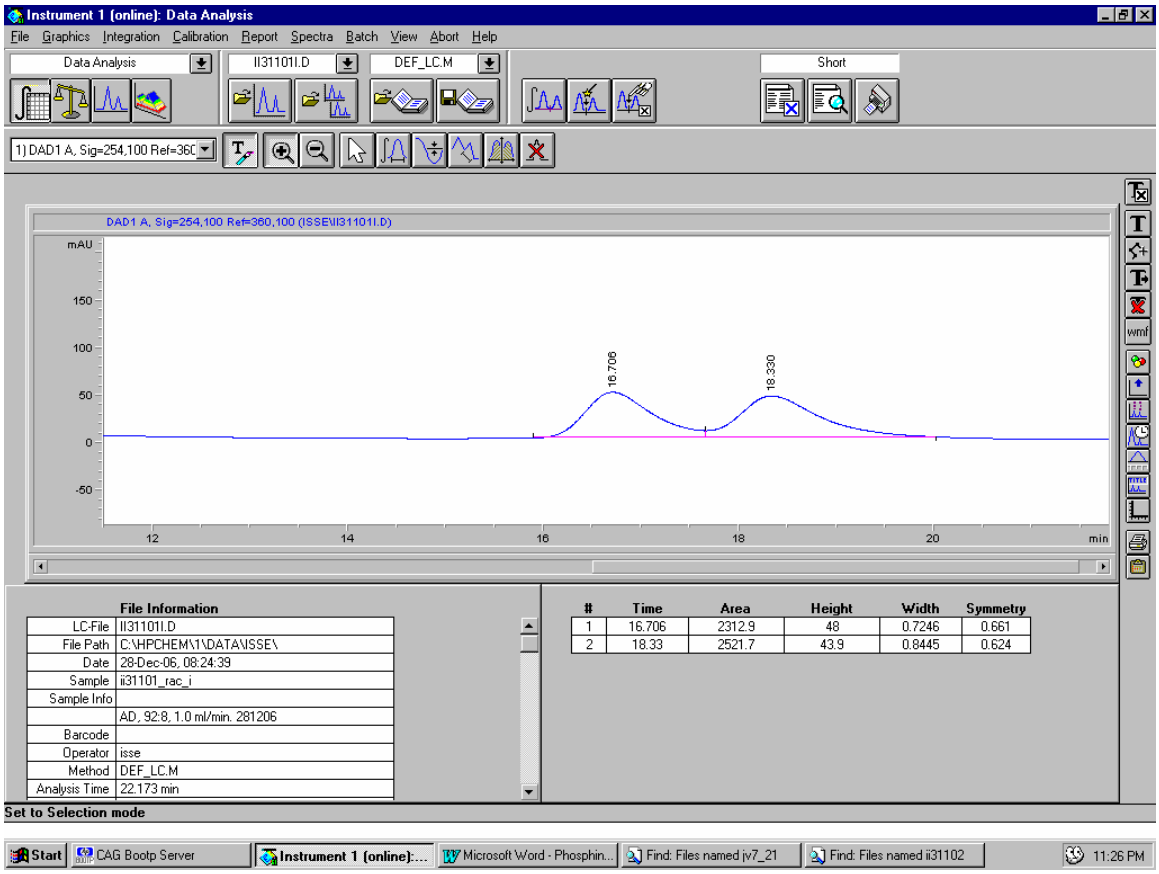
Solvent: CDCl3
Temp. 24.0 C / 297.1 K
Mercury-400BB "nmr4"

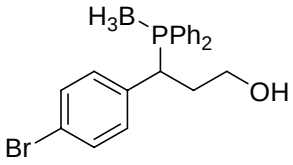
Pulse 27.6 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
30 repetitions
OBSERVE P31, 161.8967379 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 27 sec

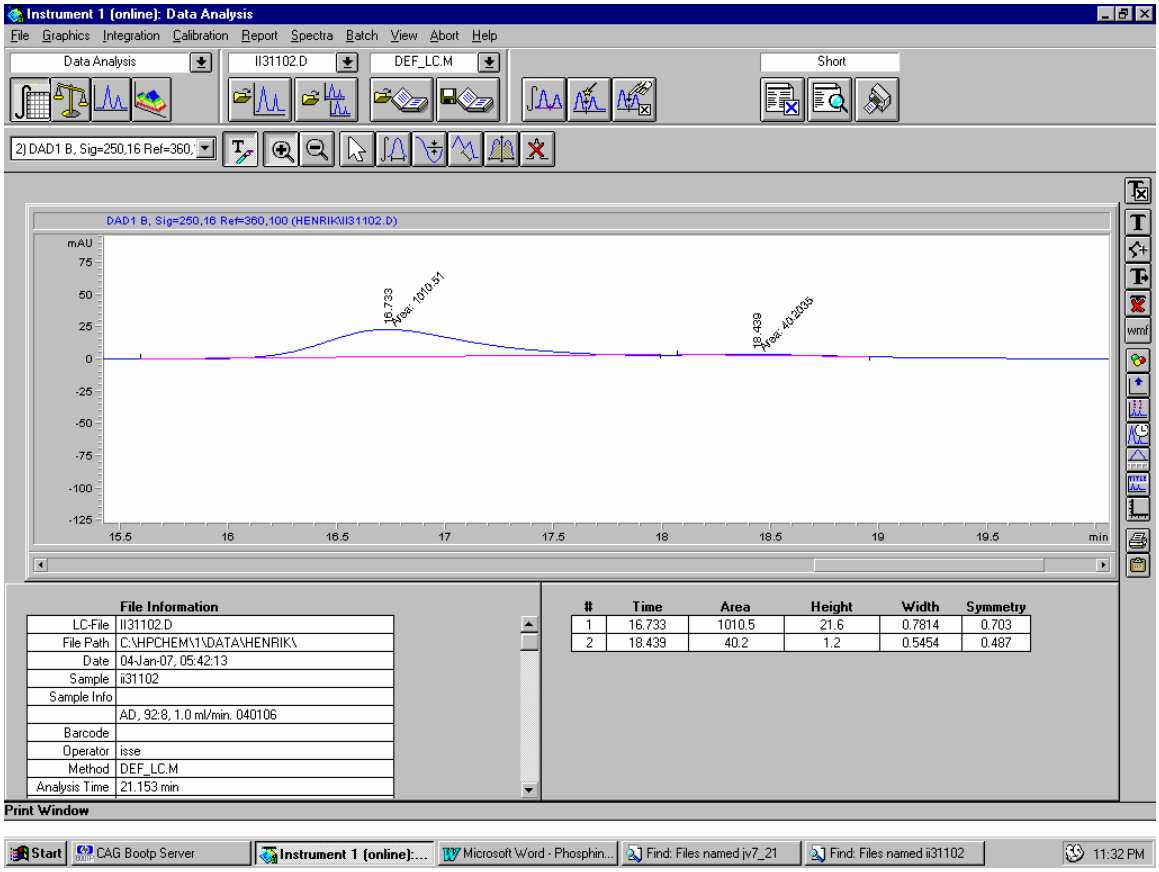


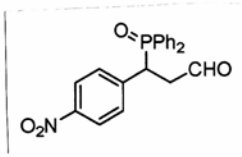


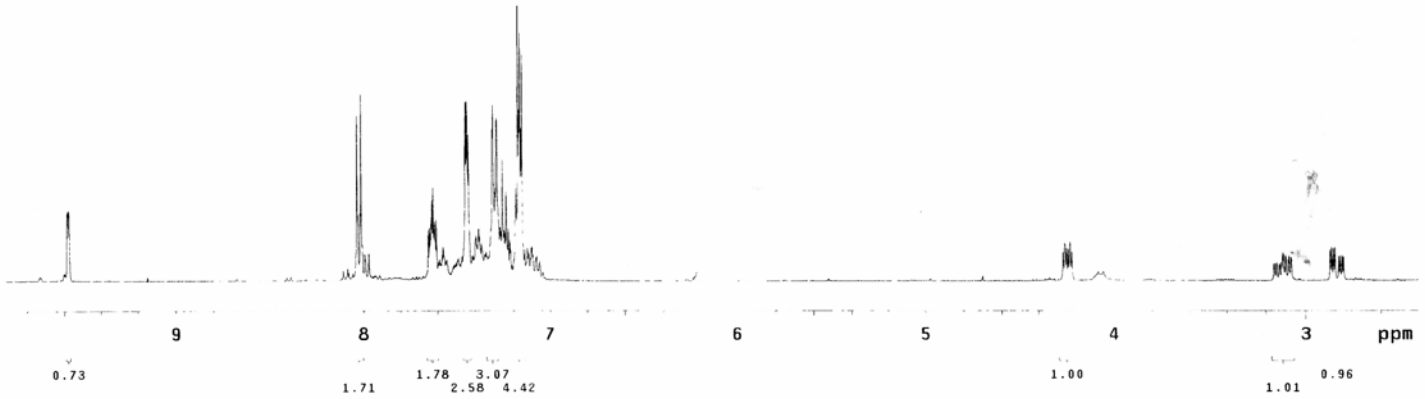

H3B
PPh2
OH
Br

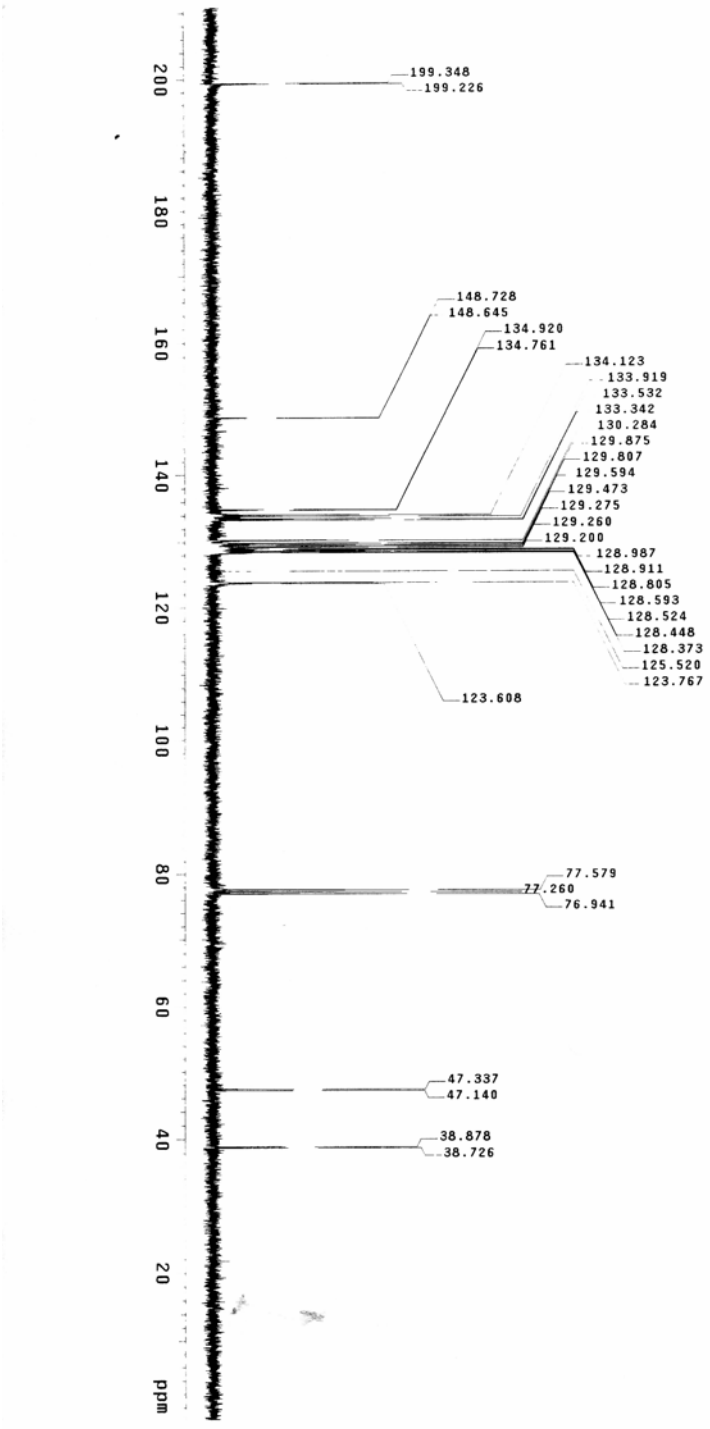

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II31101I.D
DEF_LC.M
Short
1) DAD1 A, Sig=254,100 Ref=360
DAD1 A, Sig=254,100 Ref=360,100 (ISSE\II31101I.D)
mAU
150
100
50
0
-50
16.706
18.330
12
14
16
18
20
min
File Information
LC-File II31101I.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 28-Dec-06, 08:24:39
Sample ii31101_rac_i
Sample Info
AD, 92:8, 1.0 ml/min. 281206
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 22.173 min
Time Area Height Width Symmetry
1 16.706 2312.9 48 0.7246 0.661
2 18.33 2521.7 43.9 0.8445 0.624
Set to Selection mode
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
Find: Files named ii31102
11:26 PM

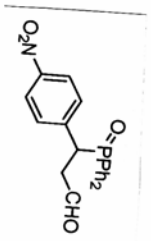

H3B
PPh2
OH
Br


Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II31102.D
DEF_LC.M
Short
2) DAD1 B, Sig=250,16 Ref=360,
DAD1 B, Sig=250,16 Ref=360,100 (HENRIK\II31102.D)
mAU
16.733
Area: 1010.51
18.439
Area: 40.2035
min
File Information
LC-File II31102.D
File Path C:\HPCHEM\1\DATA\HENRIK\
Date 04-Jan-07, 05:42:13
Sample ii31102
Sample Info
AD, 92:8, 1.0 ml/min. 040106
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 21.153 min
Time Area Height Width Symmetry
1 16.733 1010.5 21.6 0.7814 0.703
2 18.439 40.2 1.2 0.5454 0.487
Print Window
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
Find: Files named ii31102
11:32 PM


O=PPh2
CHO
O2N


9
8
7
6
5
4
3
ppm
0.73
1.71
1.78
3.07
2.58
4.42
1.00
1.01
0.96


199.348
199.226
148.728
148.645
134.920
134.761
134.123
133.919
133.532
133.342
130.284
129.875
129.807
129.594
129.473
129.275
129.260
129.200
128.987
128.911
128.805
128.593
128.524
128.448
128.373
125.520
123.767
123.608
77.579
77.260
76.941
47.337
47.140
38.878
38.726
200
180
160
140
120
100
80
60
40
20
ppm

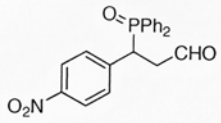

O2N
O
PPh2
CHO

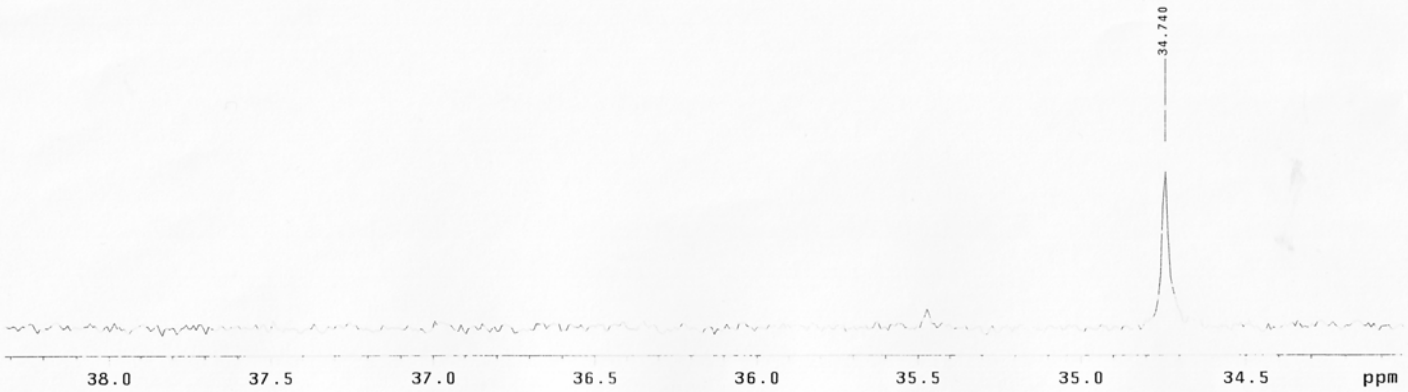
ii31004_P

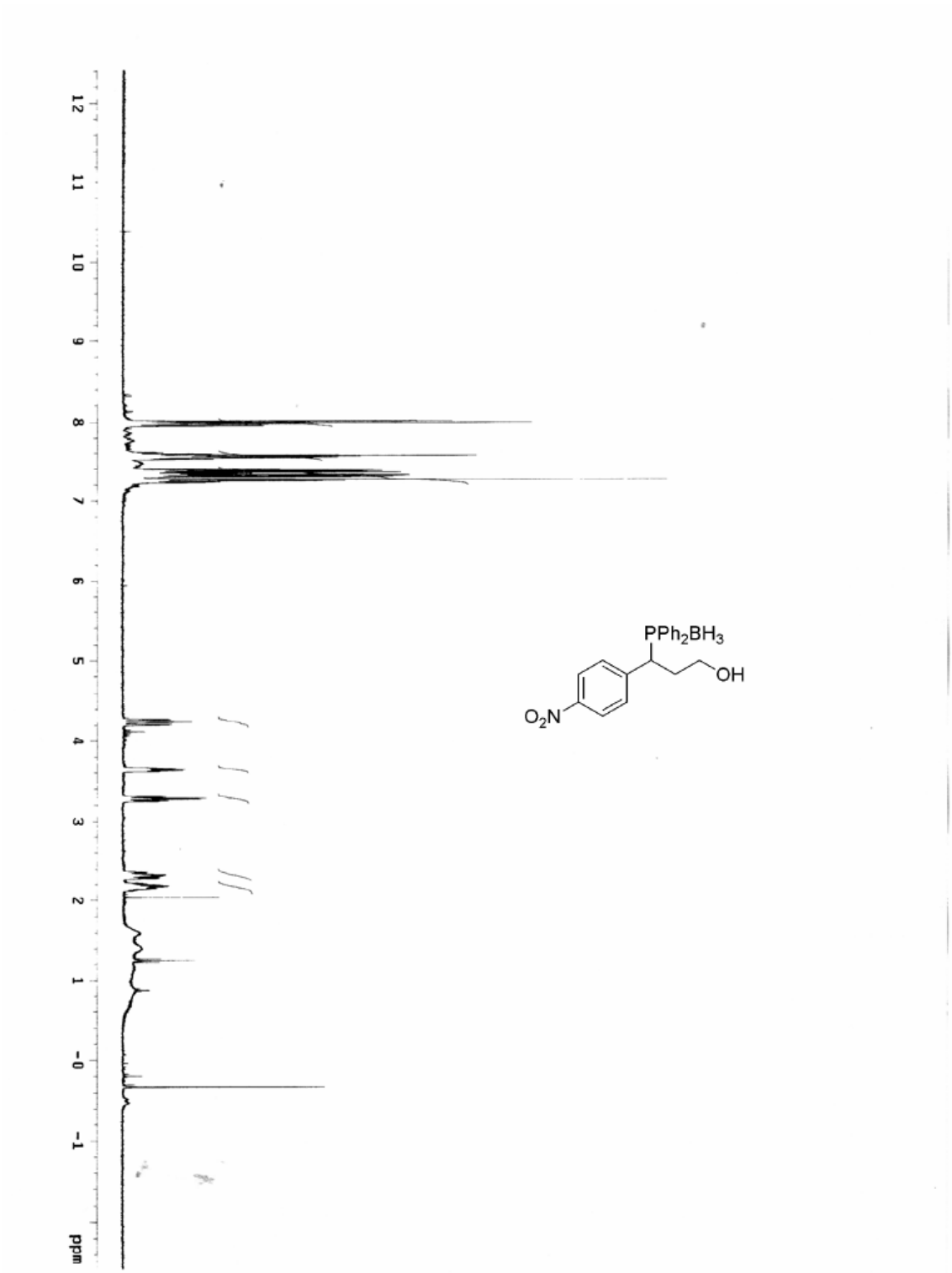
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: ii31004_P31
Mercury-400BB "nmr4"

