

Supporting Information for

Chiral Amine-thioureas Bearing Multiple Hydrogen Bonding Donors: Highly Efficient Organocatalysts for Asymmetric Michael Addition of Acetylacetone to Nitroolefins

Chun-Jiang Wang,* Zhi-Hai Zhang, Xiu-Qing Dong, and Xiao-Jun Wu

College of Chemistry and Molecular Sciences, Wuhan University, 430072, P. R. China

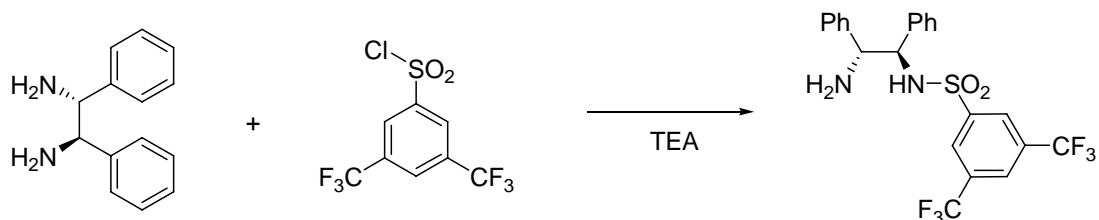
Email: cjwang@whu.edu.cn

General Remarks.

¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration). ¹³C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Enantiomeric ratios were determined by HPLC, using a chiralpak AS-H column, a chiralpak AD-H column or a chiralcel OD-H column with hexane and *i*-PrOH as solvents.

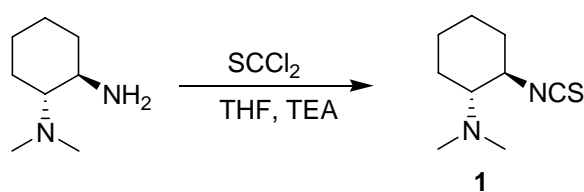
(1*S*,2*S*)-1,2-diphenyl-*N*-tosyl-ethane-1,2-diamine **2a**, (1*R*,2*R*)-1,2-diphenyl -*N*-tosyl-ethane-1,2-diamine **2b**, (1*R*,2*R*)-1,2-diphenyl-*N*-mesyl-ethane-1,2-diamine **2c**, were prepared by the reported synthetic methods.¹ The physical and spectroscopic data of **2a**, **2b** and **2c** are consistent with those reported in the literature.¹

Synthesis of *N*-((1*R*,2*R*)-2-amino-1,2-diphenylethyl)-3,5-bis(trifluoromethyl) benzenesulfonamide (**2d**)



A solution of 3,5-bis(trifluoromethyl) benzenesulfonyl chloride (312.6 mg, 1 mmol) in anhydrous THF (5 mL) was added dropwise to (1*R*,2*R*)-1,2-diphenylethane-1,2-diamine (212.3mg, 1mmol), triethylamine (277 μ L, 2 mmol) and anhydrous THF (10 mL) with ice-cooling. The reaction mixture was brought to room temperature and stirred over night. The result solution was diluted with ethyl acetate and washed with brine. The resulted organic phase was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by flash silica gel chromatography. The pure product was obtained as a white solid in 86% yield.

Mp. 158-160 $^{\circ}\text{C}$; $[\text{R}]_{\text{D}}^{25} +15.6$ (c , 0.4, CHCl_3); IR (KBr) ν 3436, 3355, 3298, 1352, 1278, 1265, 1196, 1163, 1128 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 4.18 (d, J = 4.5 Hz, 1H), 4.52 (d, J = 4.5 Hz, 1H), 7.13 (s, 10H), 7.79 (s, 1H), 7.86 (s, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 60.27, 63.68, 125.69, 126.42, 127.17, 127.32, 128.10, 128.18, 128.76, 132.17, 132.62, 138.27, 141.15, 143.40; HRMS Calcd. for $\text{C}_{22}\text{H}_{19}\text{F}_6\text{N}_2\text{O}_2\text{S}$: 489.1071, found: 489.1047;



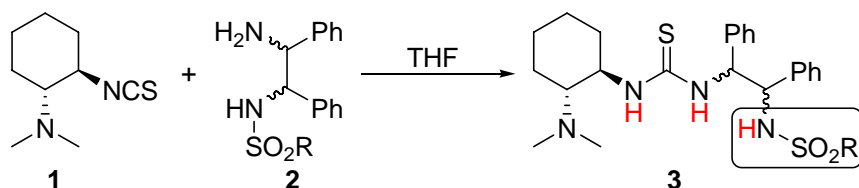
Synthesis of (1*R*,2*R*)-2-isothiocyanato-*N,N*-dimethylcyclohexanamine (**1**)

Thiophosgen (1.86 mL, 24.4 mmol) was added dropwise to a solution of (1*R*,2*R*)-*N,N*-dimethylcyclohexane-1,2-diamine (2.31 g, 16.3 mmol) and triethylamine (6.77 mL, 48.8 mmol) with ice-cooling. The reaction mixture was stirred for about 4h, and TLC analysis indicated completion of the reaction. Then the reaction mixture was concentrated *in vacuo*, and the residue was purified by flash silica gel chromatography to afford a brown oil in 91% yield.

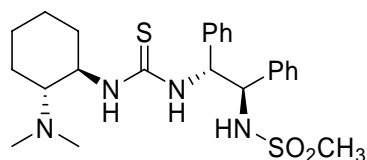
$[\alpha]_{\text{D}}^{25} -99.0$ (c 0.36, CHCl_3); IR (KBr) ν 2935, 2860, 2827, 2780, 2185, 2095, 1618 cm^{-1} ; ^1H

NMR (CDCl₃, TMS, 300 MHz) δ 1.16-1.25 (m, 4H), 1.69-1.84 (m, 3H), 2.15-2.19 (m, 1H), 2.34 (s, 6H), 2.40-2.47 (m, 1H), 3.51-3.59 (m, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 23.58, 24.32, 24.45, 33.57, 40.41, 58.23, 67.22, 76.63, 77.06, 77.48; MS (EI) *m/z* 184 ([M]⁺).

General Procedure for the Synthesis of Organocatalysts (3a-3e)



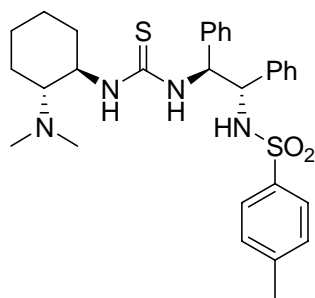
To a solution of corresponding sulfonamide **2** (1 mmol) in anhydrous THF (10 mL) was added (1*R*,2*R*)-2-isothiocyanato-*N,N*-dimethylcyclohexanamine (194 mg, 1.05 mmol) at room temperature. The solution was stirred overnight. TLC analysis indicated completion of the reaction. The reaction mixture was concentrated *in vacuo*. The residue was purified by flash silica gel chromatography.



N-((1*R*,2*R*)-2-(3-((1*R*,2*R*)-2-(dimethylamino)cyclohexyl)thioureido)-1,2-diphenylethyl)methanesulfonamide (**3c**)

The pure product was obtained as a white solid in 97% yield.

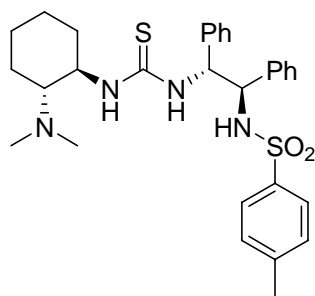
Mp. 108-110 °C; $[\alpha]_D^{25} +9.4$ (*c* 0.44, CHCl₃); IR (KBr) ν 3356, 3062, 2931, 2858, 1545, 1320, 1148 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.21-1.24 (m, 4H), 1.69-1.90 (m, 4H), 2.20 (m, 1H), 2.41 (s, 6H), 2.47 (s, 3H), 3.70 (m, 1H), 4.79 (d, *J* = 10.2 Hz, 1H), 5.88 (s, 1H), 7.08-7.22 (m, 10H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 22.65, 24.37, 24.52, 32.59, 39.86, 41.69, 55.24, 62.25, 64.38, 66.34, 127.64, 127.76, 128.44, 128.54, 138.22, 139.15, 183.50; HRMS Calcd. for C₂₄H₃₅N₄O₂S₂: 475.2201, found: 475.2168;



1-((1R,2R)-2-(dimethylamino)cyclohexyl)-3-((1S,2S)-1,2-diphenyl-2-(tosylamino)ethyl)thiourea (3a)

The pure product was obtained as a white solid in 95% yield.

Mp. 110-113 °C; $[\alpha]_D^{25} +18.5$ (*c* 0.62, CHCl₃); IR (KBr) ν 3357, 3062, 3030, 2932, 2858, 1536, 1327, 1155 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.08-1.29 (m, 4H), 1.63-1.76 (m, 3H), 1.95 (s, 6H), 2.20 (m, 1H), 2.25 (s, 3H), 2.42 (m, 1H), 3.58 (m, 1H), 4.70 (d, *J* = 10.2 Hz, 1H), 5.68 (m, 1H), 6.86-7.13 (m, 12H), 7.41 (d, *J* = 8.1 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.59, 22.37, 24.79, 25.17, 33.41, 40.13, 56.67, 62.99, 63.84, 67.25, 127.10, 127.40, 128.03, 128.13, 128.68, 129.18, 138.21, 138.50, 142.54, 183.00; HRMS Calcd. for C₃₀H₃₉N₄O₂S₂: 551.2514, found: 551.2494;

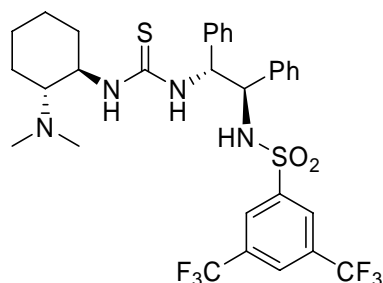


N-((1R,2R)-2-(3-((1R,2R)-2-(dimethylamino)cyclohexyl)thioureido)-1,2-diphenylethyl)-4-methylbenzenesulfonamide (3b)

The pure product was obtained as a white solid in 80% yield.

Mp. 128-130 °C; $[\alpha]_D^{25} +3.5$ (*c* 0.62, CHCl₃); IR (KBr) ν 3355, 3061, 3030, 2932, 2859, 1538, 1454, 1329, 1157 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.18-1.27 (m, 4H), 1.68-1.90 (m, 3H), 2.30 (s, 1H), 2.35 (s, 6H), 2.20-2.43 (m, 2H), 3.47 (s, 1H), 4.72 (d, *J* = 10.8 Hz, 1H), 5.81 (m, 1H), 6.86-7.21 (m, 12H), 7.40 (d, *J* = 10.8 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.67, 22.88, 24.76, 25.11, 32.89, 41.29, 56.85, 63.04, 64.79, 67.17, 126.99, 127.37, 127.95, 128.10,

128.67, 129.28, 138.46, 142.62, 182.70; HRMS Calcd. for C₃₀H₃₉N₄O₂S₂: 551.2514, found: 551.2499;

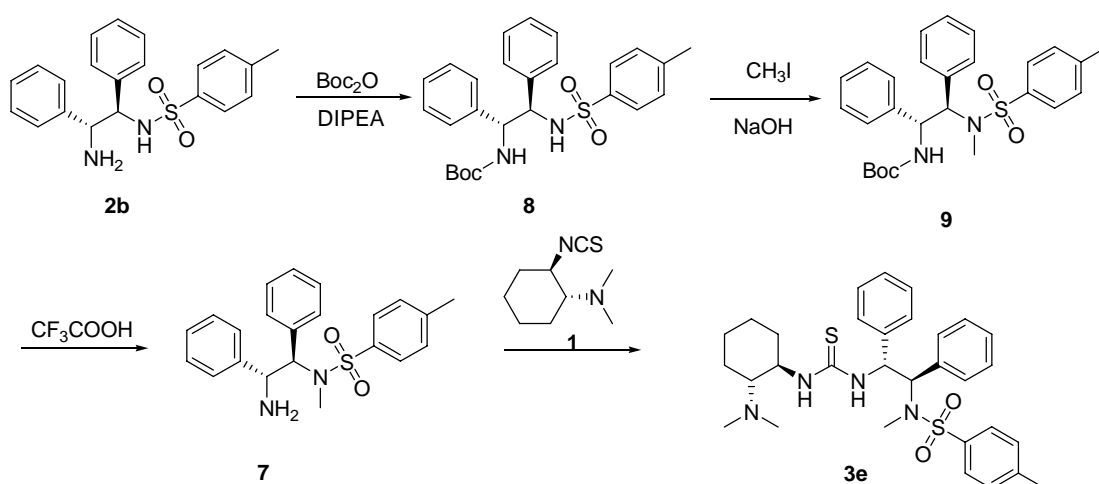


***N*-((1*R*,2*R*)-2-(3-((1*R*,2*R*)-2-(dimethylamino)cyclohexyl)thioureido)-1,2-diphenylethyl)-3,5-bis(trifluoromethyl)benzenesulfonamide (3d)**

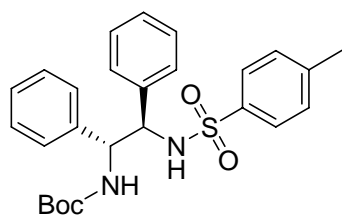
The pure product was obtained as a white solid in 99% yield.