Pulse 27.6 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
16 repetitions
OBSERVE P31, 161.8967379 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 0 min, 15 sec





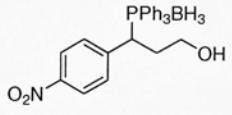

PPh2BH3
OH
O2N
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

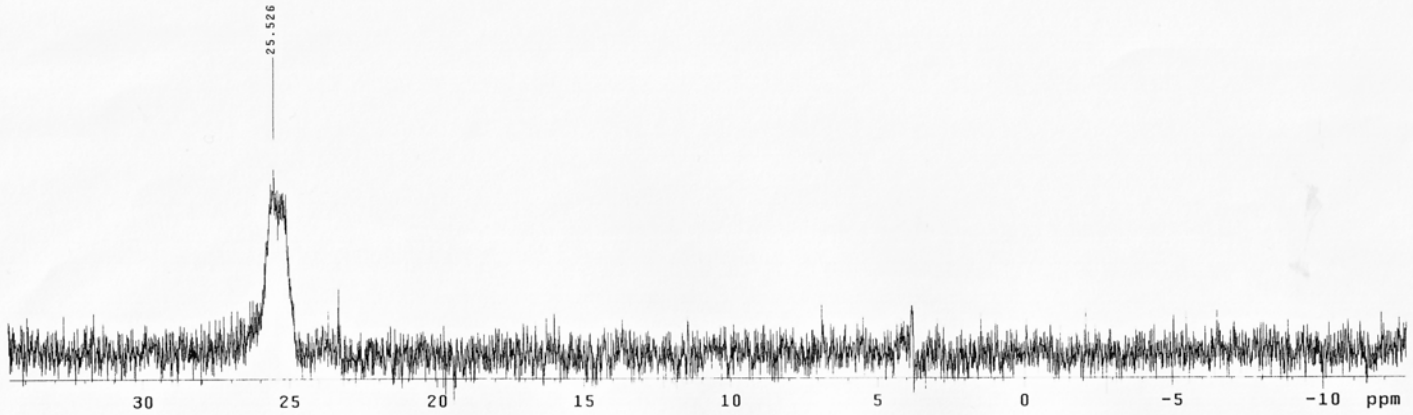
ii31002_P31

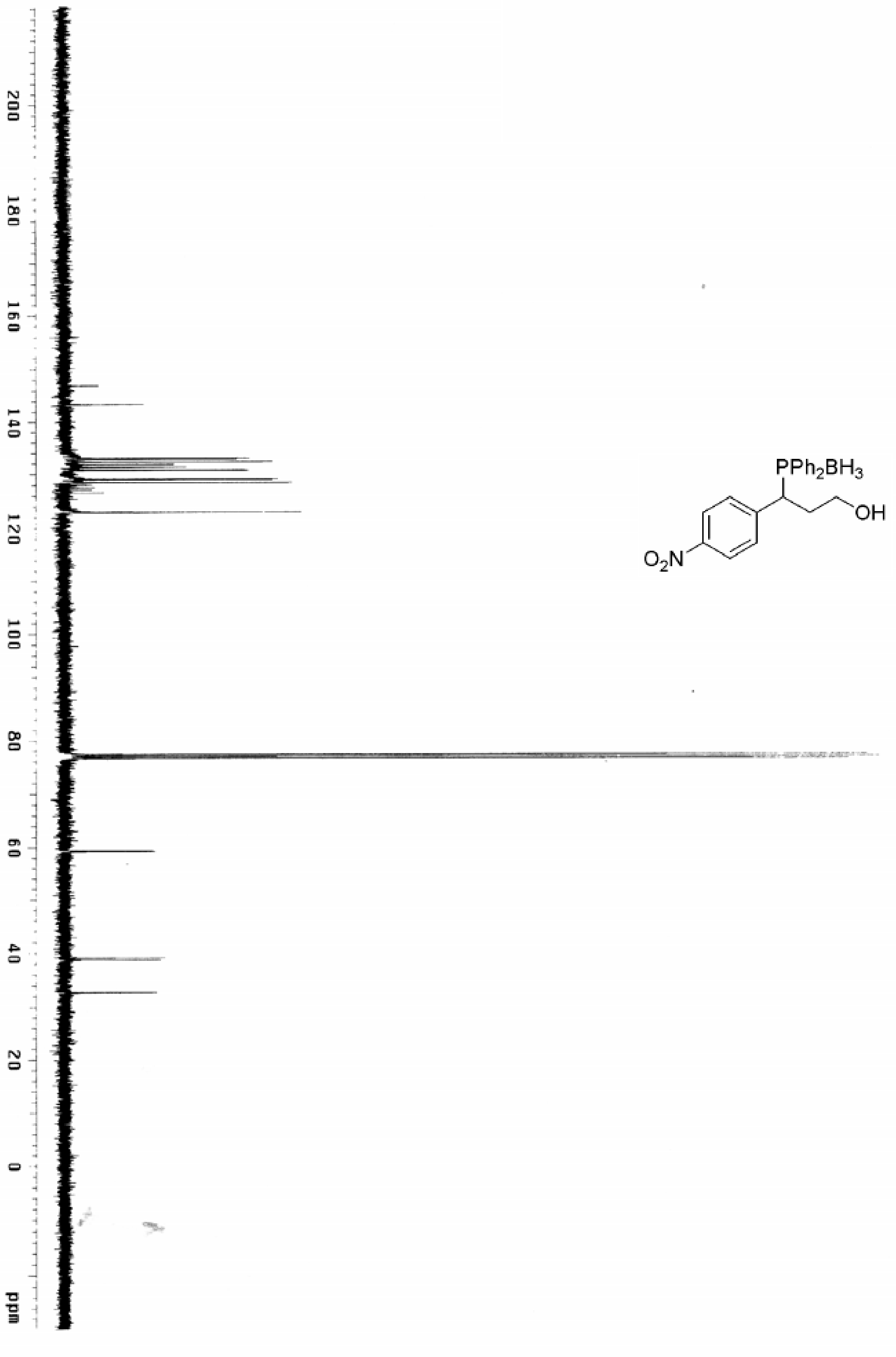
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: ii31002_P31
Mercury-400BB "nmr4"

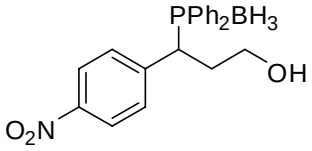
Pulse 27.6 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
128 repetitions
OBSERVE P31, 161.8967379 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 4 hr, 48 min, 3 sec

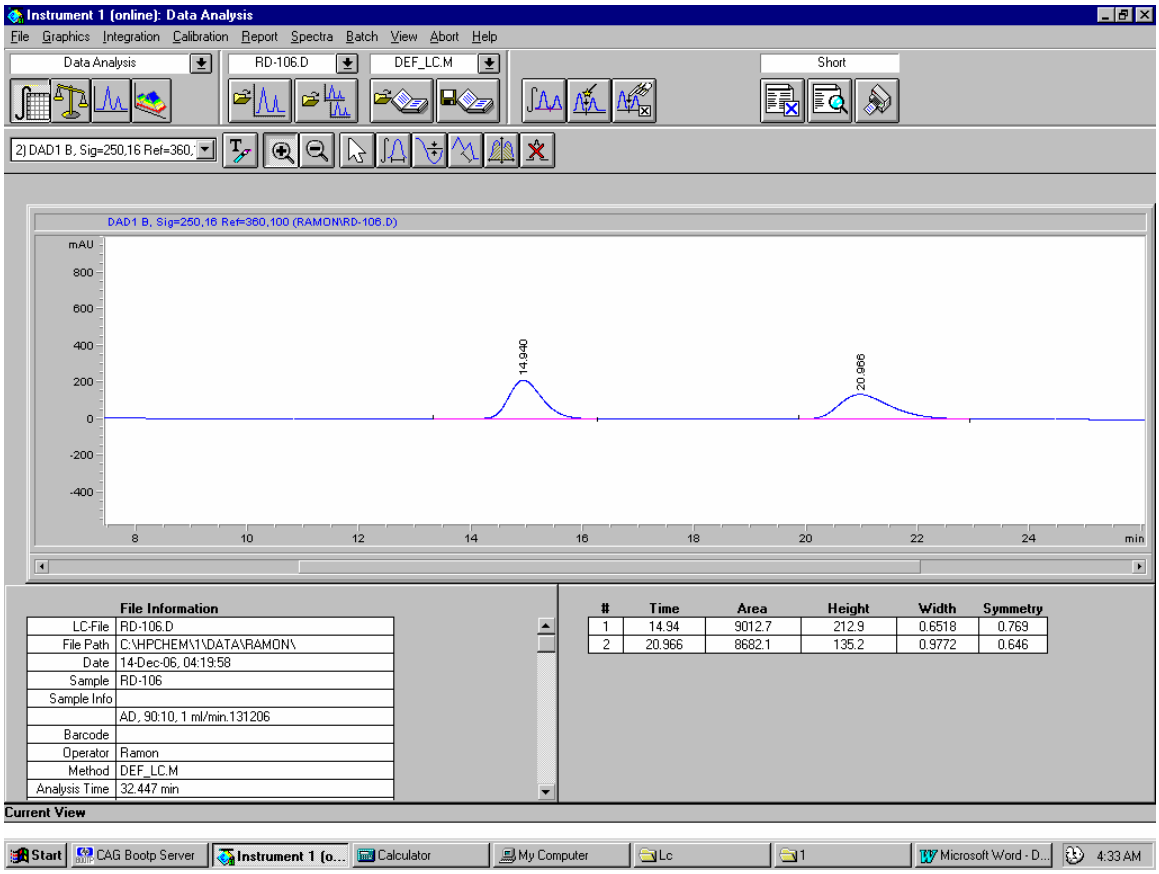


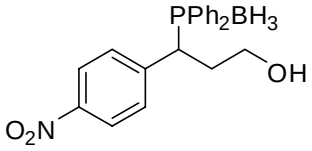


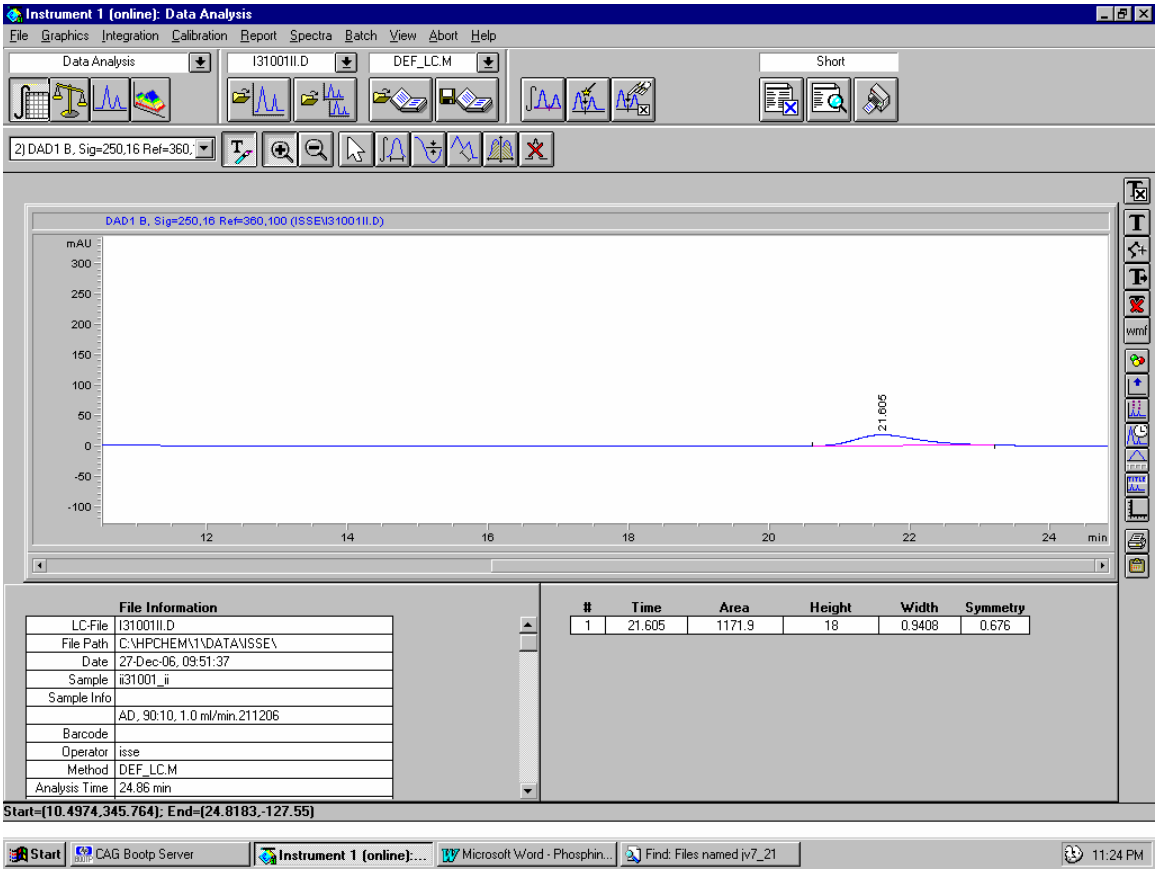


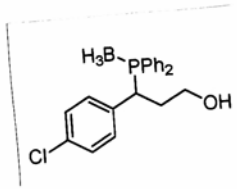
200
180
160
140
120
100
80
60
40
20
0
ppm
PPh2BH3
OH
O2N

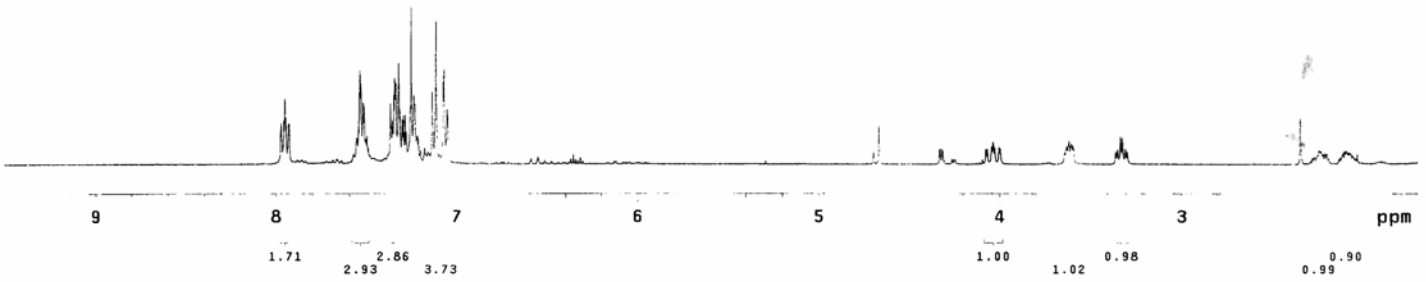

PPh2BH3
OH
O2N

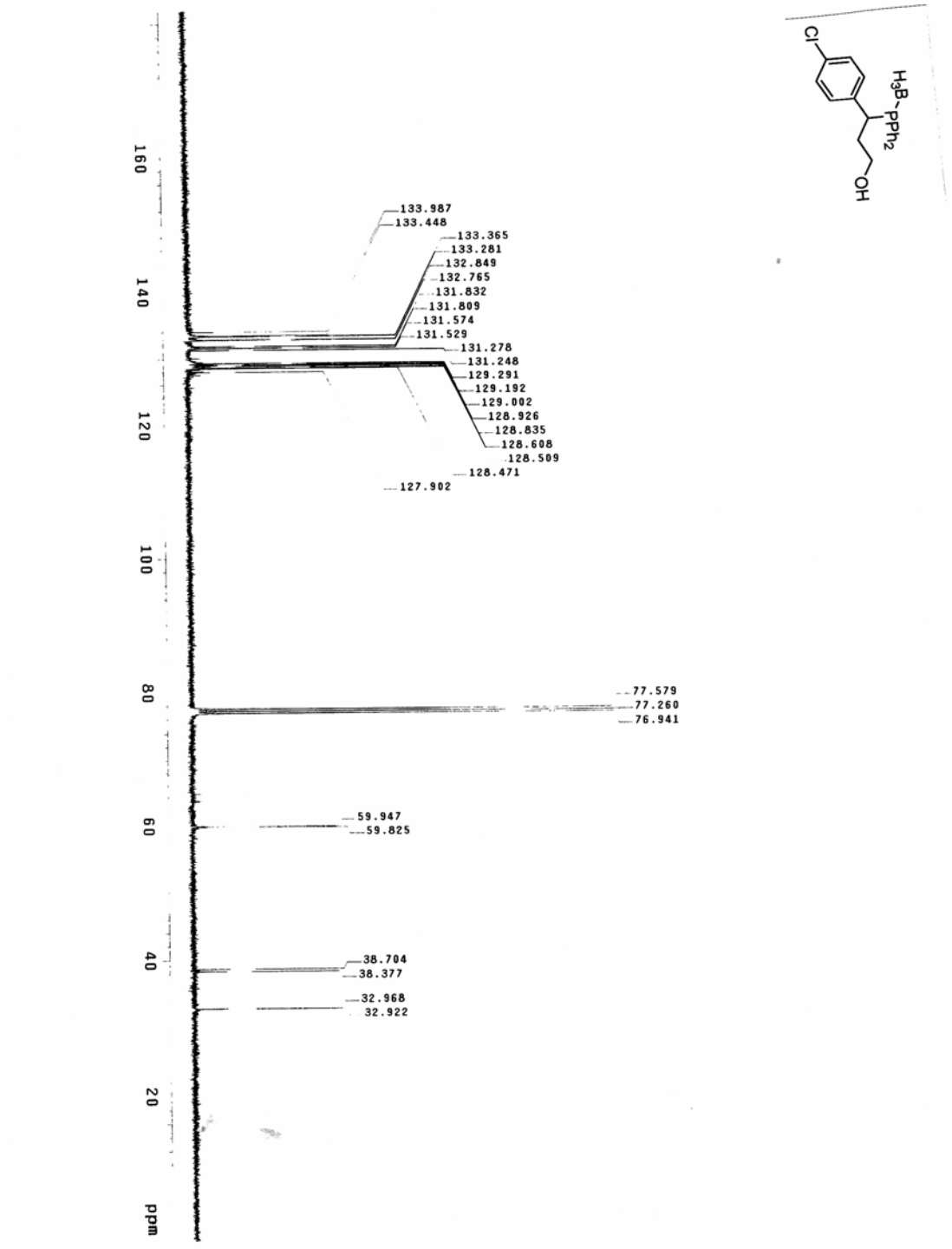

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
RD-106.D
DEF_LC.M
Short
2) DAD1 B, Sig=250,16 Ref=360,
DAD1 B, Sig=250,16 Ref=360,100 (RAMON\RD-106.D)
mAU
800
600
400
200
0
-200
-400
14.940
20.966
8
10
12
14
16
18
20
22
24
min
File Information
LC-File RD-106.D
File Path C:\HPCHEM\1\DATA\RAMON\
Date 14-Dec-06, 04:19:58
Sample RD-106
Sample Info
AD, 90:10, 1 ml/min.131206
Barcode
Operator Ramon
Method DEF_LC.M
Analysis Time 32.447 min
Time Area Height Width Symmetry
1 14.94 9012.7 212.9 0.6518 0.769
2 20.966 8682.1 135.2 0.9772 0.646
Current View
Start
CAG Bootp Server
Instrument 1 (o...
Calculator
My Computer
Lc
1
Microsoft Word - D...
4:33 AM


PPh2BH3
OH
O2N

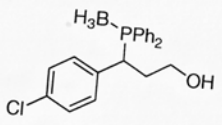

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
I31001II.D
DEF_LC.M
Short
2) DAD1 B, Sig=250,16 Ref=360,
DAD1 B, Sig=250,16 Ref=360,100 (ISSE\I31001II.D)
mAU
300
250
200
150
100
50
0
-50
-100
21.605
12
14
16
18
20
22
24
min
File Information
LC-File I31001II.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 27-Dec-06, 09:51:37
Sample ii31001_ii
Sample Info
AD, 90:10, 1.0 ml/min.211206
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 24.86 min
Time Area Height Width Symmetry
1 21.605 1171.9 18 0.9408 0.676
Start=(10.4974,345.764); End=(24.8183,-127.55)
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
11:24 PM

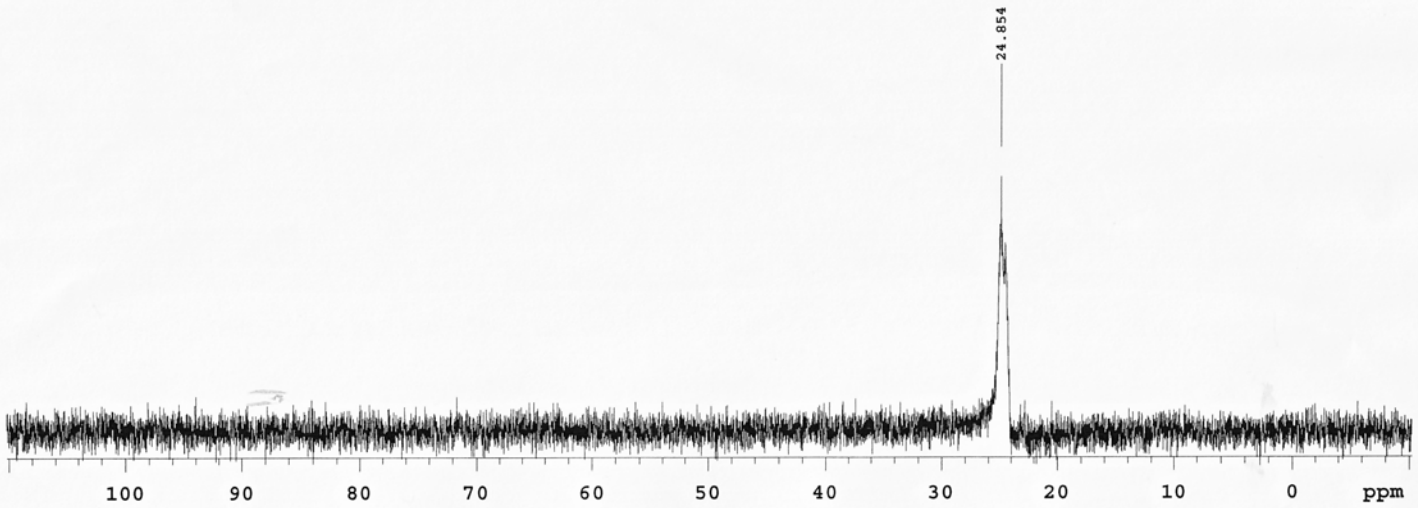

H3B
PPh2
OH
Cl

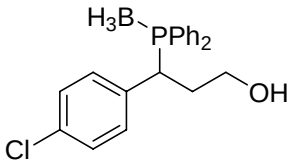

9
8
7
6
5
4
3
ppm
1.71
2.93
2.86
3.73
1.00
1.02
0.98
0.99
0.90

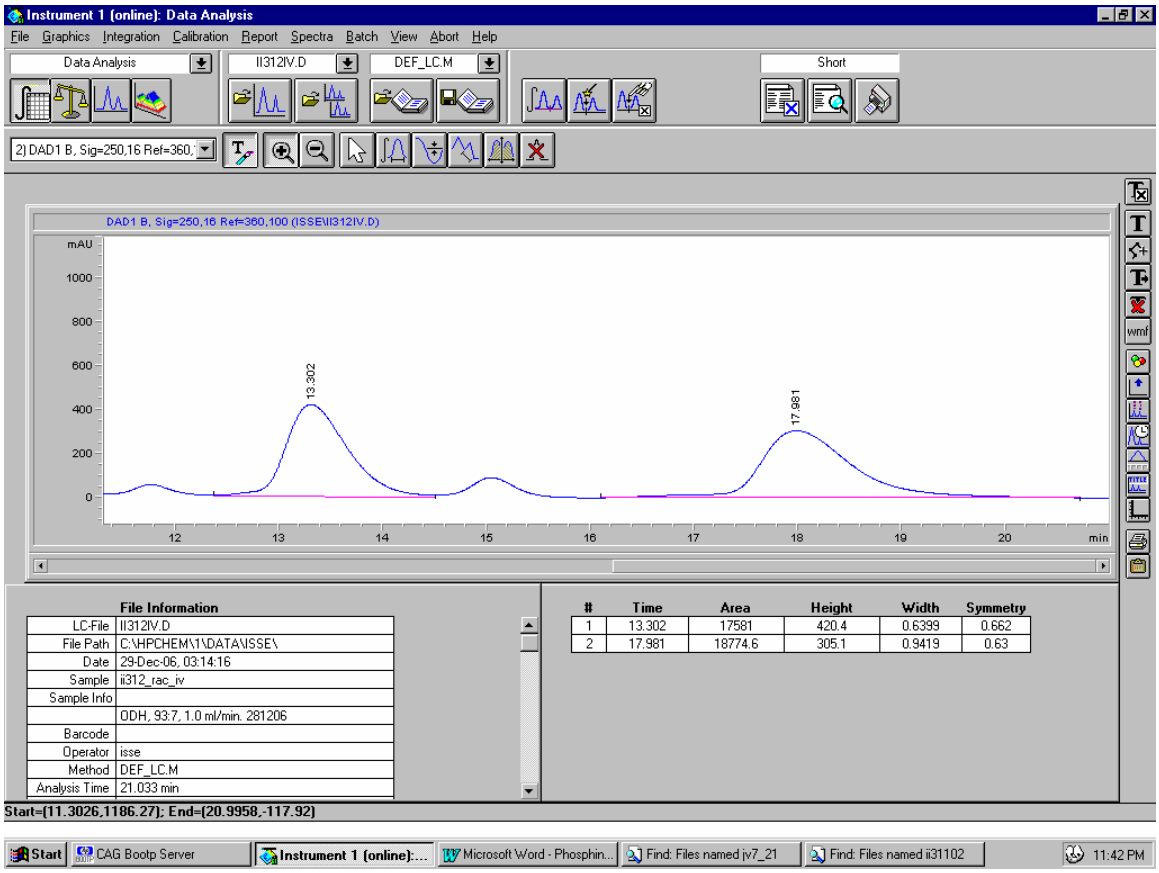

Cl
H3B-PPh2
OH
133.987
133.448
133.365
133.281
132.849
132.765
131.832
131.809
131.574
131.529
131.278
131.248
129.291
129.192
129.002
128.926
128.835
128.608
128.509
128.471
127.902
77.579
77.260
76.941
59.947
59.825
38.704
38.377
32.968
32.922
160
140
120
100
80
60
40
20
ppm

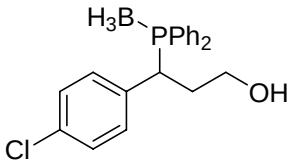
INDEX	FREQUENCY	PPM	HEIGHT
1	4023.730	24.854	45.4

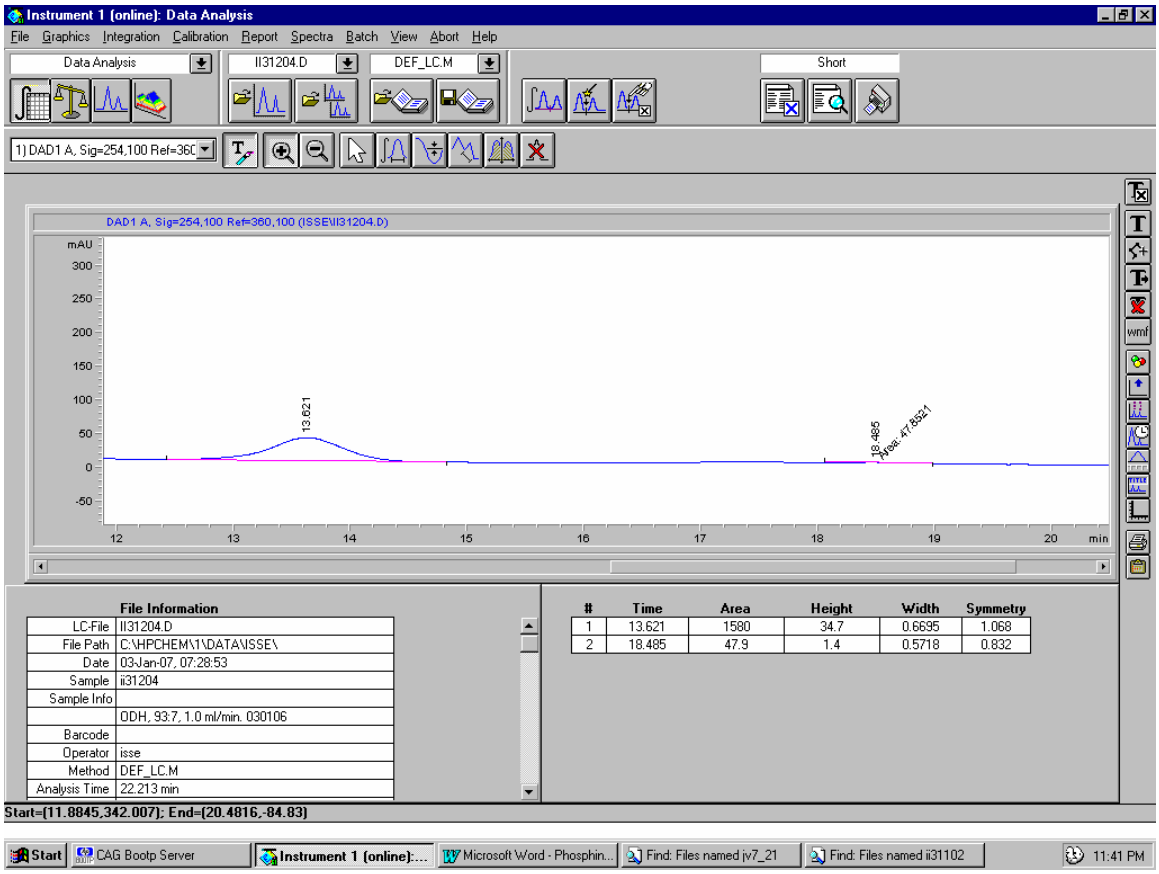


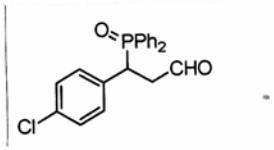


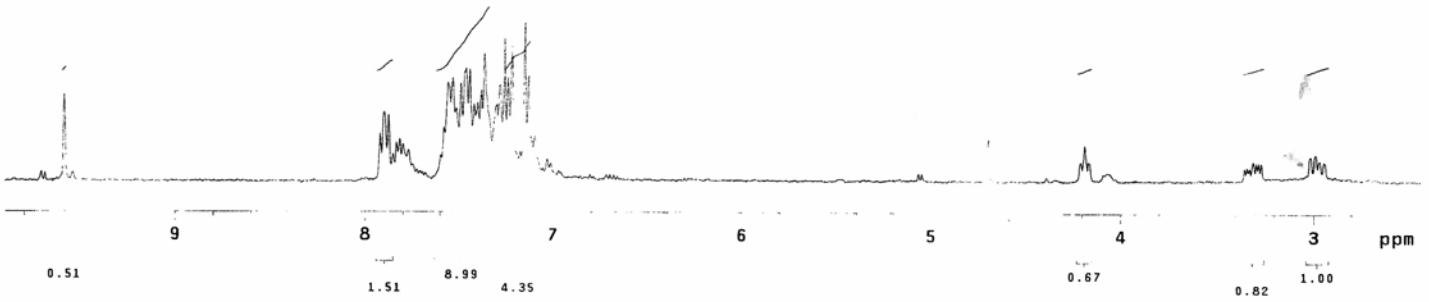

H3B
PPh2
OH
Cl

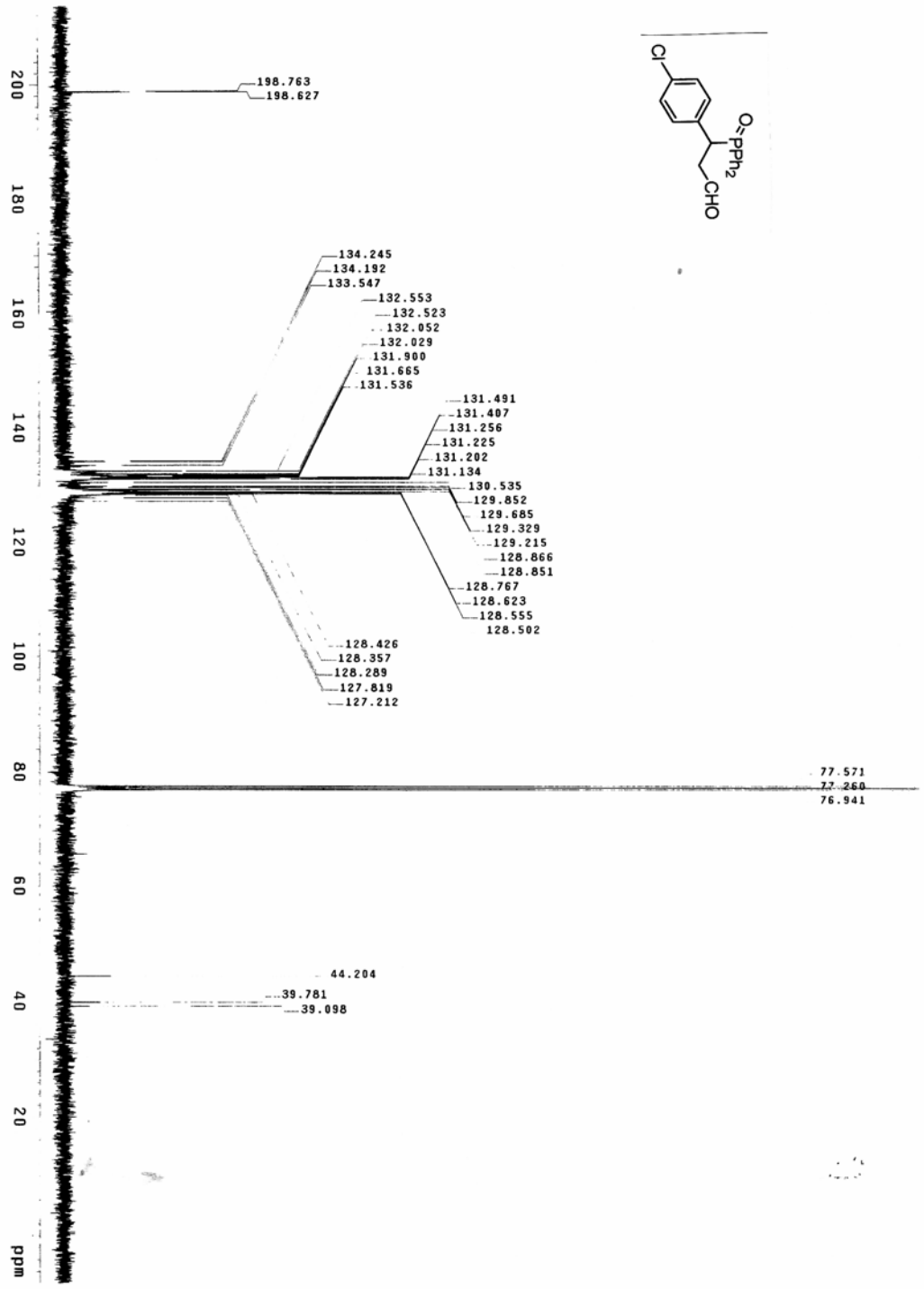

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II312IV.D
DEF_LC.M
Short
2) DAD1 B, Sig=250,16 Ref=360,
DAD1 B, Sig=250,16 Ref=360,100 (ISSE\II312IV.D)
mAU
1000
800
600
400
200
0
13.302
17.981
12
13
14
15
16
17
18
19
20
min
File Information
LC-File II312IV.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 29-Dec-06, 03:14:16
Sample ii312_rac_iv
Sample Info
ODH, 93:7, 1.0 ml/min. 281206
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 21.033 min
Time Area Height Width Symmetry
1 13.302 17581 420.4 0.6399 0.662
2 17.981 18774.6 305.1 0.9419 0.63
Start=(11.3026,1186.27); End=(20.9958,-117.92)
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
Find: Files named ii31102
11:42 PM