Mp. 168-170 °C; [α]_D²⁵ +43.6 (*c* 0.34, CHCl₃); IR (KBr) ν 3423, 2936, 1541, 1539, 1279, 1162, 1141 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.20 (m, 4H), 1.71-1.94 (m, 3H), 2.18 (m, 1H), 2.42 (s, 6H), 2.44 (m, 1H), 3.35 (m, 1H), 4.81-4.86 (m, 1H), 5.87 (m, 1H), 6.86-7.12 (m, 10H), 7.71 (s, 1H), 7.83 (s, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 24.65, 24.98, 24.76, 33.00, 41.77, 60.34, 63.04, 63.98, 65.39, 67.79, 120.79, 124.41, 125.26, 125.59, 126.49, 127.20, 127.88, 128.03, 128.25, 128.40, 128.73, 128.86, 131.89, 132.35, 136.97, 137.76, 144.45, 183.27; HRMS Calcd. for C₃₁H₃₅F₆N₄O₂S₂: 673.2106, found: 673.2058;

Synthesis of *N*-((1*R*,2*R*)-2-(3-((1*R*,2*R*)-2-(dimethylamino)cyclohexyl)thioureido)-1,2-diphenylethyl)-*N*,4-dimethylbenzenesulfonamide (3e)



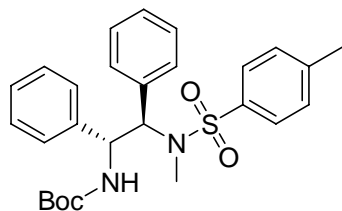
Synthesis of *tert*-butyl (1*R*,2*R*)-1,2-diphenyl-2-(tosylamino)ethylcarbamate (8)



A solution of Boc₂O (94.3 mg, 0.432 mol) in anhydrous dichloromethane (10 mL) was added to (1*R*,2*R*)-1,2-diphenyl-*N*-tosylethane-1,2-diamine (144 mg, 0.393 mmol) and DIPEA (74.3 μL, 0.432 mmol) in anhydrous dichloromethane (20 mL) with ice-cooling. After the addition, the reaction mixture was brought to room temperature and stirred overnight. TLC analysis indicated completion of the reaction. The reaction mixture was concentrated *in vacuo*. The residue was purified by flash silica gel chromatography. The pure product was obtained as a white solid in 90% yield.

Mp. 167-170 °C; $[\alpha]_D^{25} +13.0$ (*c* 0.78, CHCl₃); IR (KBr) ν 3390, 3312, 2928, 1686, 1514, 1158 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.45 (s, 9H), 2.30 (s, 3H), 4.56-4.61 (m, 1H), 4.78-4.84 (m, 1H), 5.33 (br, 1H), 6.22 (br, 1H), 6.78-6.81 (m, 2H), 6.96-7.02 (m, 7H), 7.14-7.15 (m, 3H), 7.43 (d, *J* = 8.1 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.62, 28.57, 60.23, 64.16, 80.79, 127.11, 127.54, 127.69, 128.03, 128.22, 128.72, 129.33, 138.08, 138.57, 142.88, 156.55; HRMS Calcd. for C₂₆H₃₀N₂O₄S+Na⁺: 489.1824, found: 489.1827.

Synthesis of *tert*-butyl (1*R*,2*R*)-2-(*N*-methyl-*N*-tosylamino)-1,2-diphenylethylcarbamate (9)

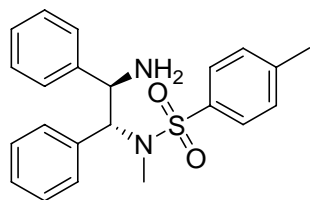


Methyl iodide (220 μL, 3.54 mmol) was added to a solution of *tert*-butyl (1*R*,2*R*)-1,2-diphenyl-2-(tosylamino)ethylcarbamate (165 mg, 0.35 mmol) and 1N NaOH (0.4 mL) in 1,4-dioxane (2 mL) at room temperature. TLC analysis indicated completion of the reaction after about 4h. The result solution was diluted with water and extracted with ethyl acetate. The resulted oil phase was washed with brine, dried with Na₂SO₄ and concentrated *in vacuo*. The residue was purified by

flash silica gel chromatography. The pure product was obtained as a white solid in 81% yield.

Mp. 67-69 °C; $[\alpha]_D^{25}$ -30.4 (*c* 0.54, CHCl₃); IR (KBr) ν 3428, 2975, 2928, 1711, 1599, 1508, 1384, 1163 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.43 (s, 9H), 2.36 (s, 3H), 2.80 (s, 3H), 5.20 (m, 1H), 5.32 (m, 1H), 5.45 (br, 1H), 6.94-7.20 (m, 12H), 7.58 (d, *J* = 6.0 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.69, 28.59, 30.07, 54.18, 64.67, 80.00, 127.34, 127.64, 127.79, 128.28, 128.37, 128.68, 129.33, 129.79, 134.73, 137.56, 140.20, 143.39, 155.81; HRMS Calcd. for C₂₇H₃₂N₂O₄S+Na⁺: 503.1980, found: 503.1926.

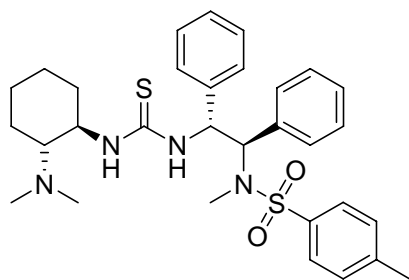
Synthesis of (1*R*,2*R*)-*N*-methyl-1,2-diphenyl- *N*-tosylethane-1,2-diamine (7)



Tert-butyl (1*R*,2*R*)-2-(*N*-methyl-*N*-tosylamino)-1,2-diphenylethylcarbamate (60 mg, 0.125 mmol) was added to CF₃COOH (0.43 mL) with ice-cooling. After the addition, the reaction mixture was brought to room temperature and stirred for 2h. TLC analysis indicated completion of the reaction. CF₃COOH was removed *in vacuo*. The residue was washed with saturated NaHCO₃ and extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo*. The residue was purified with silica gel chromatography. The pure product was obtained as a white solid in 67% yield.

Mp. 130-132 °C; $[\alpha]_D^{25}$ +5.0 (*c* 0.7, CHCl₃); IR (KBr) ν 3382, 1631, 1597, 1383, 1321, 1153, 938 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 2.35 (s, 3H), 2.84 (s, 3H), 4.44 (d, *J* = 10.2 Hz, 1H), 5.25 (d, *J* = 10.2 Hz, 1H), 7.01-7.17 (m, 12H), 7.58(d, *J* = 7.2 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.68, 29.84, 55.79, 67.51, 127.57, 127.97, 128.23, 128.60, 129.24, 129.64, 135.87, 137.10, 142.35, 143.35; HRMS Calcd. for C₂₂H₂₄N₂O₂S+Na⁺: 403.1456, found: 403.1445;

Synthesis of 1-((1*R*,2*R*)-2-(*N*-methyl-*N*-tosylamino)-1,2-diphenylethyl)-3-((1*R*,2*R*)-2-(dimethylamino)cyclohexyl)thiourea (3e)

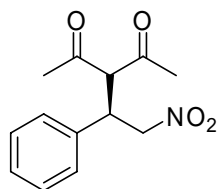


To a solution of (1*R*,2*R*)-*N*-methyl-1,2-diphenyl-*N*-tosylethane-1,2-diamine (64 mg, 0.168 mmol) in anhydrous THF (2 mL) was added (1*R*,2*R*)-2-isothiocyanato-*N,N*-dimethylcyclohexanamine (34 mg, 0.185 mmol) at room temperature. TLC indicated the completion of the reaction after about 4h. The reaction mixture was concentrated *in vacuo* and purified with silica gel chromatography. The pure product was obtained as a white solid in 78% yield.

Mp. 80-82 °C; $[\alpha]_D^{25}$ -25.0 (*c* 0.24, CHCl₃); IR (KBr) ν 3373, 3031, 2929, 2857, 1598, 1540, 1326, 1160 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.08-1.44 (m, 4H), 1.69-1.91 (m, 3H), 2.22-2.38 (m, 10H), 2.51 (m, 1H), 2.87 (s, 3H), 3.87(m, 1H), 5.25 (d, *J*=11.7 Hz, 1H), 6.32-6.39 (dd, *J* = 11.1 and 8.1 Hz, 1H), 6.75 (br, 1H), 6.92-7.31 (m, 12H), 7.58(d, *J* = 8.1, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 21.76, 24.67, 25.28, 29.92, 30.52, 32.94, 40.07, 54.89, 57.49, 64.97, 66.70, 127.19, 127.62, 128.27, 128.40, 128.60, 129.47, 129.84, 134.22, 137.48, 139.73, 143.60, 181.76; HRMS Calcd. for C₃₁H₄₁N₄O₂S₂: 565.2671, found: 565.2582.

General procedure for Micheal Addition of Acetylacetone to aryl nitroolefins

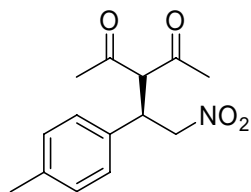
The catalyst (1.0 mg, 0.0015 mmol) was added to a vial containing 2,4-pentanedione (30.5 μ L, 0.30 mmol) and nitroolefin (0.15 mmol) in Et₂O (0.35 mL) at room temperature. TLC analysis indicated completion of the reaction after about 1-2h. Then the reaction mixture was concentrated *in vacuo*. The residue was purified by flash silica gel chromatography to afford the product.



(*R*)-3-(2-nitro-1-phenylethyl)pentane-2,4-dione (table 2, entry 1) :

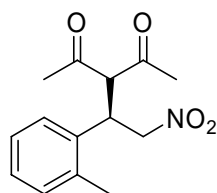
The title compound was prepared according to the general procedure as described above in 97% yield. ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.95 (s, 3H), 2.30 (s, 3H), 4.23-4.29 (m, 1H), 4.40 (d, *J* = 10.8 Hz, 1H), 4.63-4.65 (m, 2H) , 7.18-7.36 (m, 5H); HPLC (Chiralpak AS-H,

i-propanol/hexane = 15/85, 1.0mL/min, λ = 210 nm): t_{minor} = 16.7 min, t_{major} = 26.4 min, 97% *ee*.



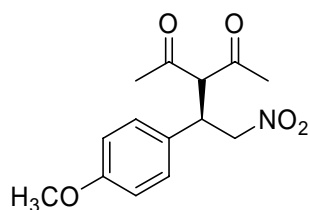
3-((*R*)-2-nitro-1-*p*-tolylethyl)pentane-2,4-dione (table 2, entry 2)

The title compound was prepared according to the general procedure as described above in 95% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.95 (s, 3H), 2.30 (s, 6H), 4.21-4.24 (m, 1H), 4.36 (d, J = 11.1 Hz, 1H), 4.59-4.61 (m, 2H), 7.05-7.14 (m, 4H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 21.33, 29.69, 30.68, 42.68, 71.08, 78.62, 128.00, 130.26, 133.00, 138.63, 201.40, 202.17; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 15/85, 1.0 mL/min, λ = 210 nm): t_{minor} = 12.1 min, t_{major} = 18.3 min, 95% *ee*.



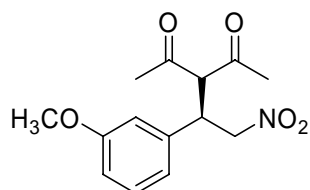
3-((*R*)-2-nitro-1-*o*-tolylethyl)pentane-2,4-dione (table 2, entry 3)

The title compound was prepared according to the general procedure, as described above in 95% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.91 (s, 3H), 2.31 (s, 3H), 2.42 (s, 3H), 4.41 (d, J = 9.3 Hz, 1H), 4.55-4.64 (m, 3H), 7.08-7.18 (m, 4H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 19.80, 30.45, 30.68, 37.90, 70.49, 78.02, 126.21, 127.04, 128.51, 131.88, 134.52, 136.94, 201.26, 201.92; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 15/85, flow rate 1.0 mL/min, λ = 210 nm): t_{minor} = 12.4 min, t_{major} = 17.8 min, *ee* = 98%.



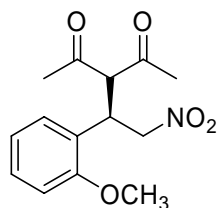
3-((*R*)-1-(4-methoxyphenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 4)

The title compound was prepared according to the general procedure, as described above in 93% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.94 (s, 3H), 2.30 (s, 3H), 3.78 (s, 3H), 4.19-4.23 (m, 1H), 4.34 (d, $J = 11.1$ Hz, 1H), 4.58-4.60 (m, 2H), 6.85 (d, $J = 8.7$ Hz, 2H), 7.10 (d, $J = 8.7$ Hz, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 29.81, 30.71, 43.00, 45.82, 70.95, 78.43, 128.17, 128.82, 129.61, 136.18, 201.26, 202.03; HPLC (Chiralpak AD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, $\lambda = 210$ nm) : $t_{\text{minor}} = 12.2$ min, $t_{\text{major}} = 17.7$ min, ee = 98%.



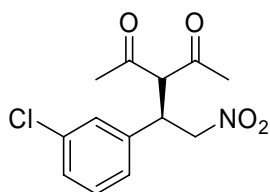
3-((R)-1-(3-methoxyphenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 5)

The title compound was prepared according to the general procedure, as described above in 96% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.97 (s, 3H), 2.30 (s, 3H), 3.78 (s, 3H), 4.21-4.25 (m, 1H), 4.37 (d, $J = 10.2$ Hz, 1H), 4.61-4.63 (m, 2H), 6.71-6.83 (m, 3H), 7.22 (s, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 29.85, 30.73, 43.00, 55.48, 70.83, 78.39, 113.80, 114.36, 120.12, 130.66, 137.78, 160.30, 201.23, 202.03; HPLC (Chiralpak AD-H, *i*-propanol/hexane = 15/85, flow rate 1.0 mL/min, $\lambda = 210$ nm) : $t_{\text{minor}} = 10.3$ min, $t_{\text{major}} = 13.1$ min, ee = 97%.



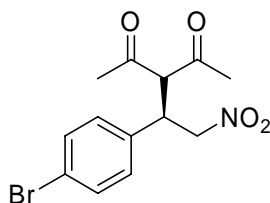
3-((R)-1-(2-methoxyphenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 6)

The title compound was prepared according to the general procedure, as described above in 97% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.94 (s, 3H), 2.28 (s, 3H), 3.89 (s, 3H), 4.49-4.61 (m, 3H), 4.75-4.82 (m, 1H), 6.88-6.92 (m, 2H), 7.07-7.09 (m, 1H), 7.25-7.29 (m, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 28.93, 29.95, 30.70, 39.03, 55.70, 69.28, 89.46, 111.45, 121.43, 130.03, 130.47, 201.82, 202.61; HPLC (Chiralpak AD-H, *i*-propanol/hexane = 3/97, flow rate 0.5 mL/min, $\lambda = 210$ nm) : $t_{\text{minor}} = 38.3$ min, $t_{\text{major}} = 41.7$ min, ee = 95%.



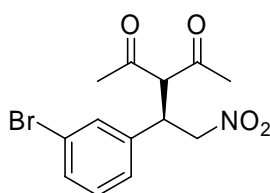
3-((R)-1-(3-chlorophenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 7)

The title compound was prepared according to the general procedure, as described above in 87% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 2.01 (s, 3H), 2.31 (s, 3H), 4.24 (m, 1H), 4.35 (d, J = 11.1 Hz, 1H), 4.62 (m, 2H), 7.08 (s, 1H), 7.20-7.29 (m, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 30.02, 30.77, 42.56, 70.58, 77.99, 126.36, 128.40, 129.13, 130.85, 135.44, 138.39, 200.71, 201.59; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 25/75, flow rate 1 mL/min, λ = 210 nm): t_{minor} = 11.5 min, t_{major} = 20.2 min, ee = 98%.



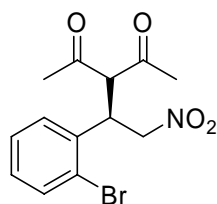
3-((R)-1-(4-bromophenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 8)

The title compound was prepared according to the general procedure, as described above in 97% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.98 (s, 3H), 2.30 (s, 3H), 4.22-4.26 (m, 1H), 4.34 (d, J = 11.1 Hz, 1H), 4.60-4.62 (m, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 29.95, 30.73, 42.41, 70.66, 78.07, 122.94, 129.88, 132.78, 135.30, 200.84, 201.64; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 15/85, flow rate 1 mL/min, λ = 210 nm): t_{minor} = 18.3min, t_{major} = 32.2 min, ee = 95%.



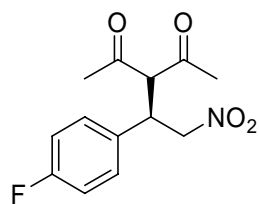
3-((R)-1-(3-bromophenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 9)

The title compound was prepared according to the general procedure, as described above in 96% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 2.01 (s, 3H), 2.31 (s, 3H), 4.21-4.25 (m, 1H), 4.35 (d, $J = 11.1$ Hz, 1H), 4.59-4.64 (m, 2H), 7.11-7.26 (m, 2H), 7.36 (s, 1H), 7.44 (d, $J = 7.2$ Hz, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 30.03, 30.77, 42.51, 70.58, 77.97, 123.57, 126.83, 131.11, 131.28, 132.05, 138.66, 200.70, 201.58; HPLC (Chiralpak AS-H, *i*-propanol/hexane=15/85, flow rate 1 mL/min, $\lambda = 210$ nm): $t_{\text{minor}} = 19.2$ min, $t_{\text{major}} = 36.2$ min, ee = 96%.



3-((R)-1-(2-bromophenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 10) .

The title compound was prepared according to the general procedure, as described above in 97% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 2.05 (s, 3H), 2.30 (s, 3H), 4.59-4.87 (m, 4H), 7.12-7.20 (m, 2H), 7.26-7.32 (m, 1H), 7.63 (d, $J = 7.2$ Hz, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 28.57, 31.28, 41.26, 69.37, 76.50, 128.54, 129.09, 130.26, 134.28, 135.22, 206.99; HPLC (Chiralpak AD-H, *i*-propanol/hexane = 3/97, flow rate 0.5 mL/min, $\lambda = 210$ nm): $t_{\text{minor}} = 35.1$ min, $t_{\text{major}} = 37.2$ min, ee = 96%.

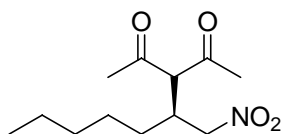


3-((R)-1-(4-fluorophenyl)-2-nitroethyl)pentane-2,4-dione (table 2, entry 11)

The title compound was prepared according to the general procedure, as described above in 96% yield. ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 1.95 (s, 3H), 2.28 (s, 3H), 4.22-4.34 (m, 2H), 4.59-4.60 (m, 2H), 6.99-7.04 (m, 2H), 7.16 (m, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 29.96, 30.68, 42.28, 70.95, 78.39, 116.50, 116.80, 129.87, 129.98, 131.98, 200.99, 201.77; HPLC (Chiralcel OD-H, *i*-propanol/hexane = 10/90, flow rate 1 mL/min, $\lambda = 210$ nm): $t_{\text{minor}} = 30.3$ min, $t_{\text{major}} = 33.4$ min, ee = 99%.

General procedure for Micheal Addition Reaction of Acetylacetone to Alkyl nitroolefins

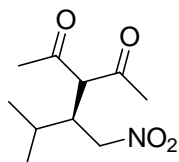
The catalyst (5.1 mg, 0.0075 mmol) was added to a vial containing 2,4-pentanedione (30.5 μ L, 0.30 mmol) and alkyl nitroolefin (0.15 mmol) in Et₂O (0.35 mL) at room temperature. After 16-28h of stirring, TLC analysis indicated completion of the reaction. The reaction mixture was concentrated *in vacuo*. The residue was purified by flash silica gel chromatography to afford the corresponding product.



3-((S)-1-nitroheptan-2-yl)pentane-2,4-dione (table 2, entry 12)

The title compound was prepared according to the general procedure, as described above in 85% yield.

¹H NMR (CDCl₃, TMS, 300 MHz) δ 0.86-0.90 (t, *J* = 5.7 Hz, 3H), 1.27-1.44 (m, 8H), 2.27 (s, 3H), 2.28 (s, 3H), 2.82-2.86 (m, 1H), 4.00 (d, *J* = 8.7 Hz, 1H), 4.46-4.57 (m, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 14.15, 22.61, 26.54, 29.65, 30.08, 31.24, 31.63, 37.24, 69.61, 75.96, 202.61, 203.05; HRMS Calcd. for C₁₂H₂₁NO₄: 243.1471, found:243.1477; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 5/95, flow rate 0.8 mL/min, λ = 210 nm): *t*_{minor} = 9.5 min, *t*_{major} = 21.5 min, ee = 85%.

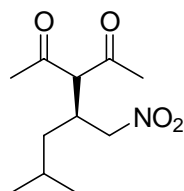


3-((S)-3-methyl-1-nitrobutan-2-yl)pentane-2,4-dione (table 2, entry 13) :

The title compound was prepared according to the general procedure, as described above in 80% yield.

¹H NMR (CDCl₃, TMS, 300 MHz) δ 0.91 (d, *J* = 6.6 Hz, 3H), 0.98 (d, *J* = 6.6 Hz, 3H), 1.72-1.81 (m, 1H), 2.24 (s, 3H), 2.31 (s, 3H), 2.81-2.84 (m, 1H), 4.12 (d, *J* = 6.6 Hz, 1H), 4.40-4.46 (m, 1H),

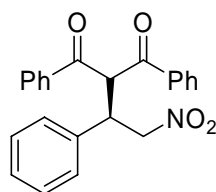
4.55-4.62 (m, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 18.77, 21.03, 21.83, 29.78, 31.03, 42.86, 68.30, 74.92; HPLC (Chiralpak AD-H, *i*-propanol/hexane = 5/95, flow rate 1 mL/min, λ = 210 nm): t_{major} = 11.0 min, t_{minor} = 11.9 min, ee = 81%.