H3B
PPh2
OH
Cl

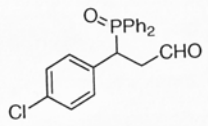

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II31204.D
DEF_LC.M
Short
1) DAD1 A, Sig=254,100 Ref=360
DAD1 A, Sig=254,100 Ref=360,100 (ISSE\II31204.D)
mAU
300
250
200
150
100
50
0
-50
13.621
18.485
Area: 47.8521
12
13
14
15
16
17
18
19
20
min
wmf
File Information
LC-File II31204.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 03-Jan-07, 07:28:53
Sample ii31204
Sample Info
ODH, 93:7, 1.0 ml/min. 030106
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 22.213 min
Time Area Height Width Symmetry
1 13.621 1580 34.7 0.6695 1.068
2 18.485 47.9 1.4 0.5718 0.832
Start=(11.8845,342.007); End=(20.4816,-84.83)
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
Find: Files named ii31102
11:41 PM

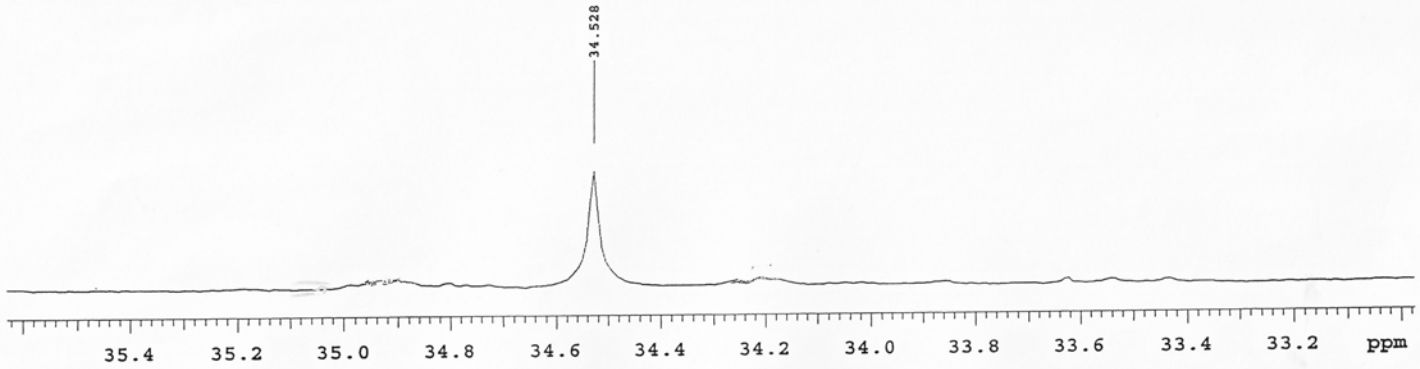

O
PPh2
CHO
Cl

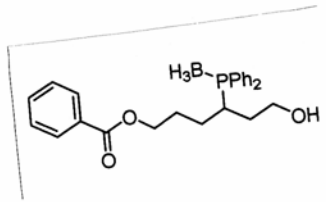

9
8
7
6
5
4
3
ppm
0.51
1.51
8.99
4.35
0.67
0.82
1.00

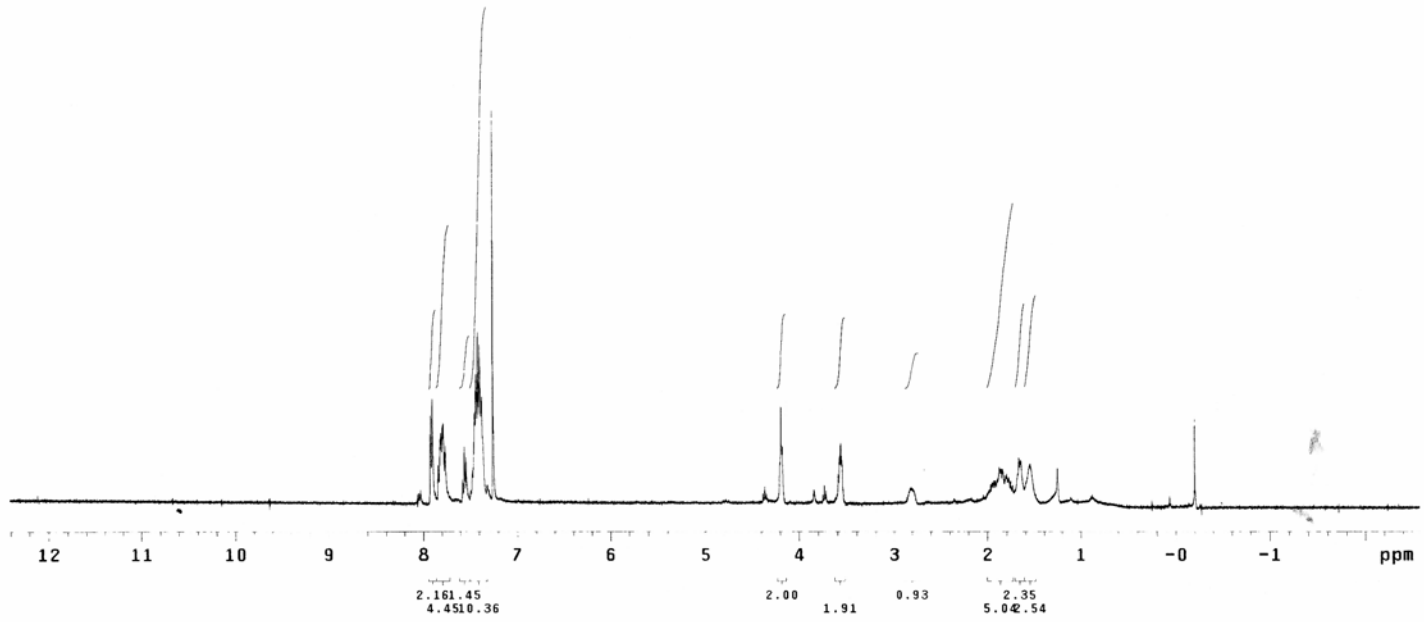

198.763
198.627
134.245
134.192
133.547
132.553
132.523
132.052
132.029
131.900
131.665
131.536
131.491
131.407
131.256
131.225
131.202
131.134
130.535
129.852
129.685
129.329
129.215
128.866
128.851
128.767
128.623
128.555
128.502
128.426
128.357
128.289
127.819
127.212
77.571
77.260
76.941
44.204
39.781
39.098
Cl
O=PPh2
CHO
200
180
160
140
120
100
80
60
40
20
ppm

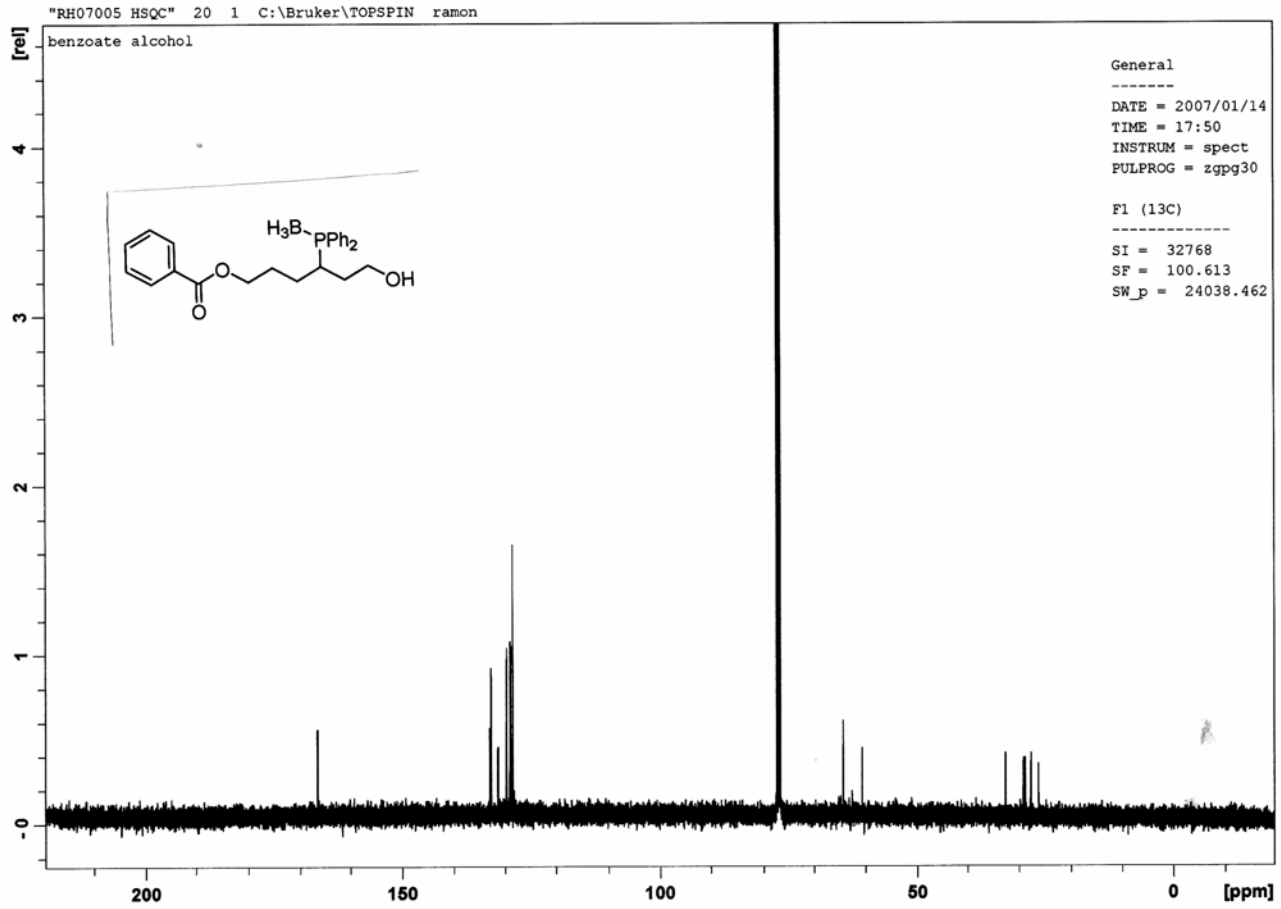
INDEX	FREQUENCY	PPM	HEIGHT
1	5589.916	34.528	21.0

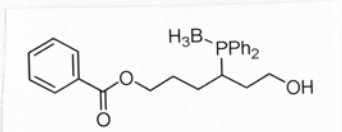


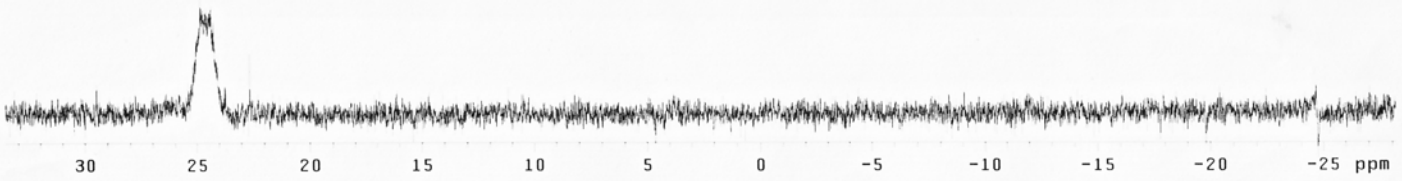


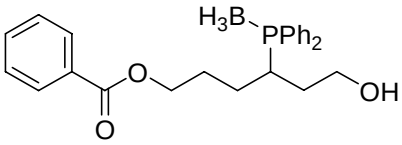

H3B-PPh2
O
O
OH

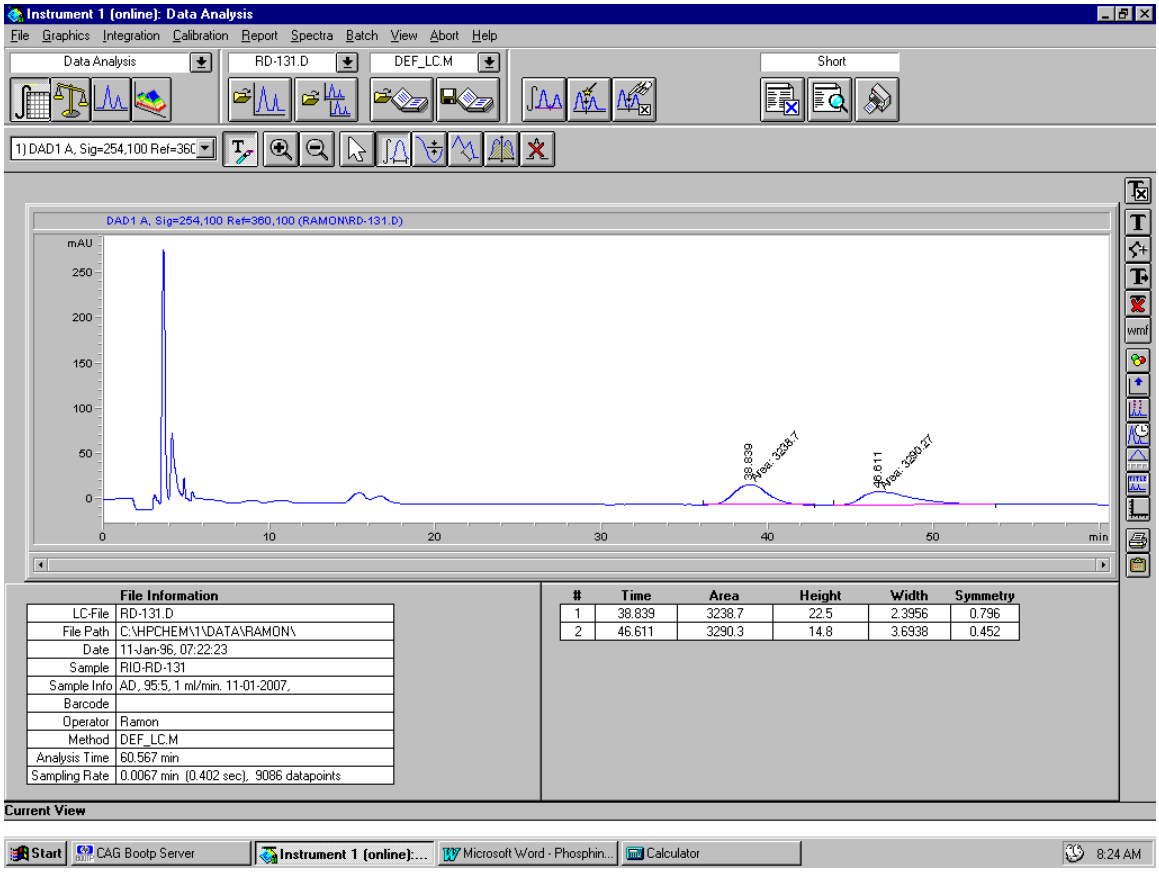

12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.16
1.45
4.45
10.36
2.00
1.91
0.93
2.35
5.04
2.54

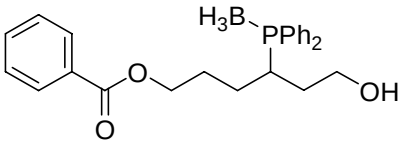

"RH07005 HSQC" 20 1 C:\Bruker\TOPSPIN ramon
benzoate alcohol
[rel]
4
3
2
1
-0
H_3B
PPh_2
O
O
OH
General
DATE = 2007/01/14
TIME = 17:50
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.613
SW_p = 24038.462
200
150
100
50
0
[ppm]

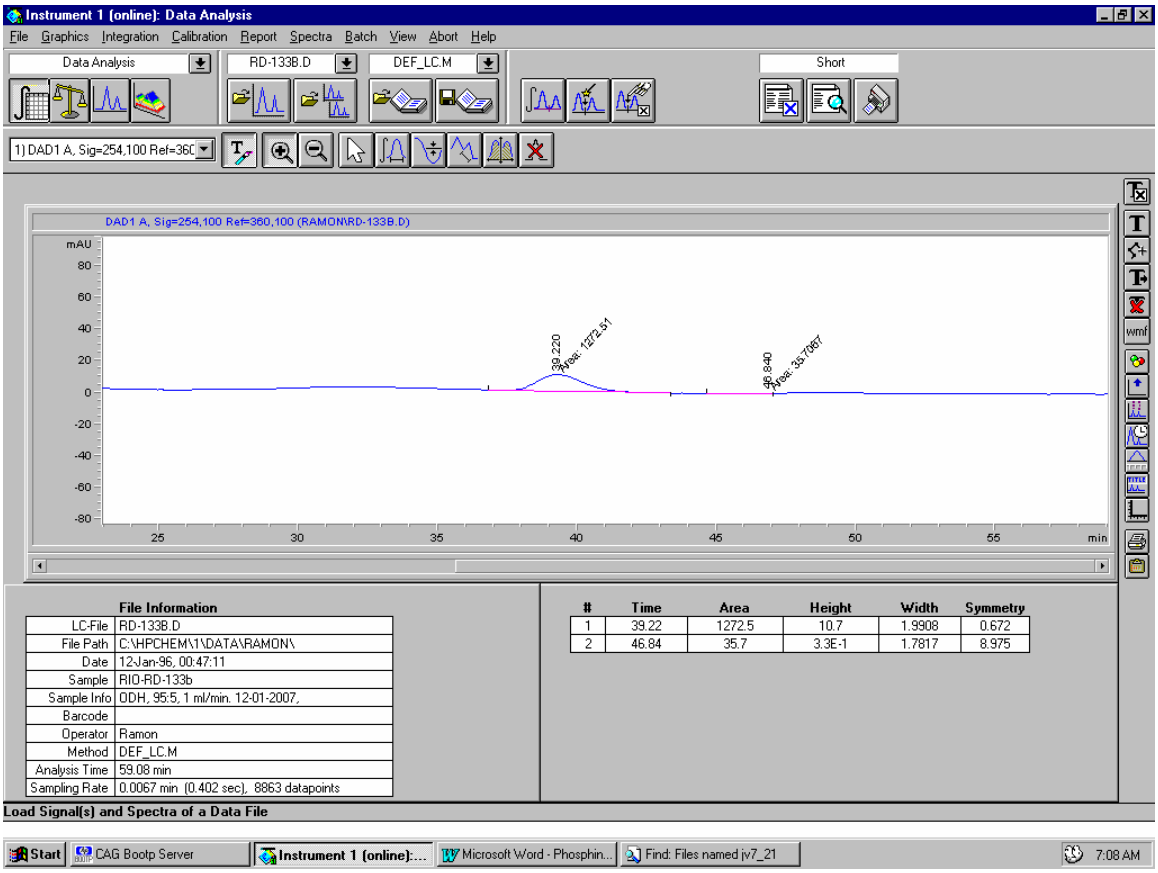

H3B
PPh2
O
O
OH

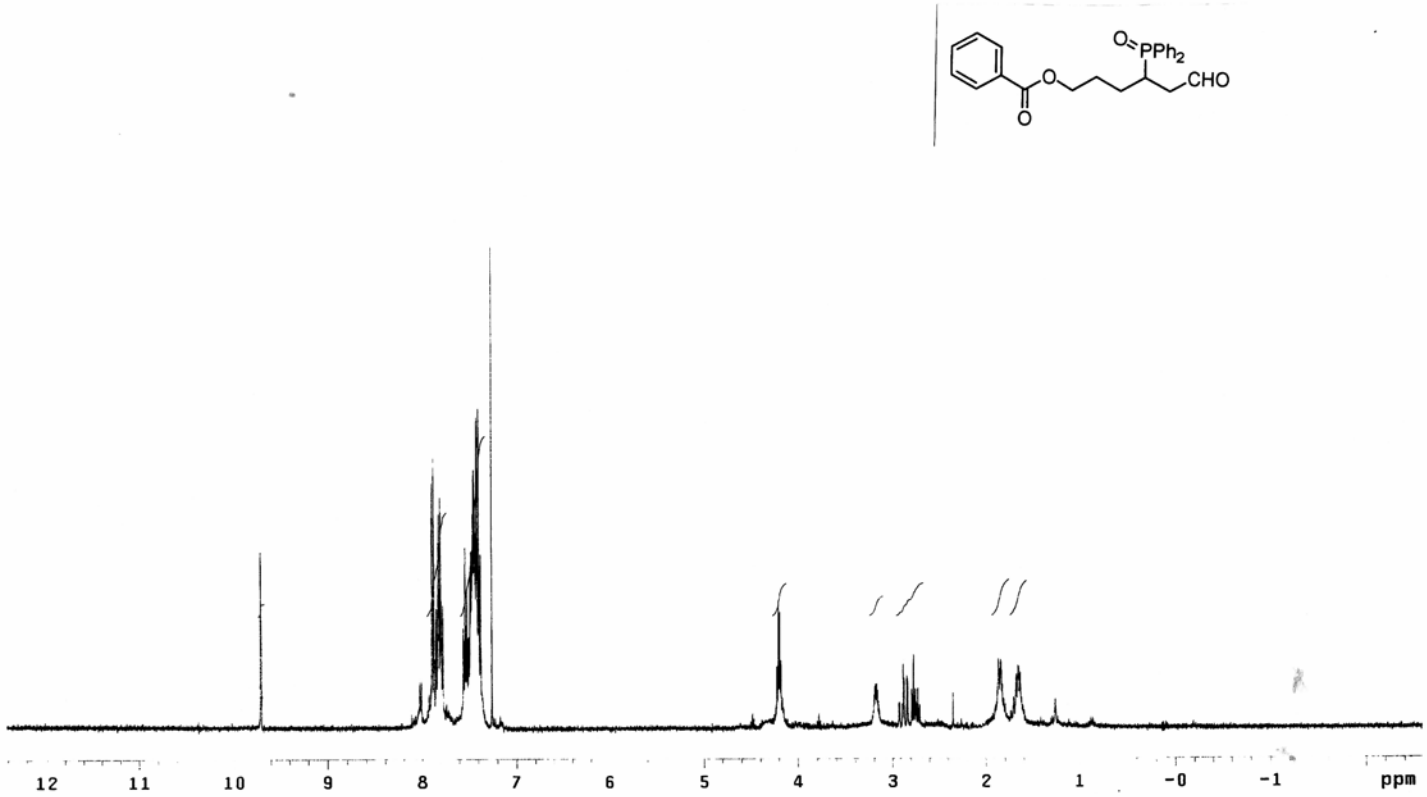

30
25
20
15
10
5
0
-5
-10
-15
-20
-25 ppm

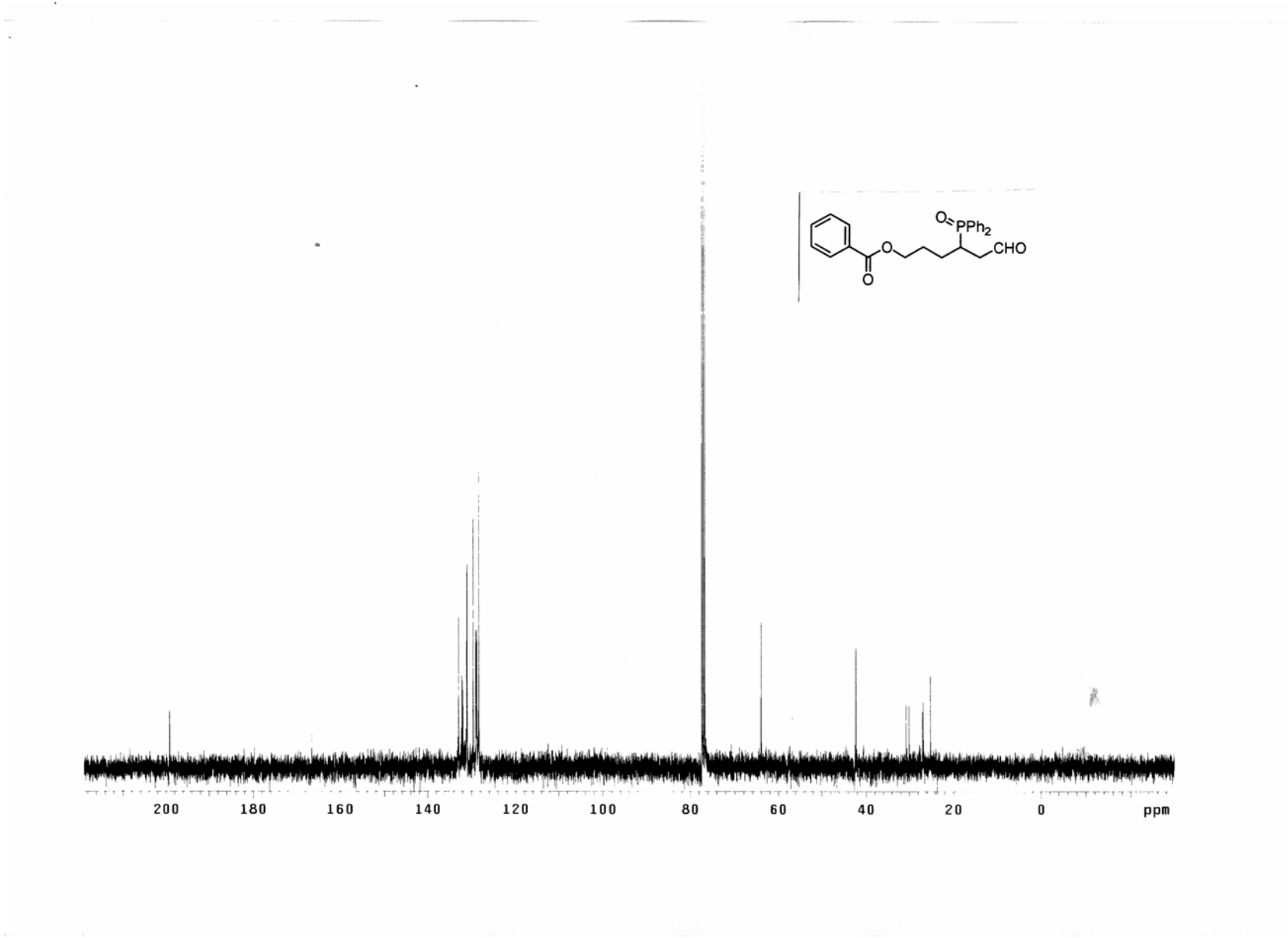

H3B
PPh2
O
OH
O

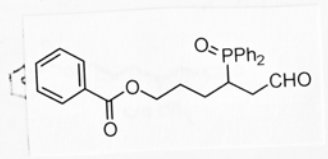

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
RD-131.D
DEF_LC.M
Short
1) DAD1 A, Sig=254,100 Ref=360
DAD1 A, Sig=254,100 Ref=360,100 (RAMON\RD-131.D)
mAU
250
200
150
100
50
0
0
10
20
30
40
50
min
38.839
Area: 3238.7
46.611
Area: 3290.27
File Information
LC-File RD-131.D
File Path C:\HPCHEM\1\DATA\RAMON\
Date 11-Jan-96, 07:22:23
Sample RIO-RD-131
Sample Info AD, 95:5, 1 ml/min. 11-01-2007,
Barcode
Operator Ramon
Method DEF_LC.M
Analysis Time 60.567 min
Sampling Rate 0.0067 min (0.402 sec), 9086 datapoints
Time Area Height Width Symmetry
1 38.839 3238.7 22.5 2.3956 0.796
2 46.611 3290.3 14.8 3.6938 0.452
Current View
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Calculator
8:24 AM


H3B
PPh2
O
OH
O

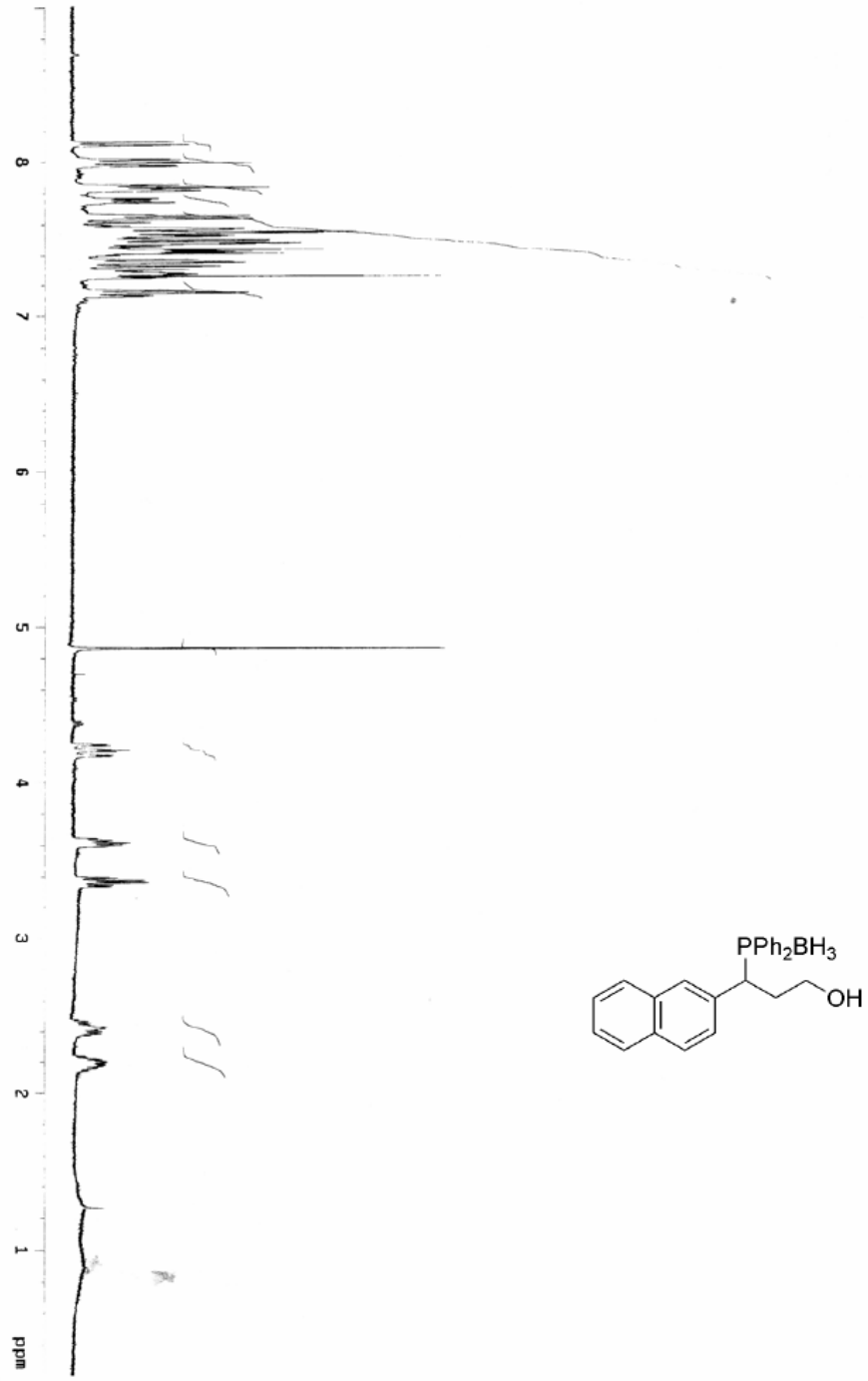

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
RD-133B.D
DEF_LC.M
Short
1) DAD1 A, Sig=254,100 Ref=360
DAD1 A, Sig=254,100 Ref=360,100 (RAMON\RD-133B.D)
mAU
min
39.220
Area: 1272.51
46.840
Area: 35.7067
File Information
LC-File RD-133B.D
File Path C:\HPCHEM\1\DATA\RAMON\
Date 12-Jan-96, 00:47:11
Sample RIO-RD-133b
Sample Info ODH, 95:5, 1 ml/min. 12-01-2007,
Barcode
Operator Ramon
Method DEF_LC.M
Analysis Time 59.08 min
Sampling Rate 0.0067 min (0.402 sec), 8863 datapoints
Time Area Height Width Symmetry
1 39.22 1272.5 10.7 1.9908 0.672
2 46.84 35.7 3.3E-1 1.7817 8.975
Load Signal(s) and Spectra of a Data File
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
7:08 AM