3-((*S*)-4-methyl-1-nitropentan-2-yl)pentane-2,4-dione (table 2, entry 15) :

The title compound was prepared according to the general procedure, as described above in 83% yield.

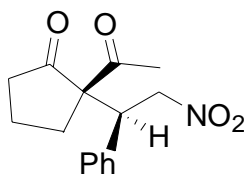
^1H NMR (CDCl_3 , TMS, 300 MHz) δ 0.89-1.03 (m, 6H), 1.32-1.40 (m, 1H), 1.70 (s, 1H), 2.21-2.34 (m, 6H), 2.90 (m, 1H), 3.98 (d, J = 8.1 Hz, 1H), 4.51 (m, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 21.08, 22.98, 24.74, 29.69, 30.96, 34.62, 38.22, 69.16, 75.67, 202.25, 202.78; HRMS Calcd. for $\text{C}_{11}\text{H}_{19}\text{NO}_4$: 229.1314, found:229.1318; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 10/90, flow rate 1 mL/min, λ = 210 nm): t_{minor} = 7.2 min, t_{major} = 13.3 min, ee = 82%.



2-((*R*)-2-nitro-1-phenylethyl)-1,3-diphenylpropane-1,3-dione

The title compound was prepared according to the general procedure, as described above in 95% yield.

^1H NMR (CDCl_3 , TMS, 300 MHz) δ 4.61-4.64 (m, 1H), 5.00 (d, J = 7.2 Hz, 2H), 5.84 (d, J = 8.1 Hz, 1H), 7.19-7.23 (m, 5H), 7.34-7.40 (m, 4H), 7.49-7.55 (m, 2H), 7.79 (d, J = 7.2 Hz, 1H), 7.87 (d, J = 8.1 Hz, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 44.23, 60.04, 77.51, 127.42, 128.48, 128.85, 129.03, 129.08, 129.22, 132.74, 134.08, 134.35, 135.98, 136.35, 136.97, 193.84, 194.47; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1 mL/min, λ = 254 nm): t_{minor} = 20.97 min, t_{major} = 29.07 min, ee = 85%.



2-acetyl-2-(3-nitro-2-phenylpropyl)cyclopentanone

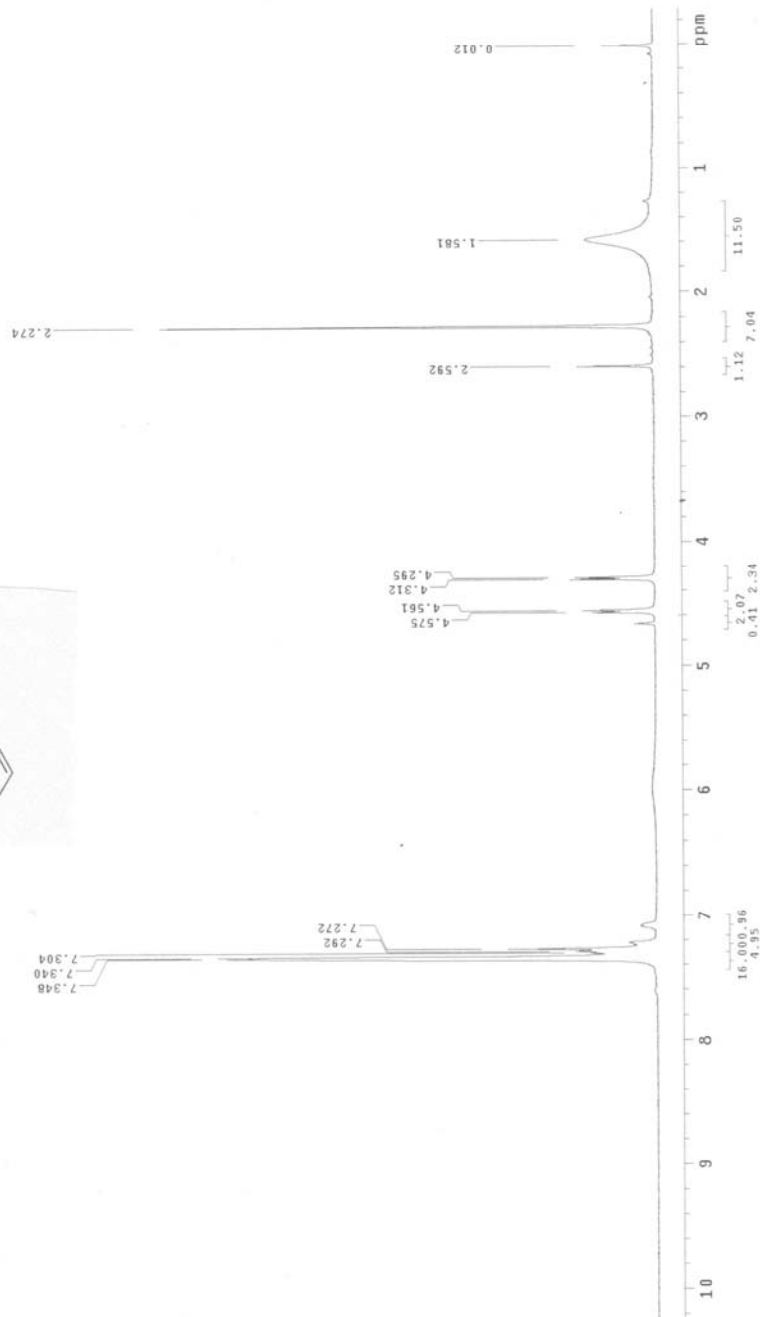
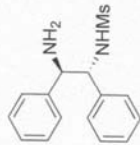
The catalyst (5.0mg, 0.0075mmol) was added to a vial containing nitroolefin (0.15 mmol) in dichloromethane (0.25 mL) at -50 °C, and then 2-acetylcyclopentanone (0.15 mmol) was added. TLC analysis indicated completion of the reaction after about 10h. The reaction mixture was then passed through a plug of silica gel for the removal of the catalyst. The plug was eluted with ethyl acetate (3-4 mL). The combined filtrate was concentrated in *vacuo* and the diastereoselectivity of the crude product was determined as 85:15 according to the crude ¹H NMR. The residue was purified by flash silica gel chromatography to afford the product in 92% yield.

¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.65-1.80 (m, 3H), 1.93-2.03 (m, 1H), 2.15-2.28 (m, 1H), 2.34 (s, 3H), 2.56-2.61 (m, 1H), 4.40 (dd, *J* = 3.9 Hz, 1H), 4.50 (dd, *J* = 3.9 Hz, 1H), 4.87(t, *J* = 11.7 Hz, 1H), 7.20-7.32 (m, 5H); HPLC (Chiralcel OD-H, *i*-propanol/hexane = 20/80, flow rate 1 mL/min, λ = 220 nm): *t*_{minor} = 16.06 min, *t*_{major} = 72.09 min, ee = 96% (major diastereomer).

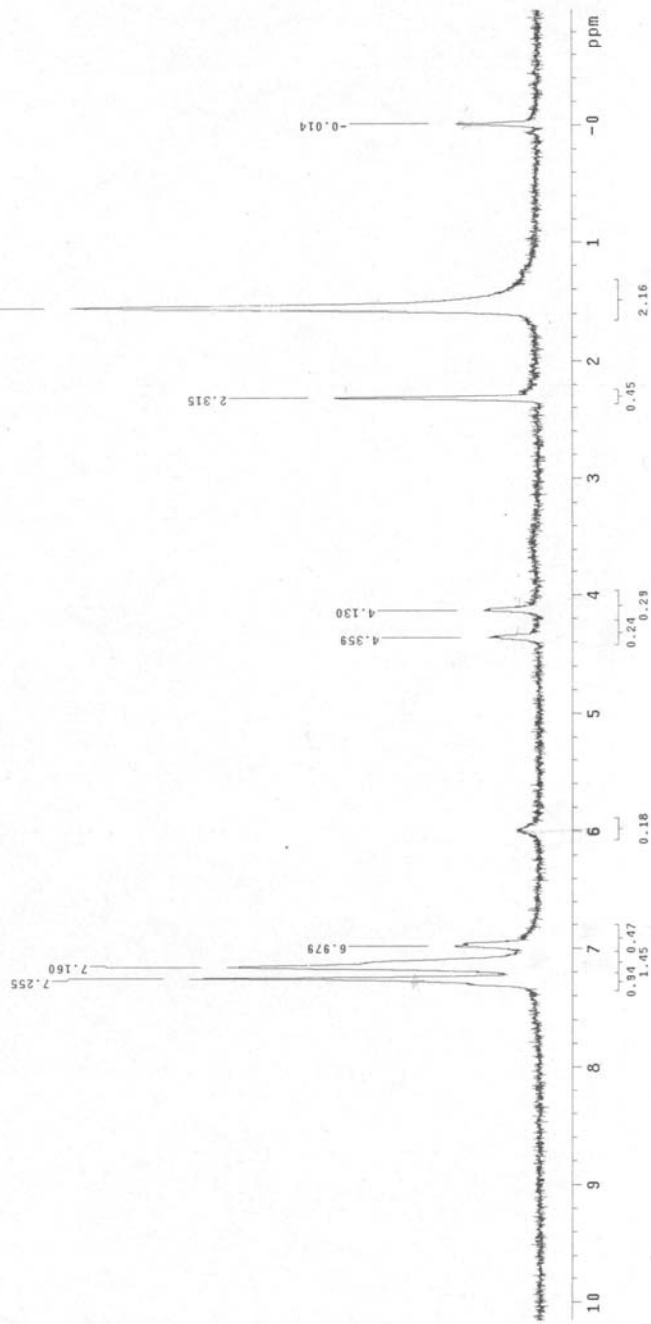
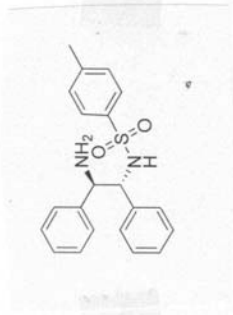
References

1. (a) L. S. Santos, R. A. Pilli, V. H. Rawal, *J. Org. Chem.* 2004, **69**, 1283. (b) D. Chen, Y.-C. Xue, X. Cui, Q.-W. Wang, J. Zhu, J.-G. Deng, *J. Org. Chem.* 2005, **70**, 3584.