O=PPh2
CHO
O
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

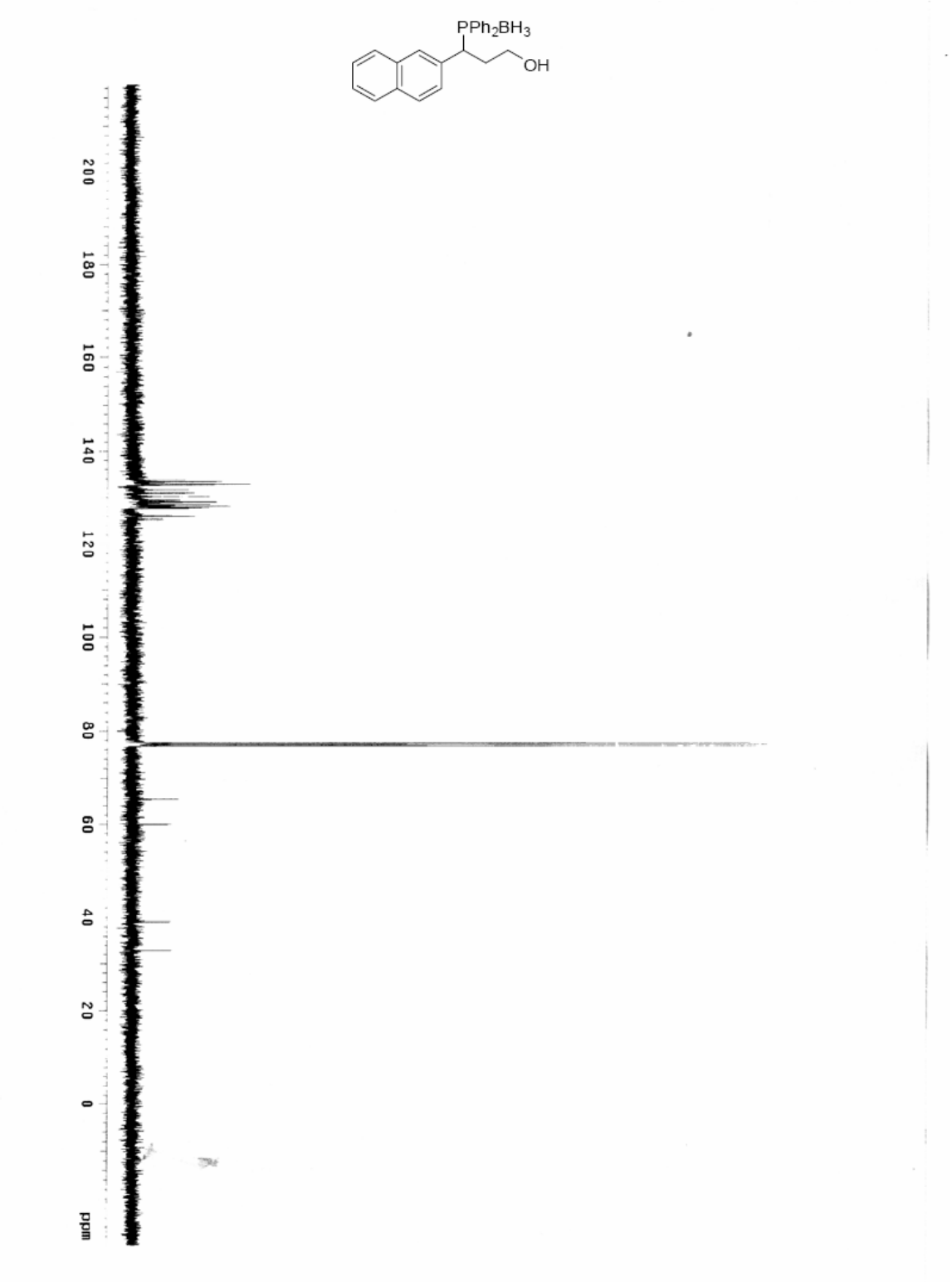

O
PPh2
O
O
CHO
200
180
160
140
120
100
80
60
40
20
0
ppm

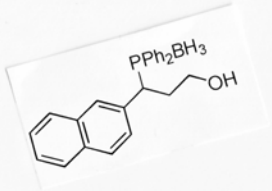

O=PPh2
CHO
O
O

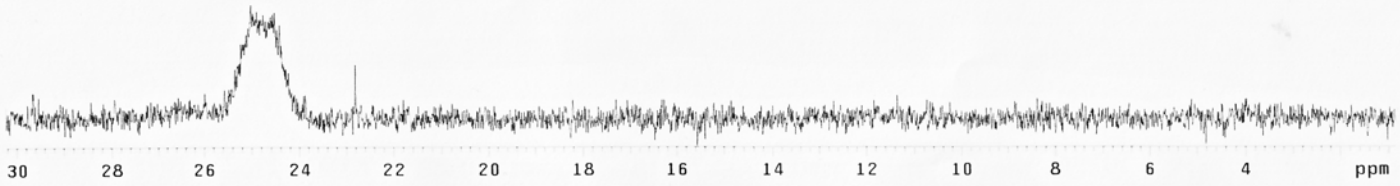
37.697
160
140
120
100
80
60
40
20
0
-20
-40
ppm

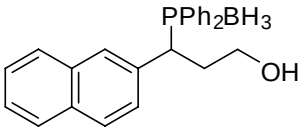

8
7
6
5
4
3
2
1
ppm

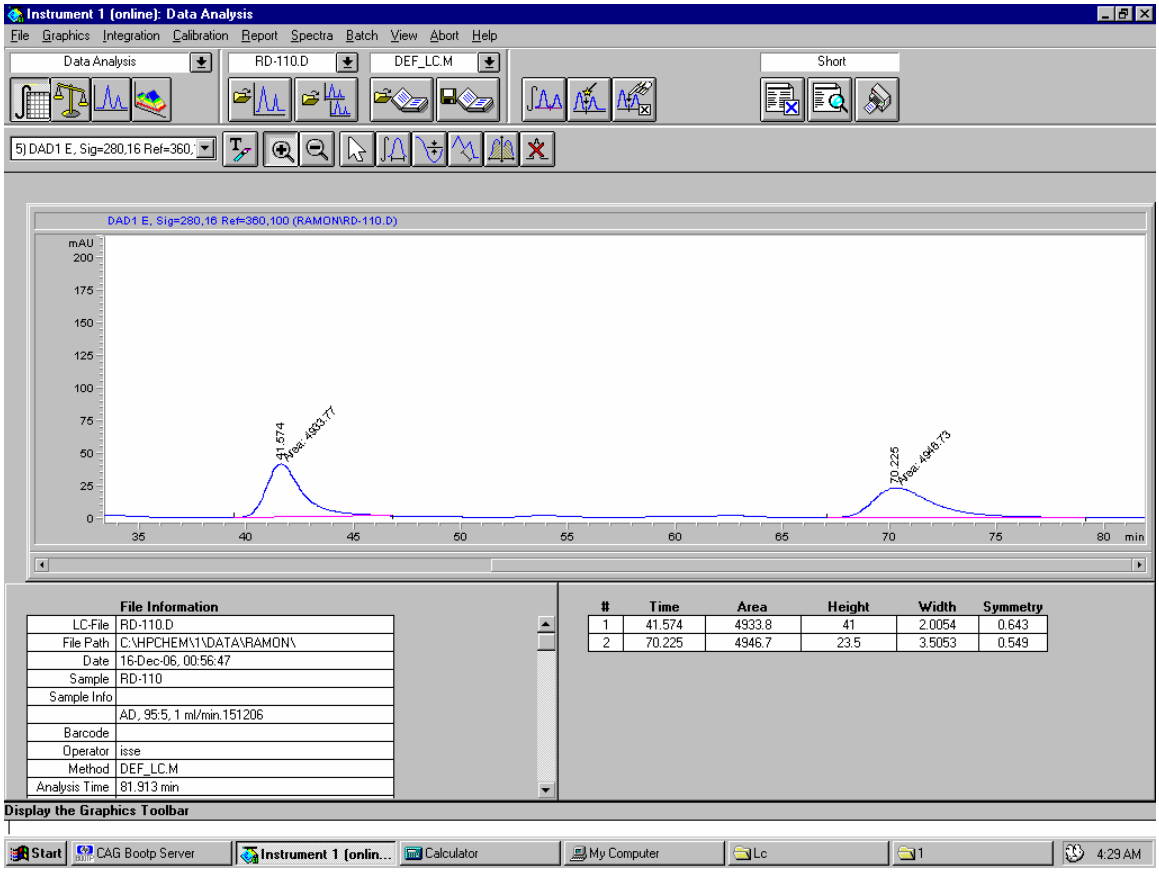
PPh2BH3
OH

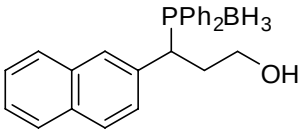

PPh2BH3
OH
200
180
160
140
120
100
80
60
40
20
0
ppm

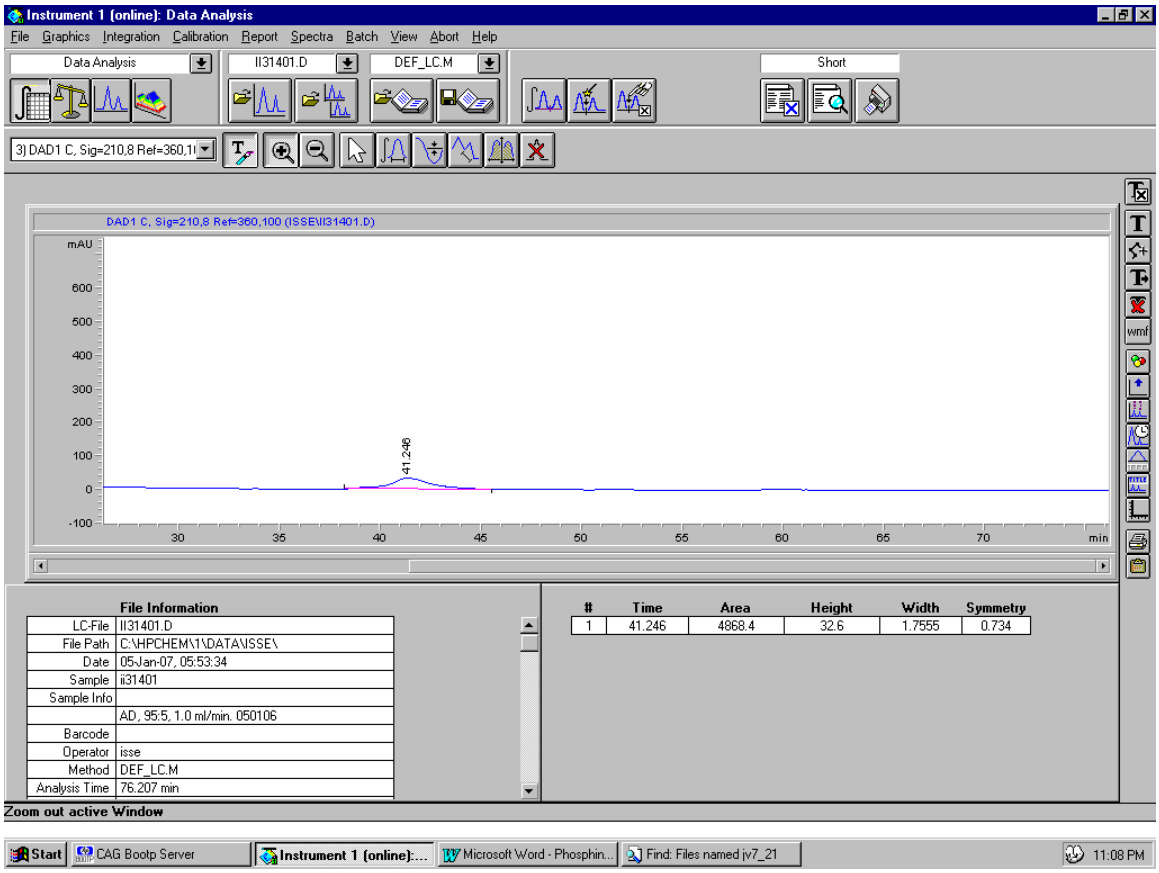

PPh2BH3
OH

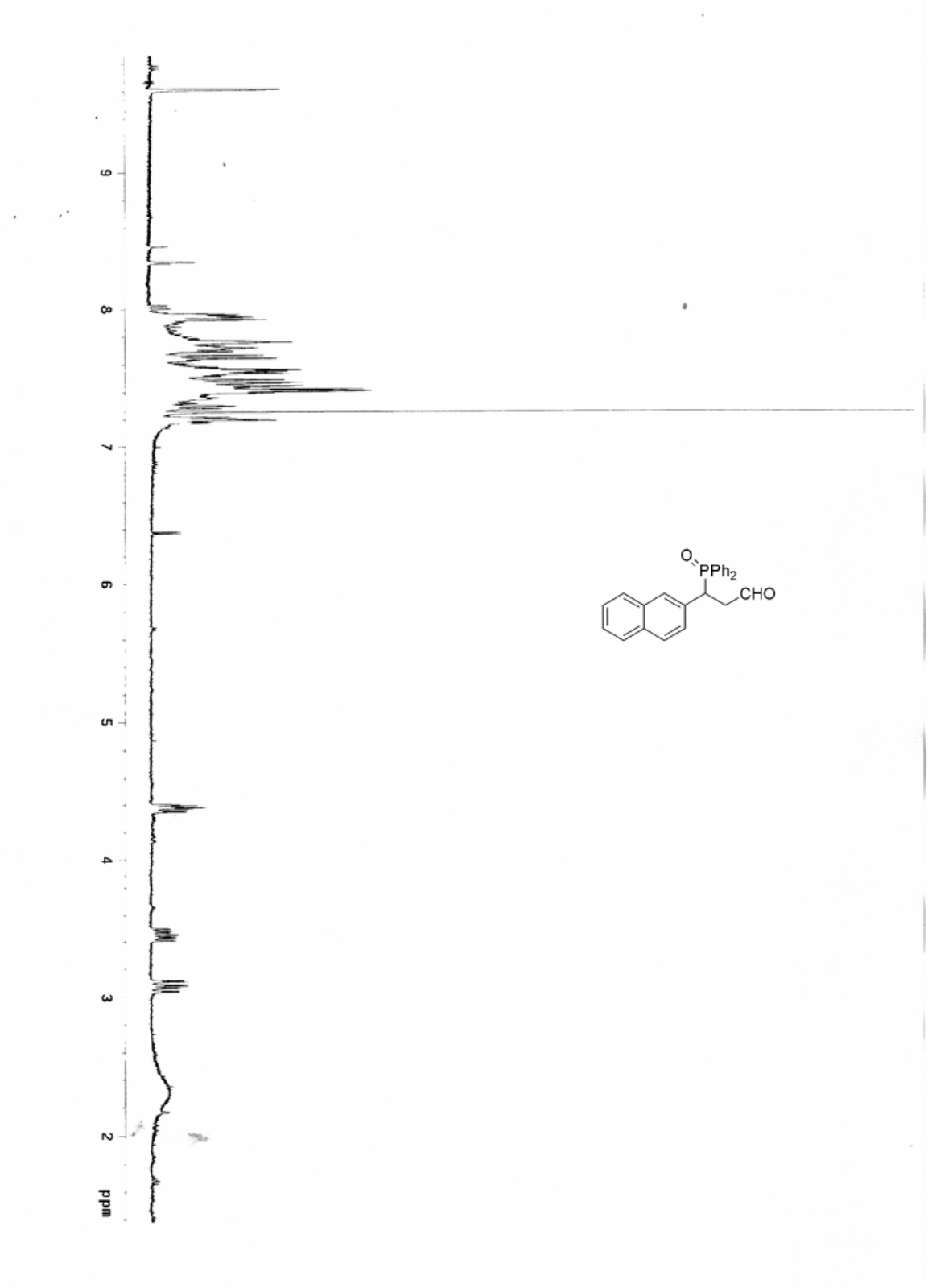

30
28
26
24
22
20
18
16
14
12
10
8
6
4
ppm

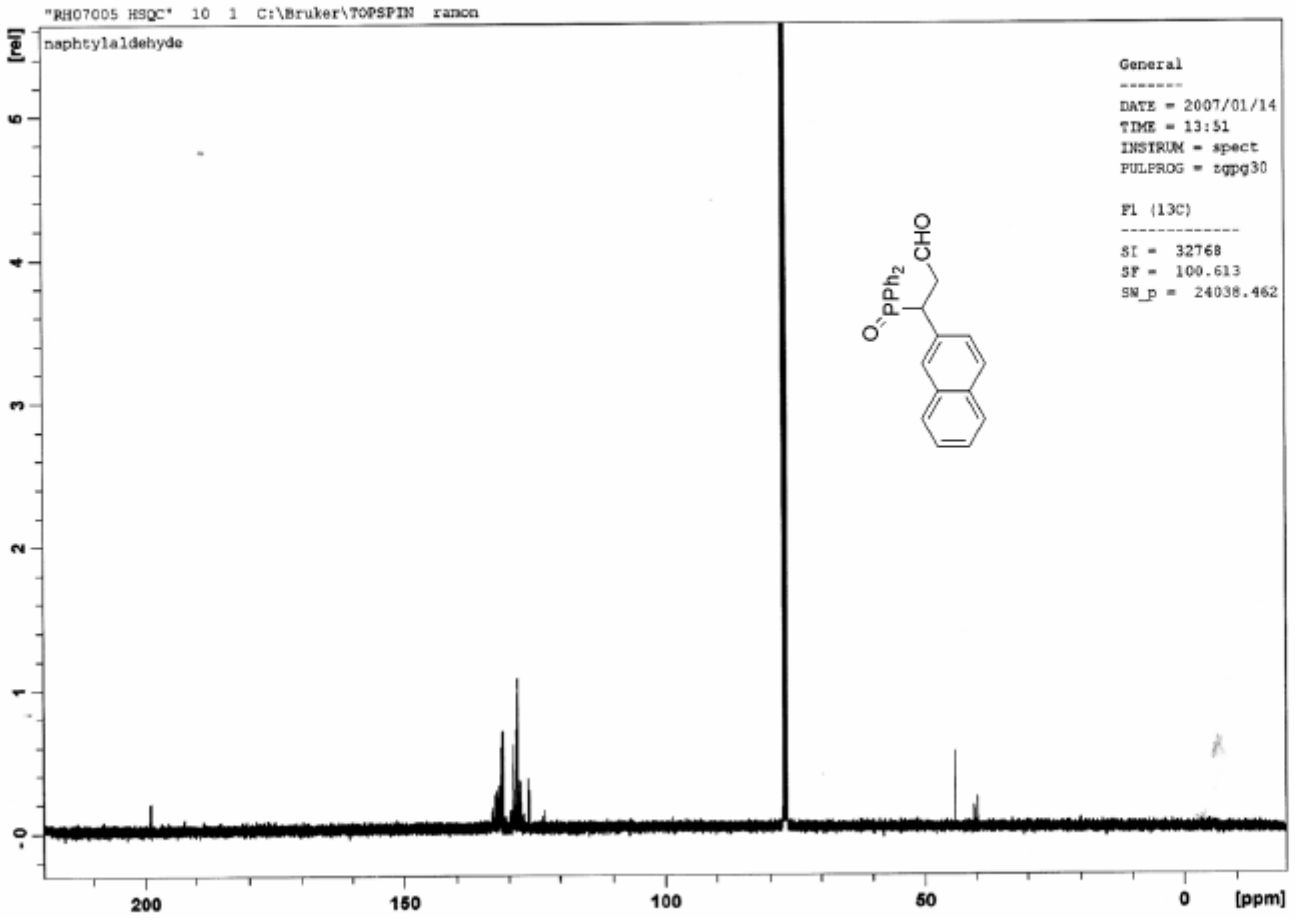

PPh2BH3
OH

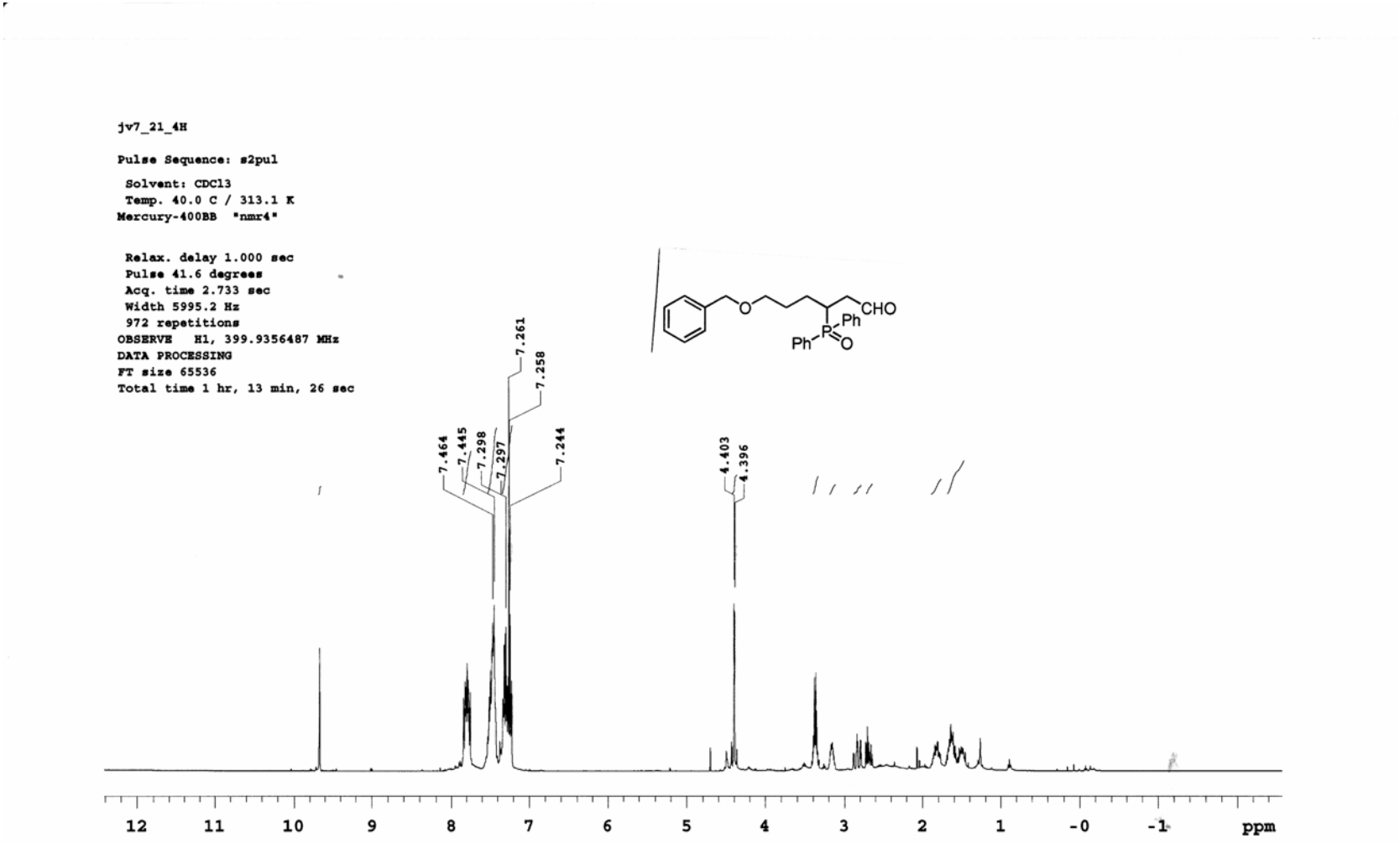

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
RD-110.D
DEF_LC.M
Short
5) DAD1 E, Sig=280,16 Ref=360,
DAD1 E, Sig=280,16 Ref=360,100 (RAMON\RD-110.D)
mAU
200
175
150
125
100
75
50
25
0
41.574
Area: 4933.77
70.225
Area: 4946.73
35 40 45 50 55 60 65 70 75 80 min
File Information
LC-File RD-110.D
File Path C:\HPCHEM\1\DATA\RAMON\
Date 16-Dec-06, 00:56:47
Sample RD-110
Sample Info
AD, 95:5, 1 ml/min.151206
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 81.913 min
Time Area Height Width Symmetry
1 41.574 4933.8 41 2.0054 0.643
2 70.225 4946.7 23.5 3.5053 0.549
Display the Graphics Toolbar
Start
CAG Bootp Server
Instrument 1 (onlin...
Calculator
My Computer
Lc
1
4:29 AM


PPh2BH3
OH


Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
II31401.D
DEF_LC.M
Short
3] DAD1 C, Sig=210,8 Ref=360,1
DAD1 C, Sig=210,8 Ref=360,100 (ISSE\II31401.D)
mAU
600
500
400
300
200
100
0
-100
41.246
30
35
40
45
50
55
60
65
70
min
File Information
LC-File II31401.D
File Path C:\HPCHEM\1\DATA\ISSE\
Date 05-Jan-07, 05:53:34
Sample ii31401
Sample Info
AD, 95:5, 1.0 ml/min. 050106
Barcode
Operator isse
Method DEF_LC.M
Analysis Time 76.207 min
Time Area Height Width Symmetry
1 41.246 4868.4 32.6 1.7555 0.734
Zoom out active Window
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
11:08 PM


9
8
7
6
5
4
3
2
ppm
O
PPh2
CHO


"RH07005 HSQC" 10 1 C:\Bruker\TOPSPIN ramon
naphtylaldehyde
[rel]
6
5
4
3
2
1
-0
200
150
100
50
0
[ppm]
General
DATE = 2007/01/14
TIME = 13:51
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.613
SW_p = 24038.462
O=PPh2
CHO

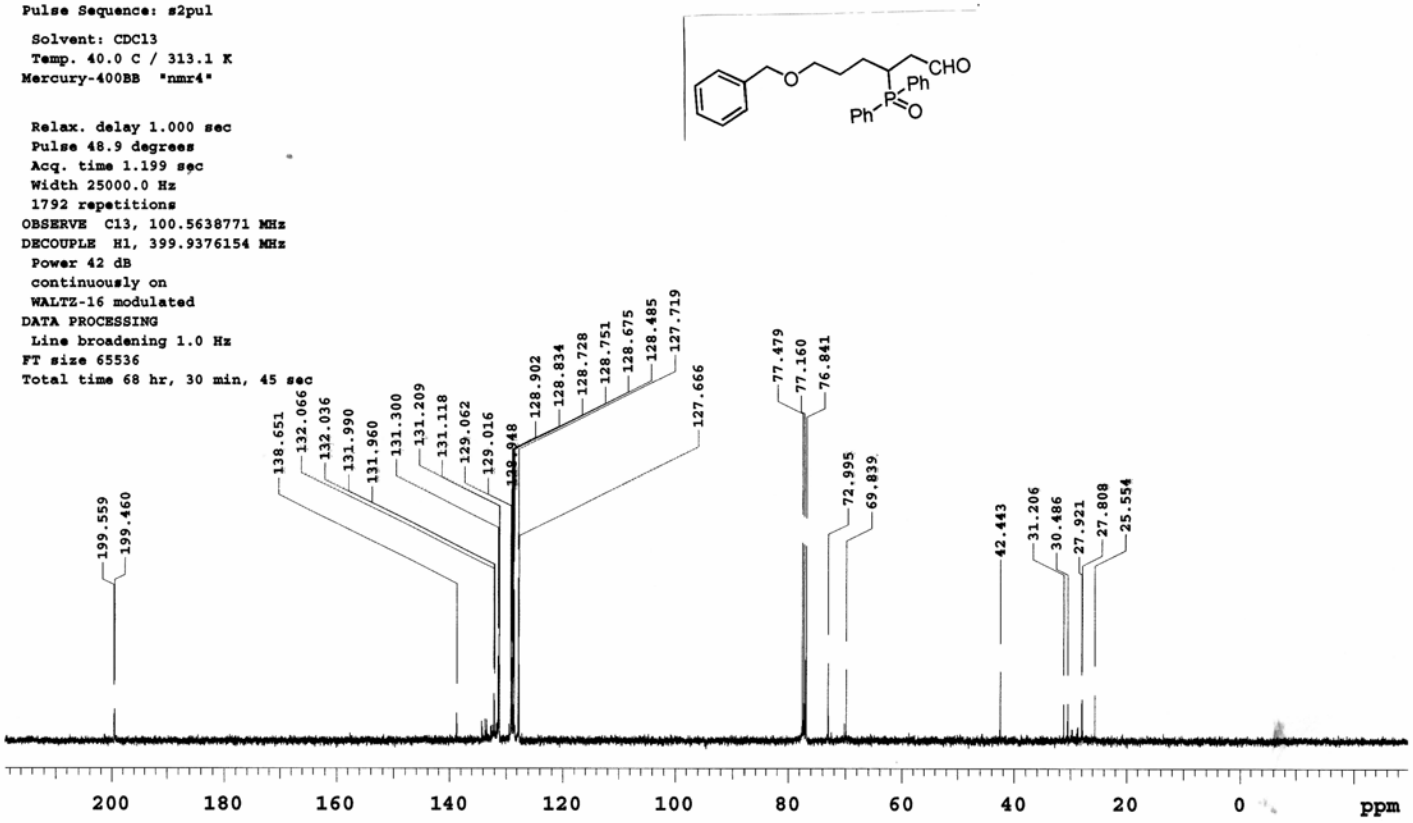

jv7_21_4H
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 40.0 C / 313.1 K
Mercury-400BB "nmr4"
Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.733 sec
Width 5995.2 Hz
972 repetitions
OBSERVE H1, 399.9356487 MHz
DATA PROCESSING
FT size 65536
Total time 1 hr, 13 min, 26 sec
7.464
7.445
7.298
7.297
7.261
7.258
7.244
4.403
4.396
O
CHO
Ph
P
Ph
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

jv07_21_4

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 40.0 C / 313.1 K
Mercury-400BB "nmr4"

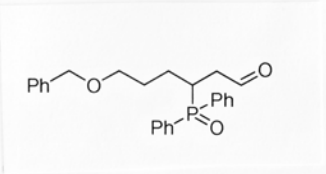
Relax. delay 1.000 sec
Pulse 48.9 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
1792 repetitions
OBSERVE C13, 100.5638771 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 68 hr, 30 min, 45 sec

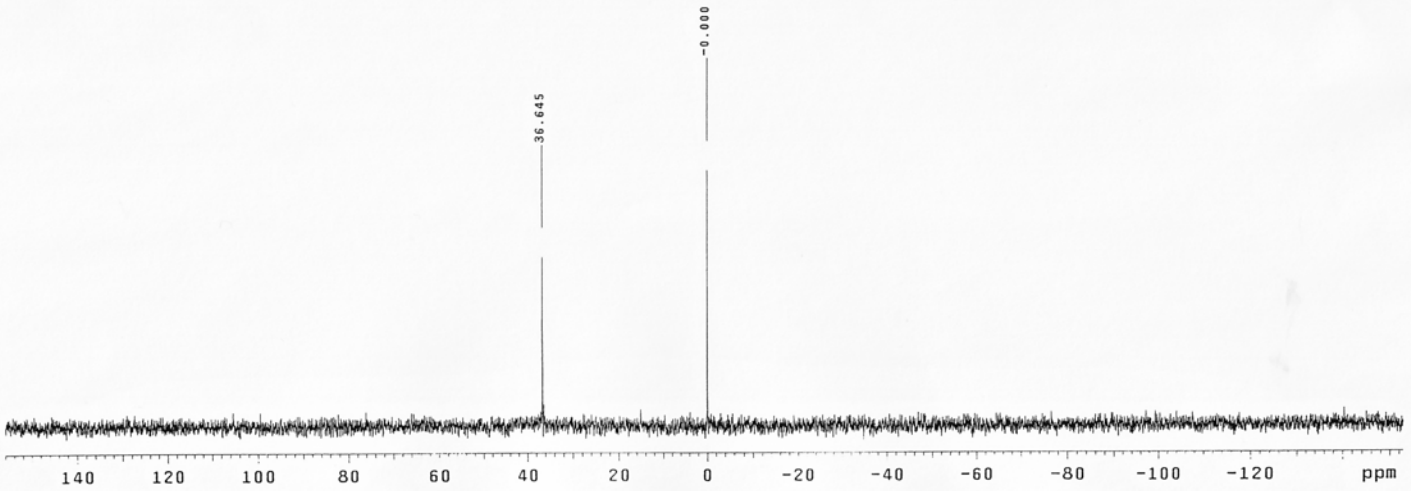


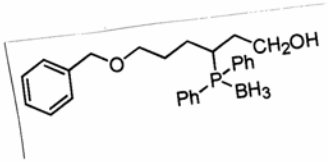
jv07_21_3P

Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 25.0 C / 298.1 K
File: jv07_21_3P
Mercury-400BB "nmr4"

 Pulse 27.6 degrees
 Acq. time 0.800 sec
 Width 50000.0 Hz
 98 repetitions
OBSERVE P31, 161.8969256 MHz
DECOUPLE H1, 399.9376154 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 5.0 Hz
FT size 131072
Total time 56 min, 25 sec





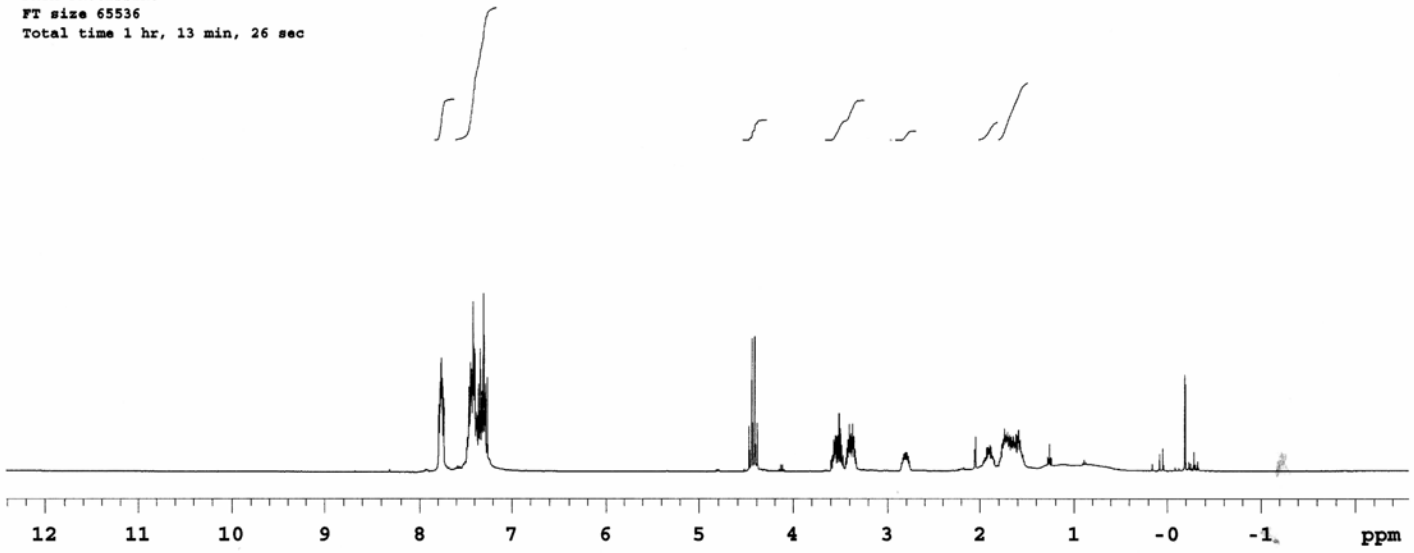


jv07_17_4

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: jv07_17_4
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.733 sec
Width 5995.2 Hz
68 repetitions
OBSERVE H1, 399.9356487 MHz
DATA PROCESSING
FT size 65536
Total time 1 hr, 13 min, 26 sec