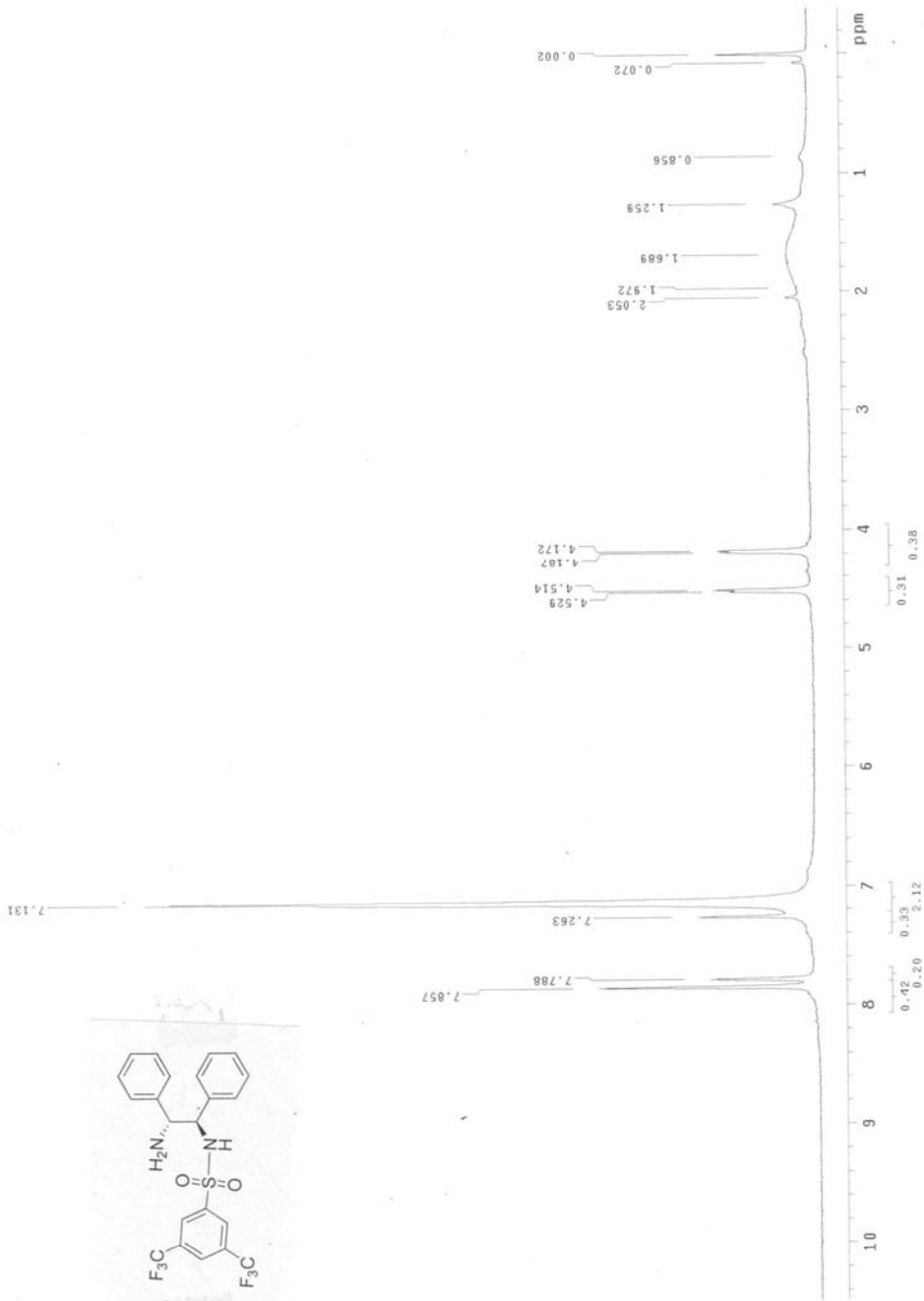
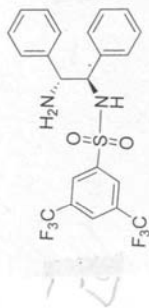
221256



9-3-10



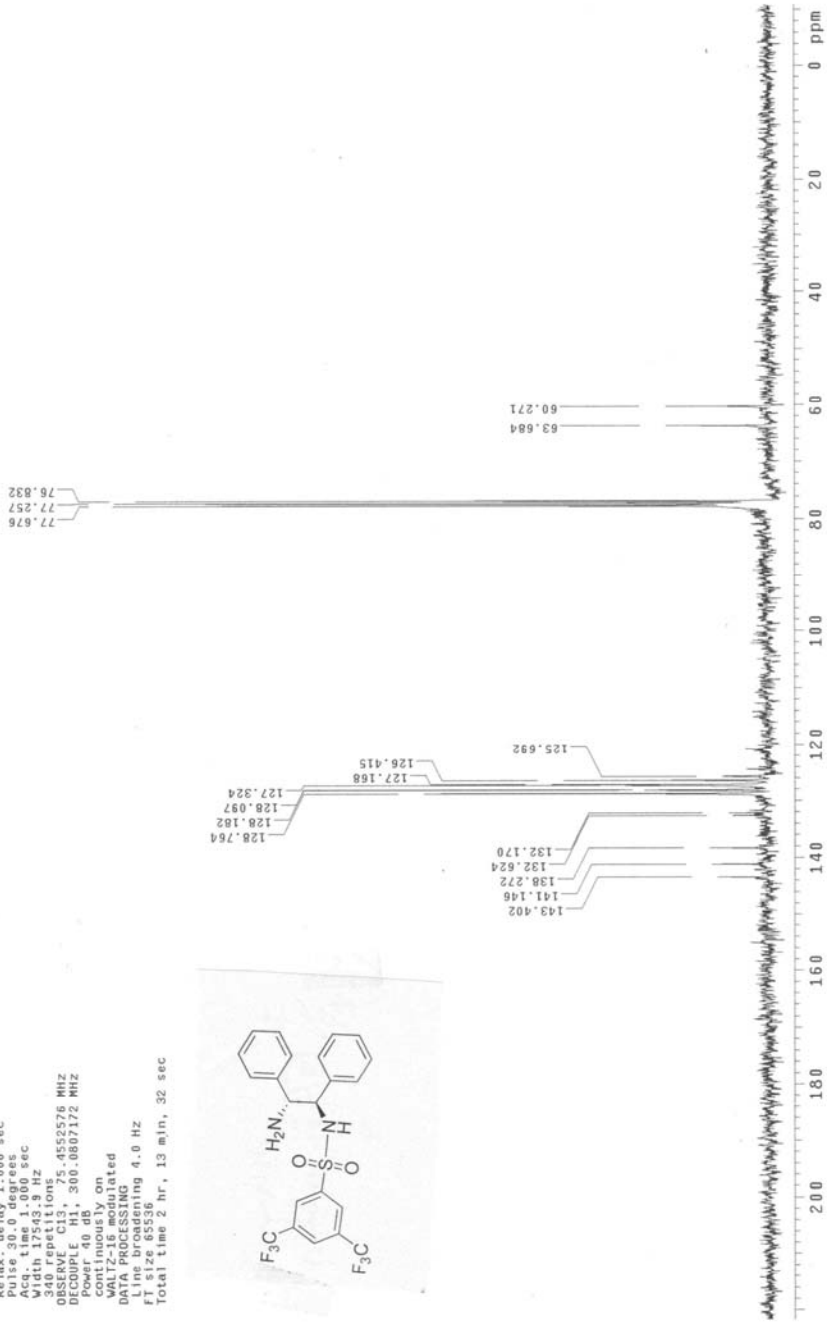
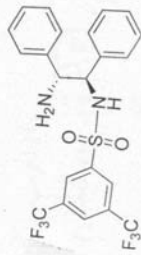
ZN1530A



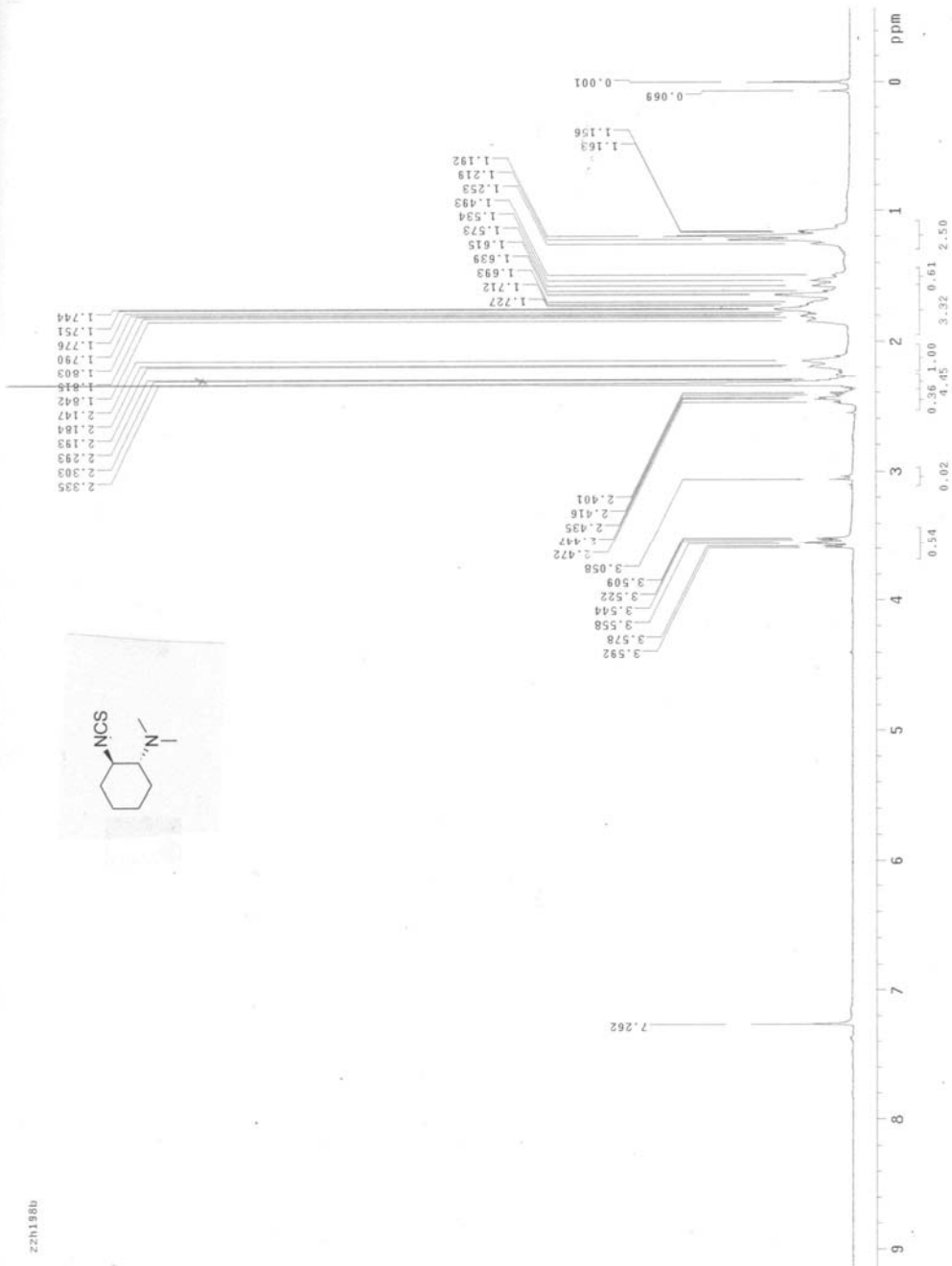
z2h12c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. time 0.000 sec
Acq. time 1.000 sec
Width 17543.8 Hz
340 repetitions
OBSERVE C13, 75.4552575 MHz
PULSE P1, 300.0007172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 size 65536
Total time 2 hr, 13 min, 32 sec



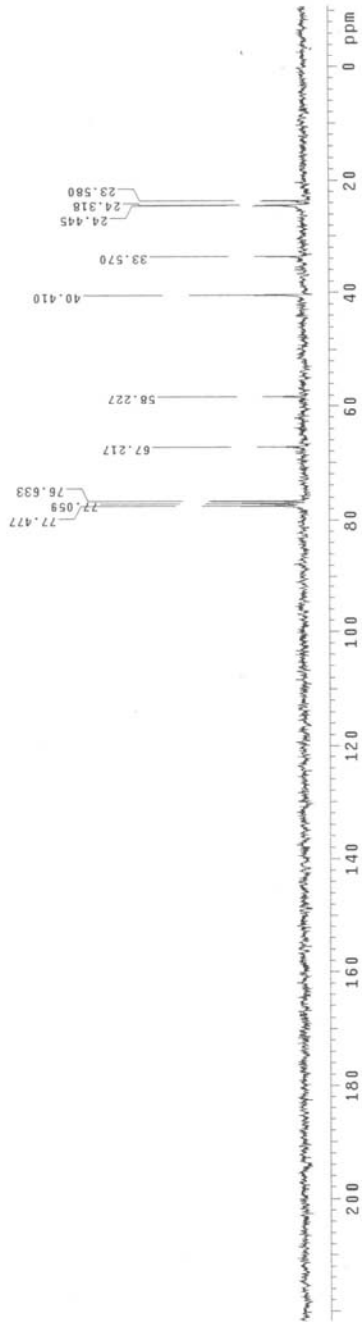
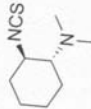
22h186b