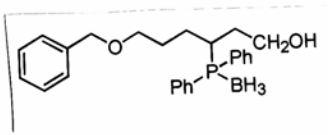


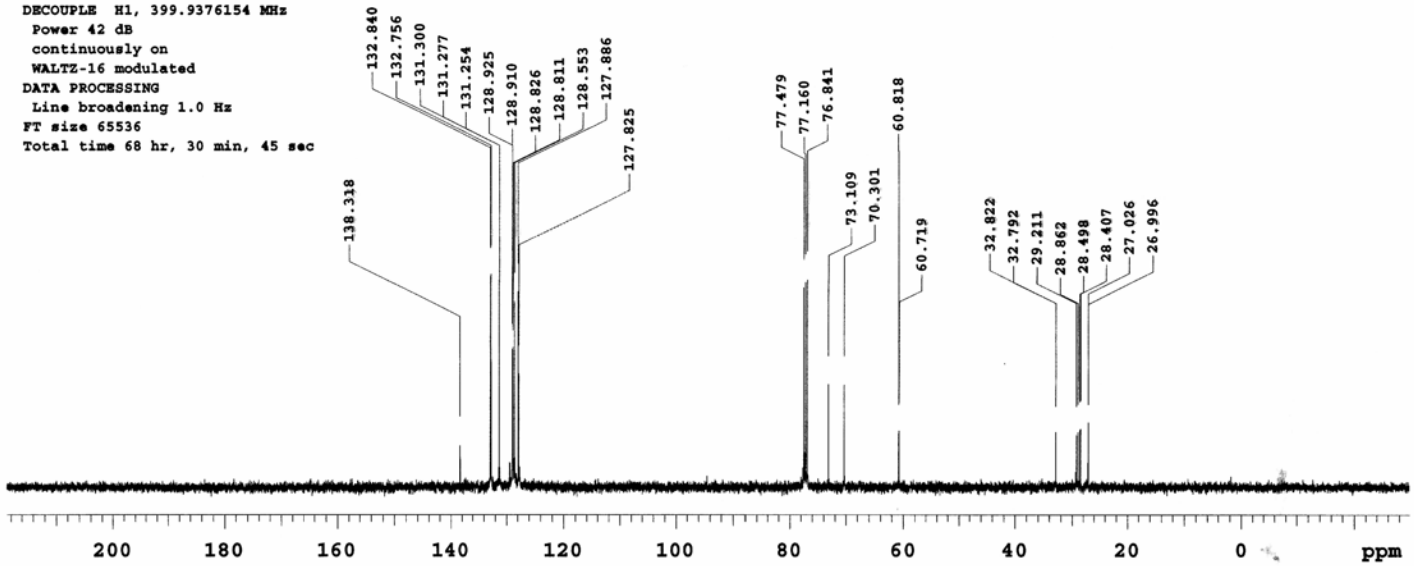
jv07_17_4

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: jv07_17_4C
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 48.9 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 100.5638855 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 68 hr, 30 min, 45 sec

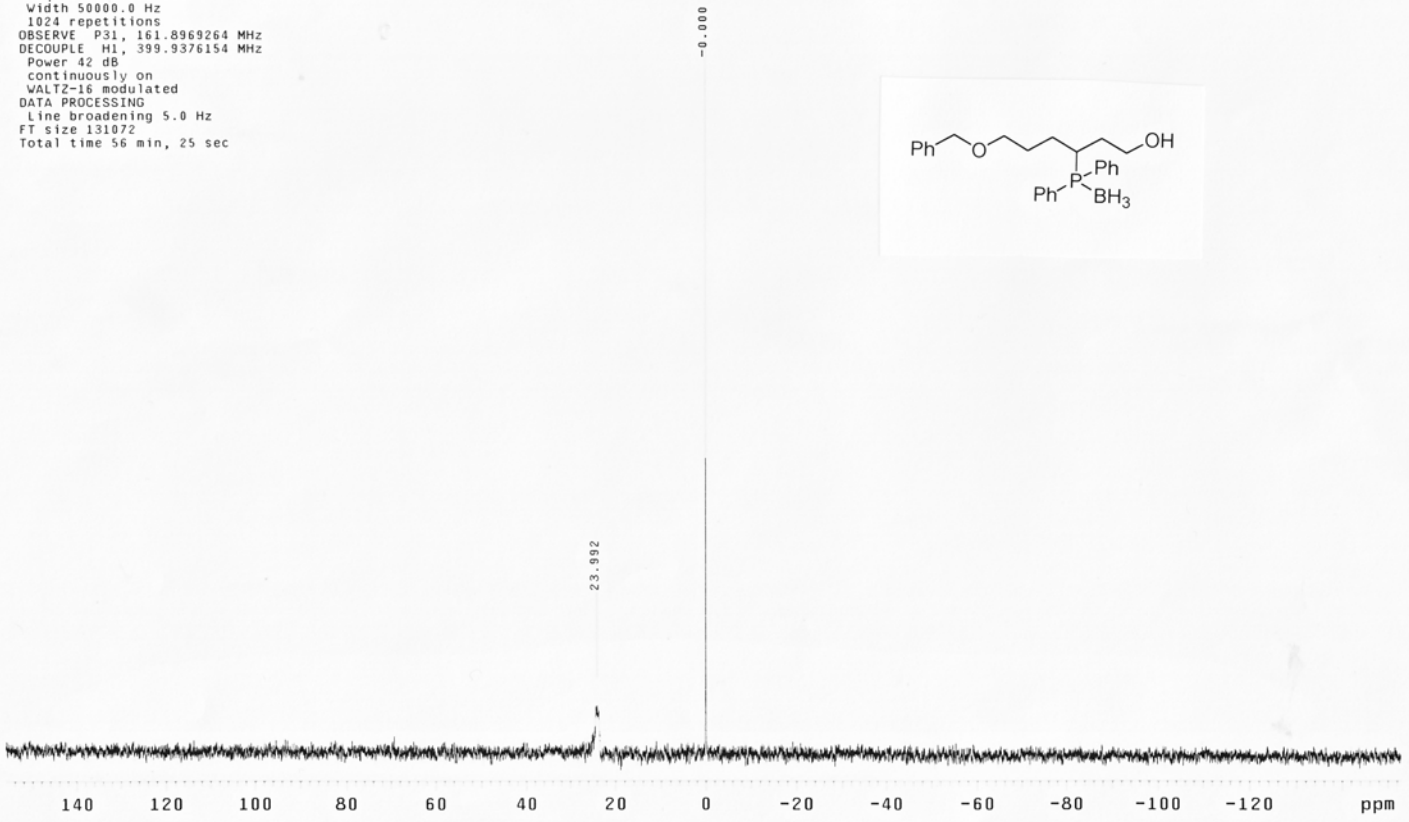


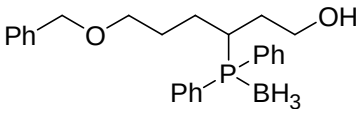


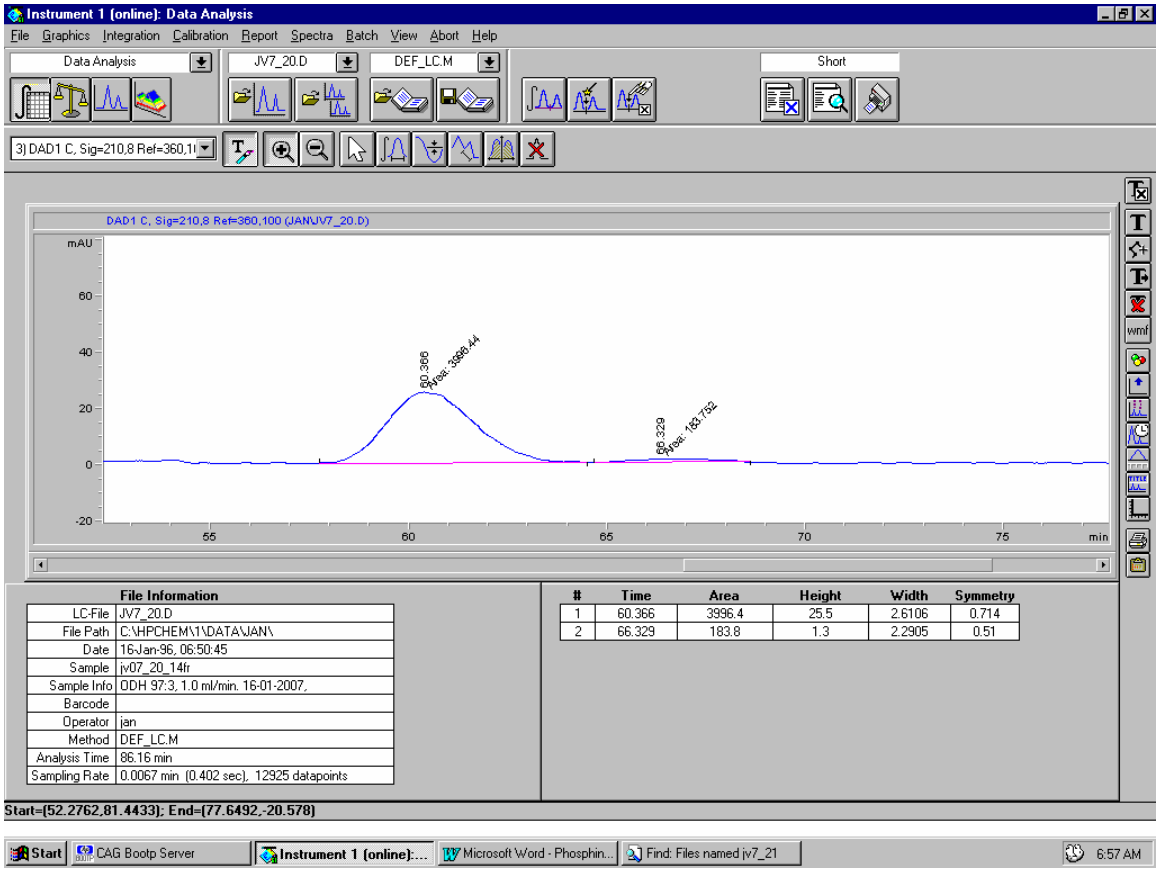
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

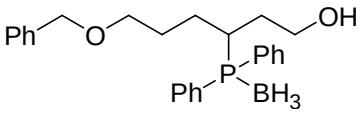
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: jv07_20_3P
Mercury-400BB "nmr4"

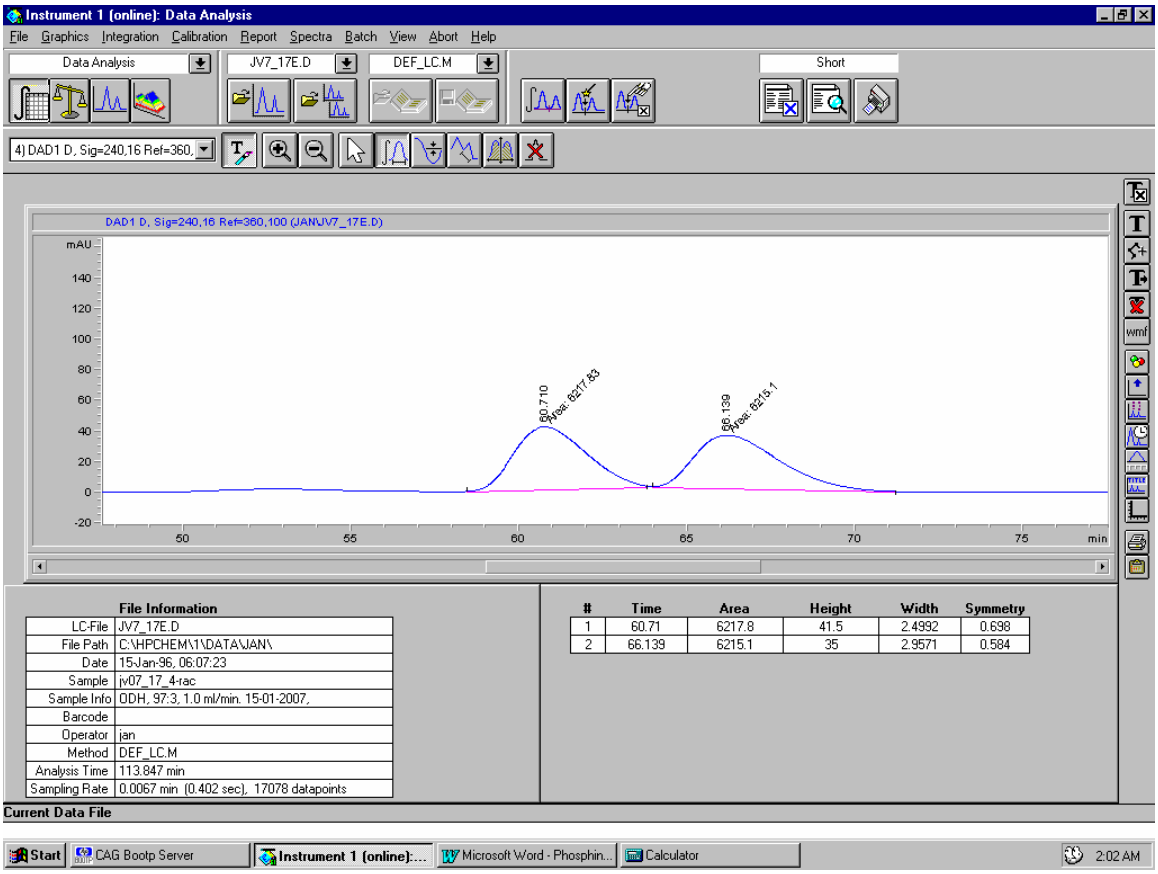
Pulse 27.6 degrees
Acq. time 0.800 sec
Width 50000.0 Hz
1024 repetitions
OBSERVE P31, 161.8969264 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FT size 131072
Total time 56 min, 25 sec




Ph
O
OH
Ph
P
Ph
BH3


Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
JV7_20.D
DEF_LC.M
Short
3) DAD1 C, Sig=210,8 Ref=360,1
DAD1 C, Sig=210,8 Ref=360,100 (JAN\JV7_20.D)
mAU
60
40
20
0
-20
55
60
65
70
75
min
60.366
Area: 3996.44
66.329
Area: 183.752
File Information
LC-File JV7_20.D
File Path C:\HPCHEM\1\DATA\JAN\
Date 16-Jan-96, 06:50:45
Sample jv07_20_14fr
Sample Info ODH 97:3, 1.0 ml/min. 16-01-2007,
Barcode
Operator jan
Method DEF_LC.M
Analysis Time 86.16 min
Sampling Rate 0.0067 min (0.402 sec), 12925 datapoints
Time Area Height Width Symmetry
1 60.366 3996.4 25.5 2.6106 0.714
2 66.329 183.8 1.3 2.2905 0.51
Start=(52.2762,81.4433); End=(77.6492,-20.578)
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Find: Files named jv7_21
6:57 AM


Ph
O
OH
Ph
P
Ph
BH3

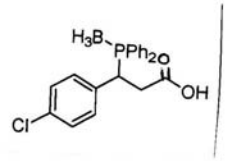

Instrument 1 (online): Data Analysis
File Graphics Integration Calibration Report Spectra Batch View Abort Help
Data Analysis
JV7_17E.D
DEF_LC.M
Short
4) DAD1 D, Sig=240,16 Ref=360,
DAD1 D, Sig=240,16 Ref=360,100 (JAN\JV7_17E.D)
mAU
140
120
100
80
60
40
20
0
-20
50
55
60
65
70
75
min
60.710
Area: 6217.83
66.139
Area: 6215.1
File Information
LC-File JV7_17E.D
File Path C:\HPCHEM\1\DATA\JAN\
Date 15-Jan-96, 06:07:23
Sample jv07_17_4-rac
Sample Info ODH, 97:3, 1.0 ml/min. 15-01-2007,
Barcode
Operator jan
Method DEF_LC.M
Analysis Time 113.847 min
Sampling Rate 0.0067 min (0.402 sec), 17078 datapoints
Time Area Height Width Symmetry
1 60.71 6217.8 41.5 2.4992 0.698
2 66.139 6215.1 35 2.9571 0.584
Current Data File
Start
CAG Bootp Server
Instrument 1 (online):...
Microsoft Word - Phosphin...
Calculator
2:02 AM

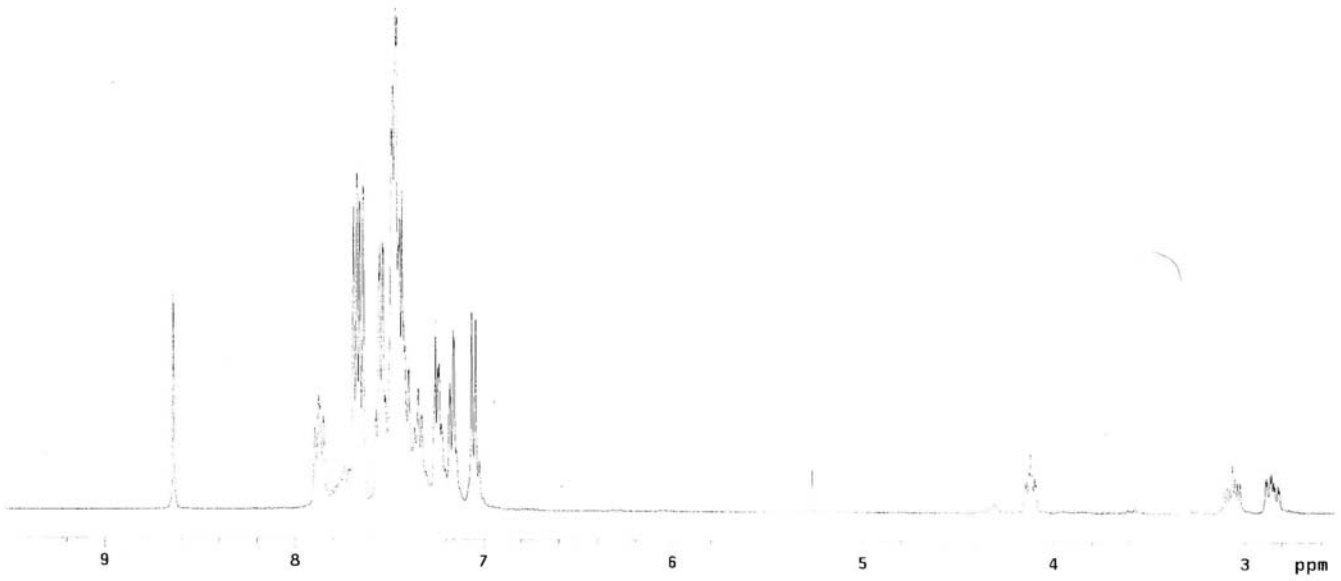
ii31209_flash

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: ii31209_flash_EtOAc
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.733 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9356500 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 2 sec





ii31209_flash_13C

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: ii31209_flash_13C
Mercury-400BB "nmr4"

Relax. delay 1.000 sec
Pulse 48.9 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
672 repetitions
OBSERVE C13, 100.5638785 MHz
DECOUPLE H1, 399.9376154 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13 hr, 42 min, 9 sec