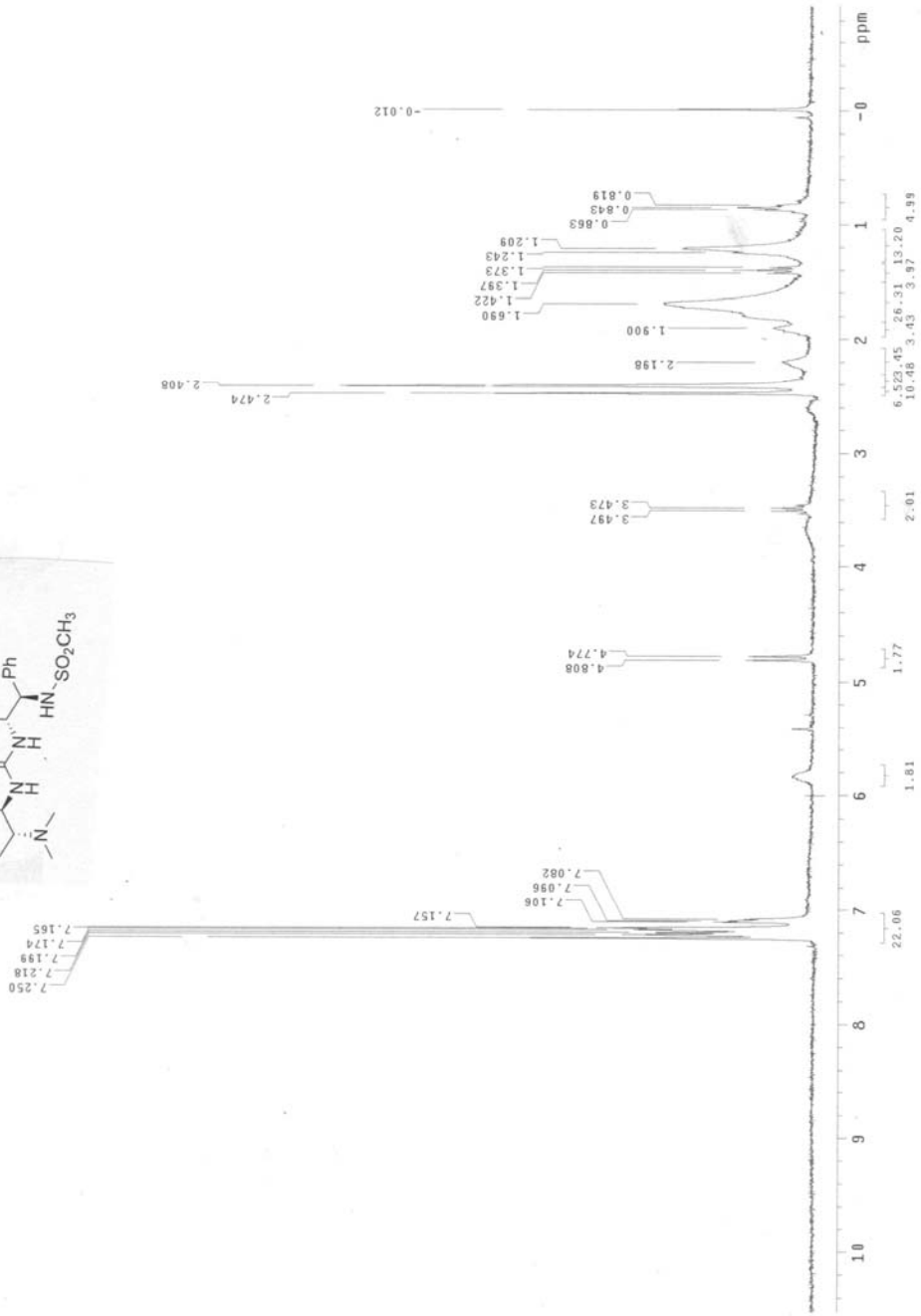
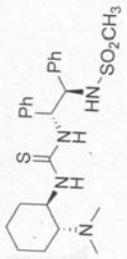
z2h14c

Solvent: CDCl3
Ambient Temperature
Mercury-300BB5 "mercury300"

Relax. delay 1.000 sec
Pulse 30.0 degree
Pulse program zgpg30
Width 17543.8 Hz
28 repetitions
OBSERVE C13, 75.4552576 MHz
DUAL F1 H, 300.0807172 MHz
Power 40 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 resolution 4.0 Hz
FT size 65536
Total time 2 hr, 13 min, 32 sec



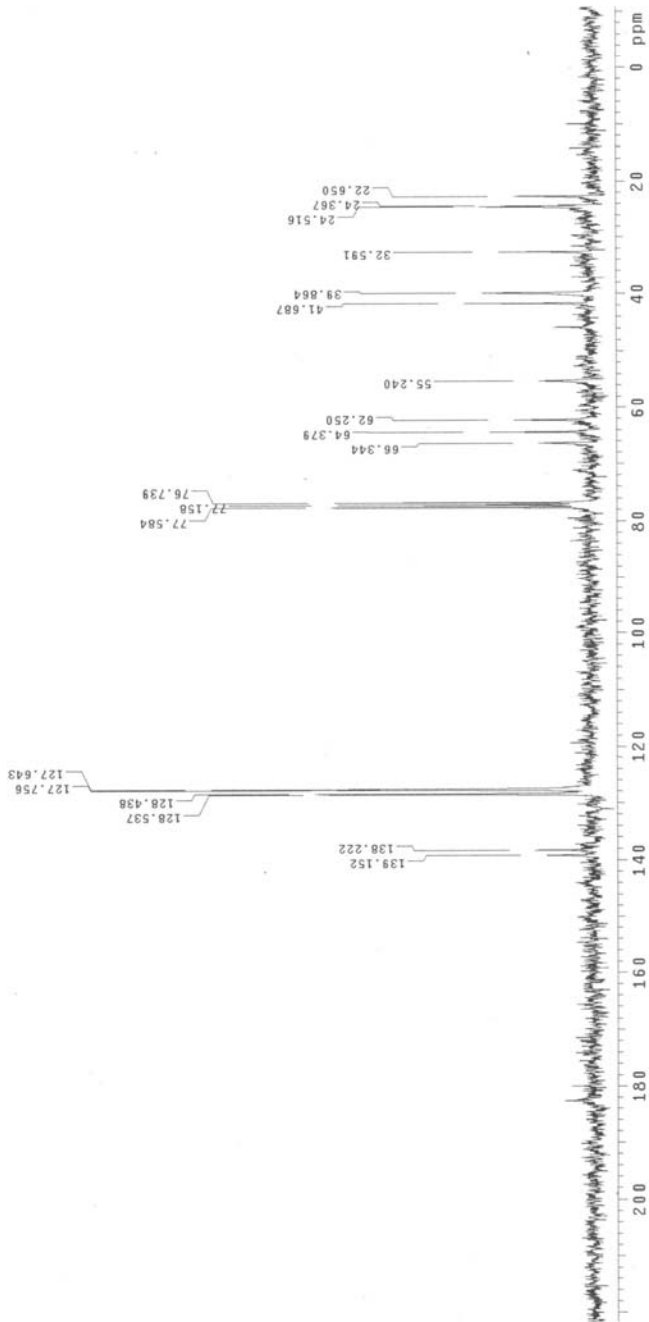
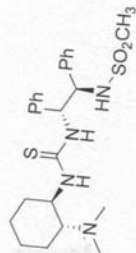
zzh261c



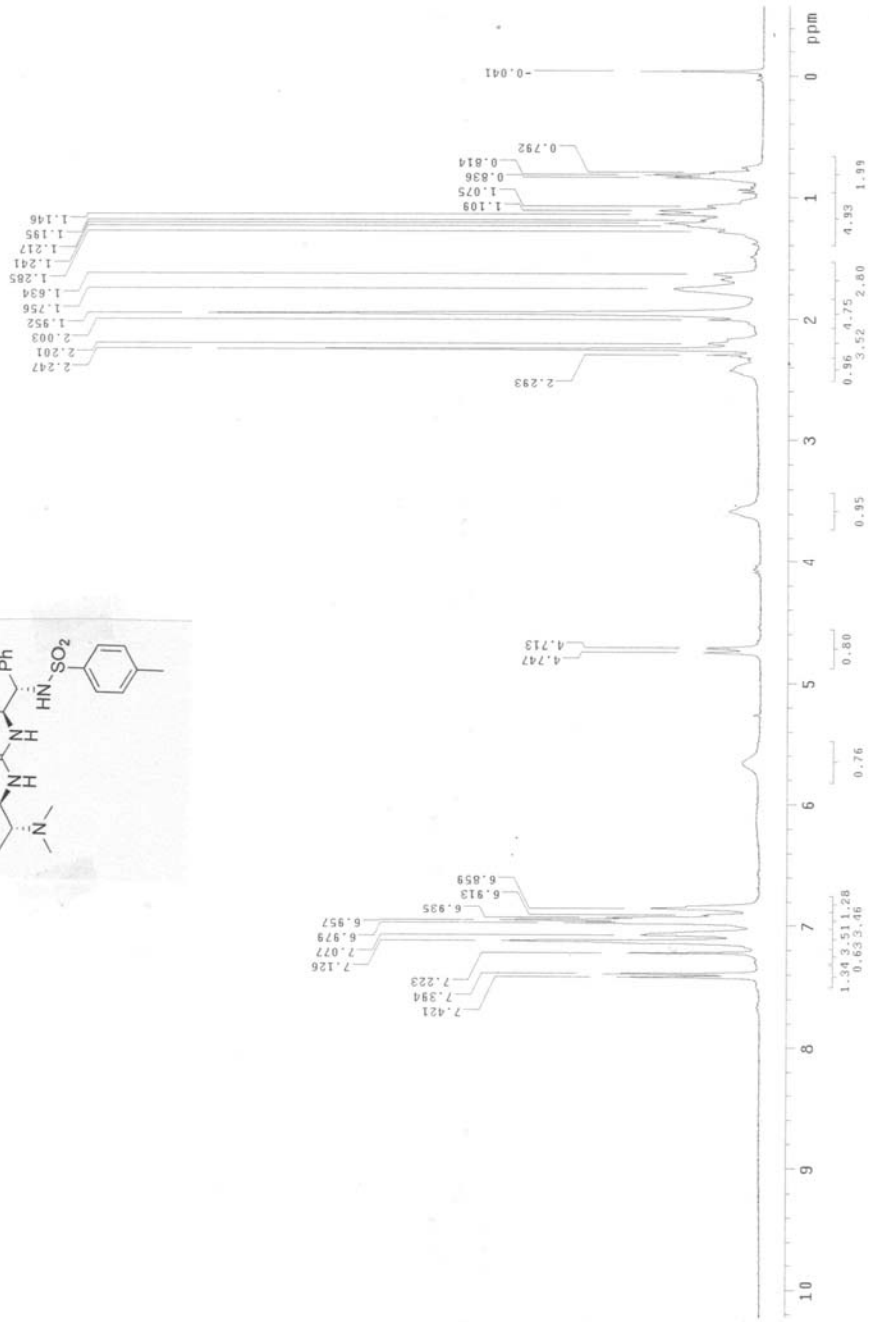
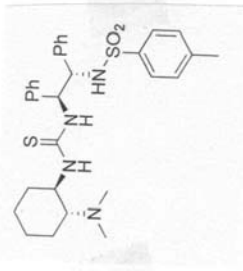
z2h13c

Solvent: CDCl3
Ambient Temperature
Mercury-300BBB "mercury300"

Relax. delay 1.000 sec
Pulse 30.0 degree
Acq. time 0.00 sec
Width 17543.9 Hz
80 repetitions
OBSERVE C13, 75.4552576 MHz
DUMPER 10, 300.0807172 MHz
Power 40 db.
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 resolution 4.0 Hz
FT size 65536
Total time 2 hr, 13 min, 32 sec



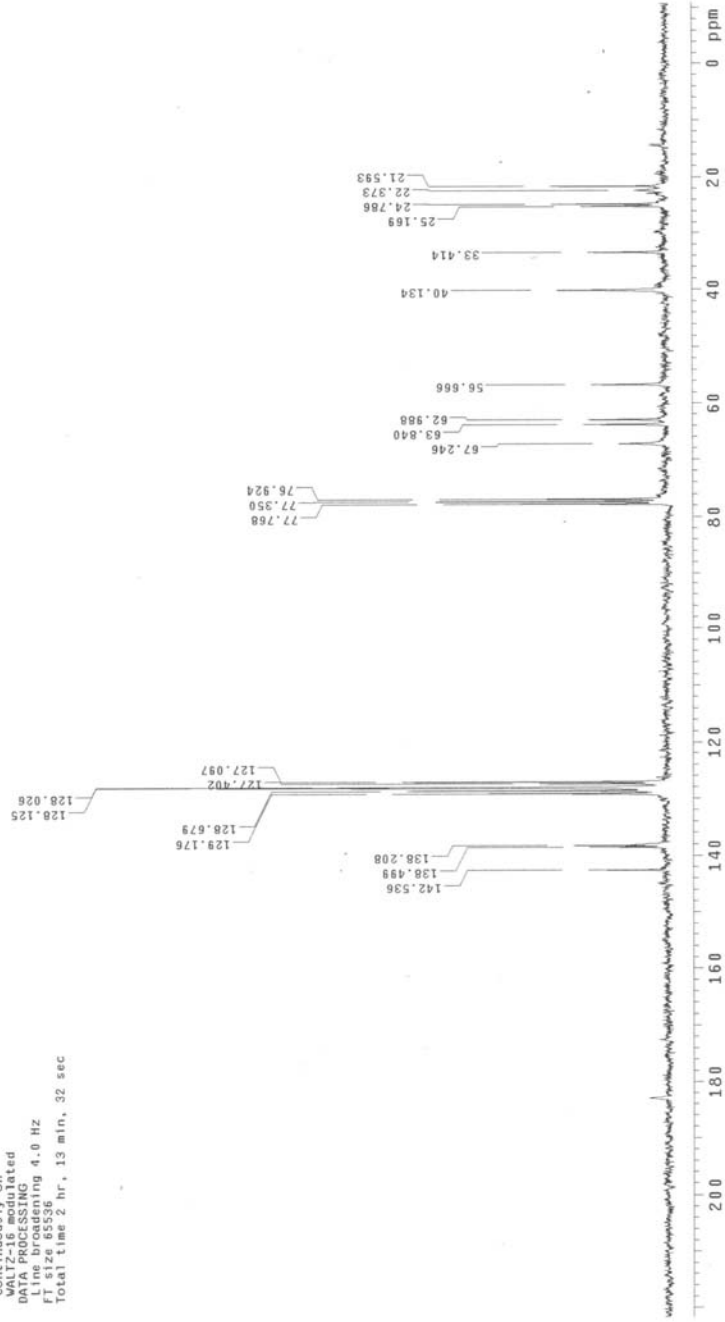
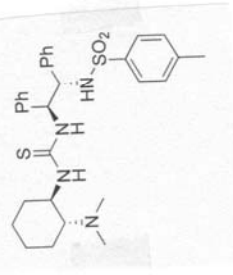
2210281



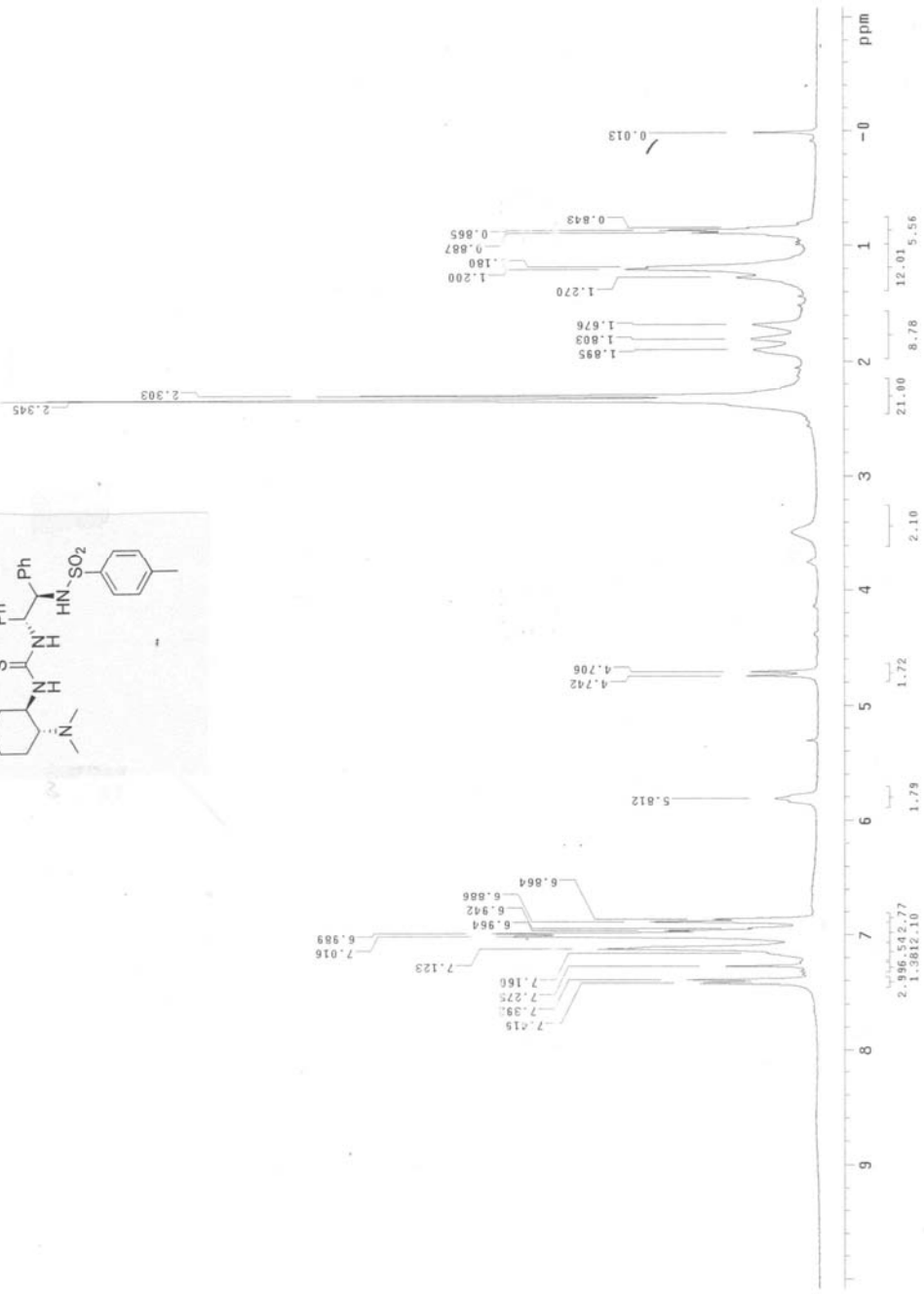
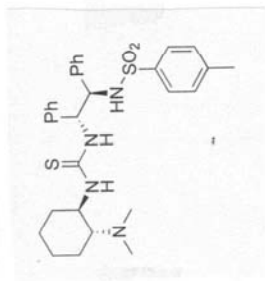
zzh15c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 30.0 degree
Pulse width 10.00 sec
Width 17543.8 Hz
288 repetitions
OBSERVE C13, 75.4552576 MHz
DPCUPLE C13, 300.6807172 MHz
Power 40 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 spin 4.0 Hz
FT size 65536
Total time 2 hr, 13 min, 32 sec



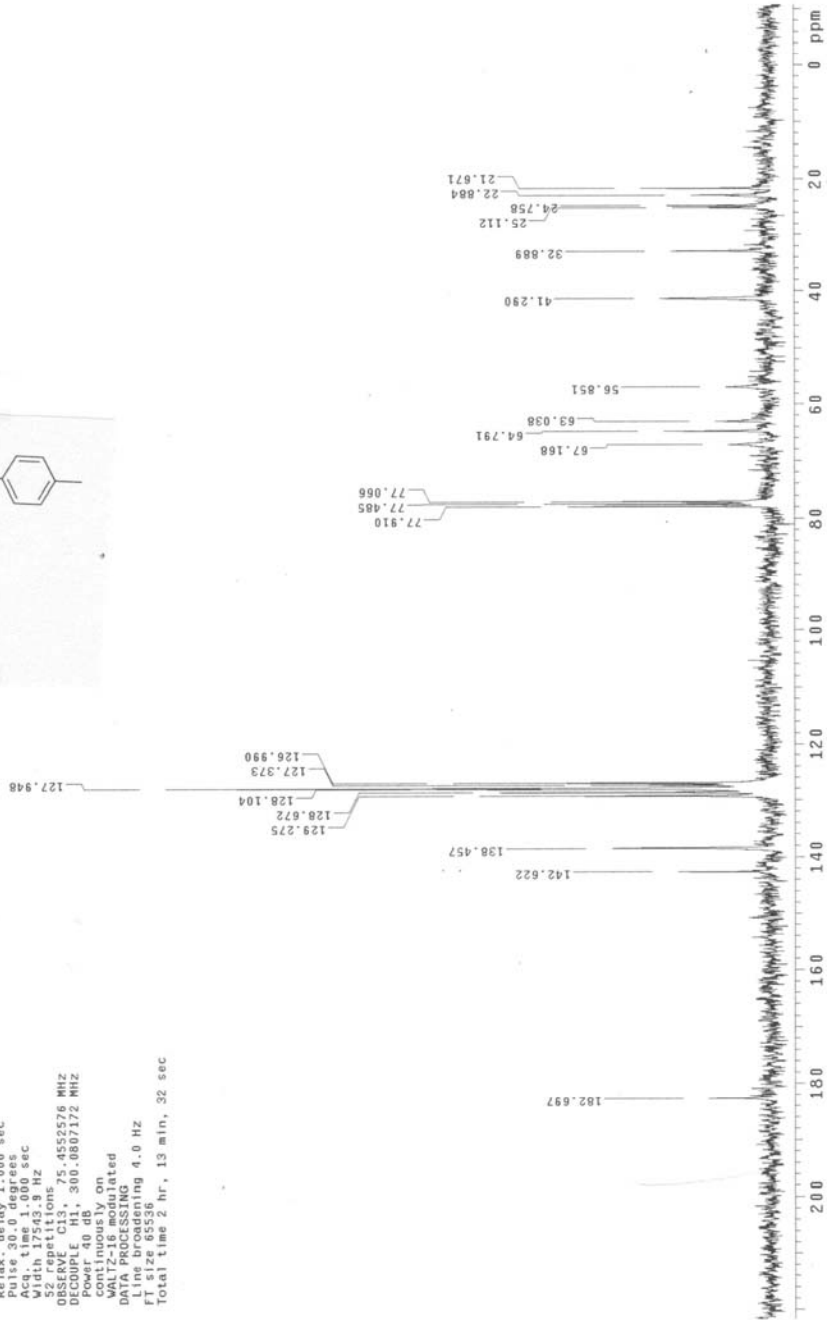
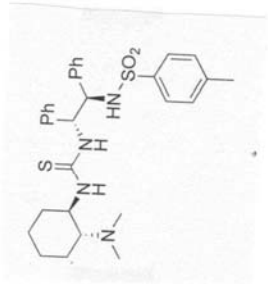
ZZ12.05



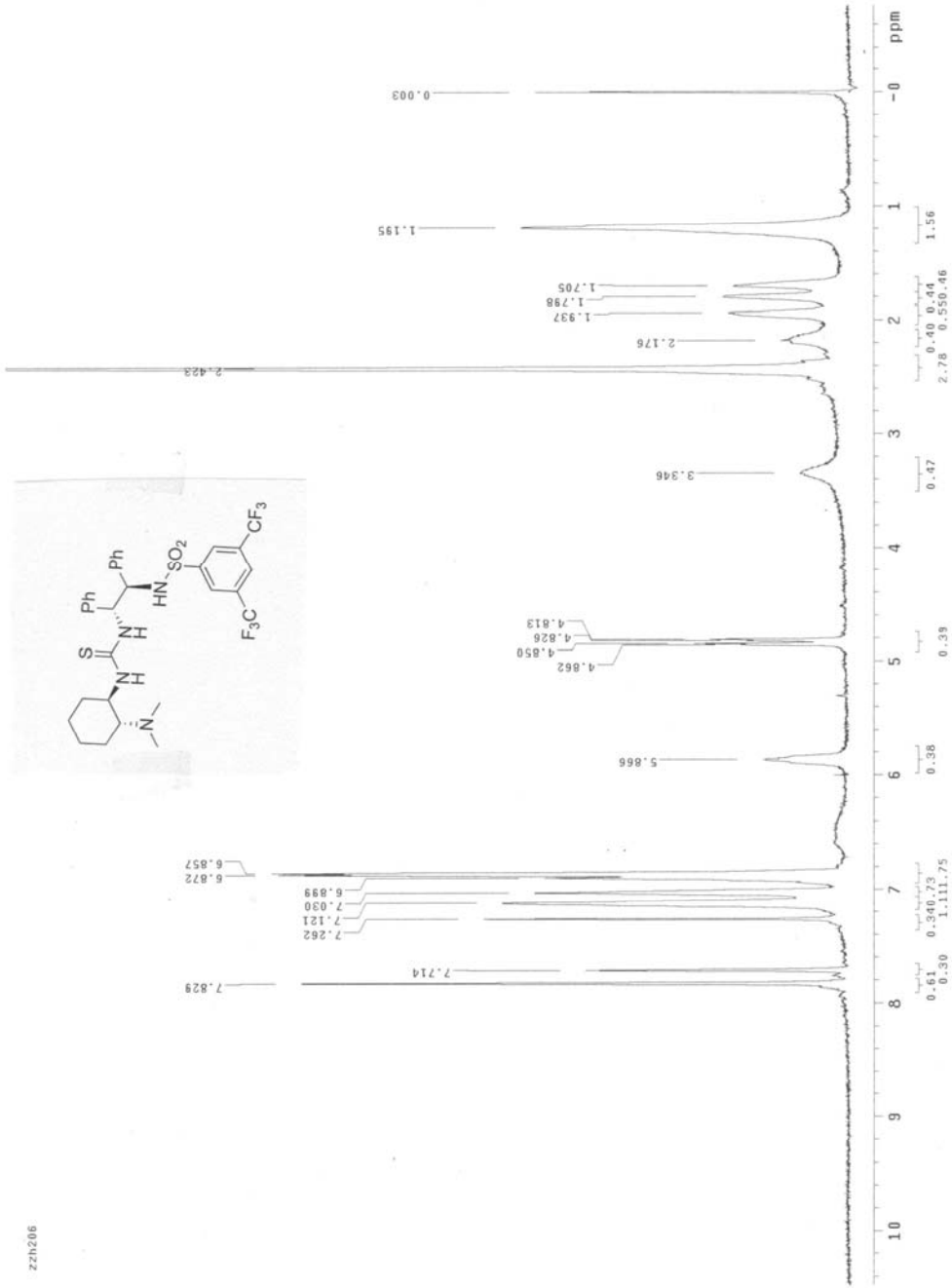
zzh1ic

Solvent: CDCl3
Ambient Temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. 300.000 MHz
Acq. time 1.000 sec
Width 17543.8 Hz
52 repetitions
OBSERVE C13, 75.4552576 MHz
PULSE C13, 300.0001712 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
SFO 300.0001712 MHz
FT size 65536
Total time 2 hr, 13 min, 32 sec



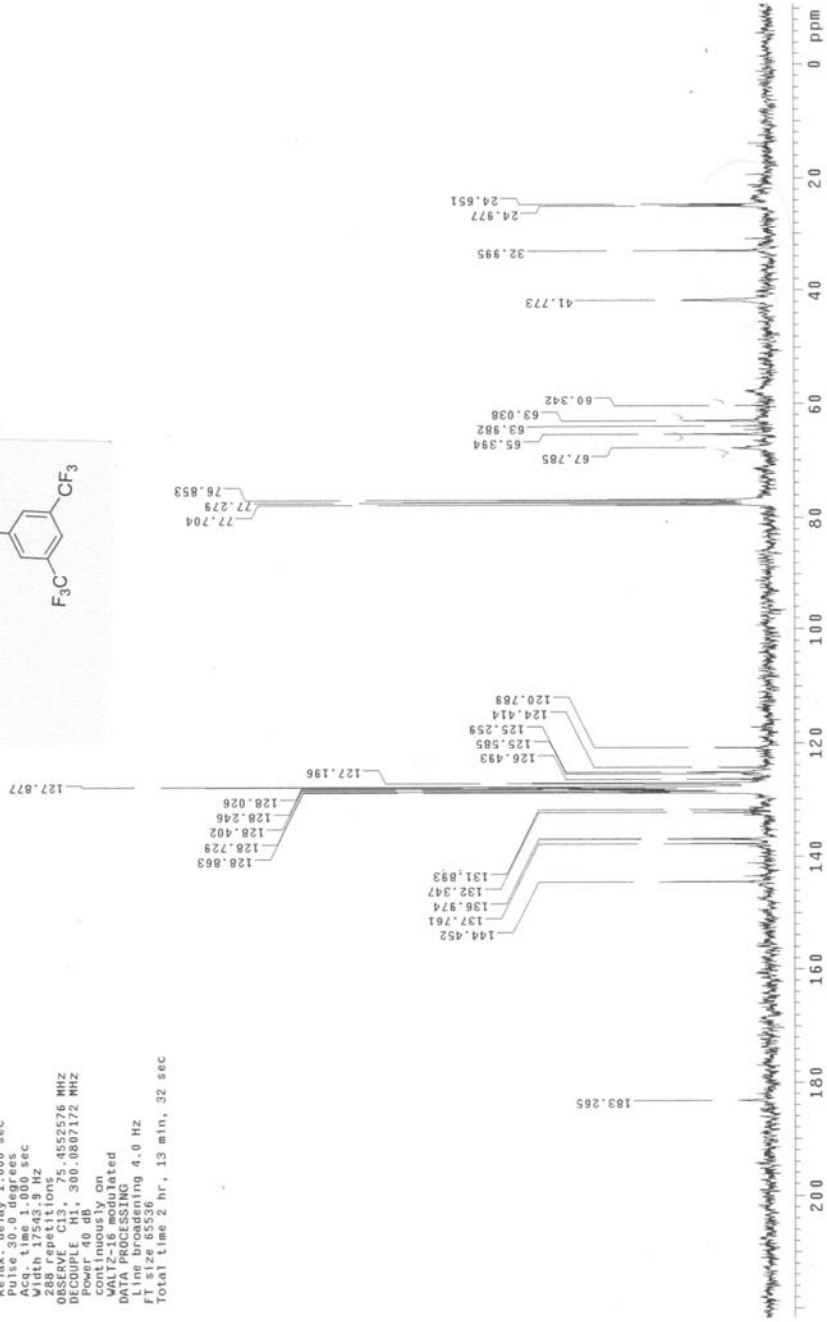
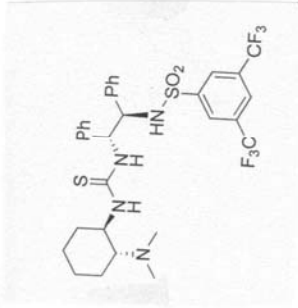
9024206



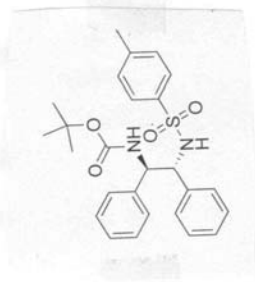
zzh10c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. time 1.000 sec
Width 17543.9 Hz
288 repetitions
OBSERVE C13, 75.4552576 MHz
PULSE P1, 300.0007172 MHz
Power 40 dB, continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536, 4.0 Hz
Total time 2 hr, 13 min, 32 sec



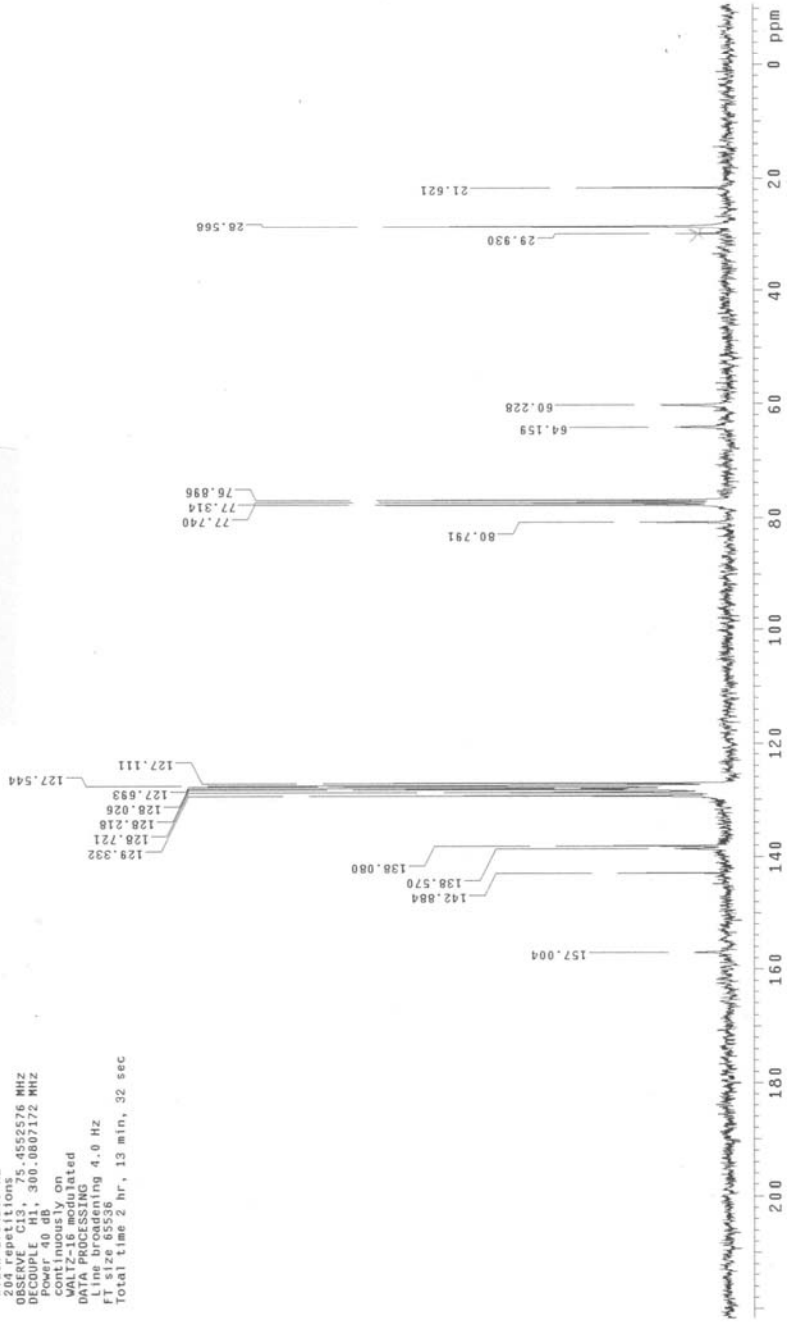
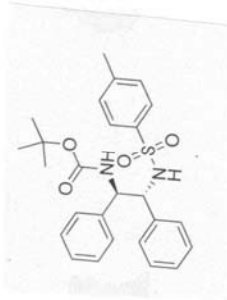
2212104



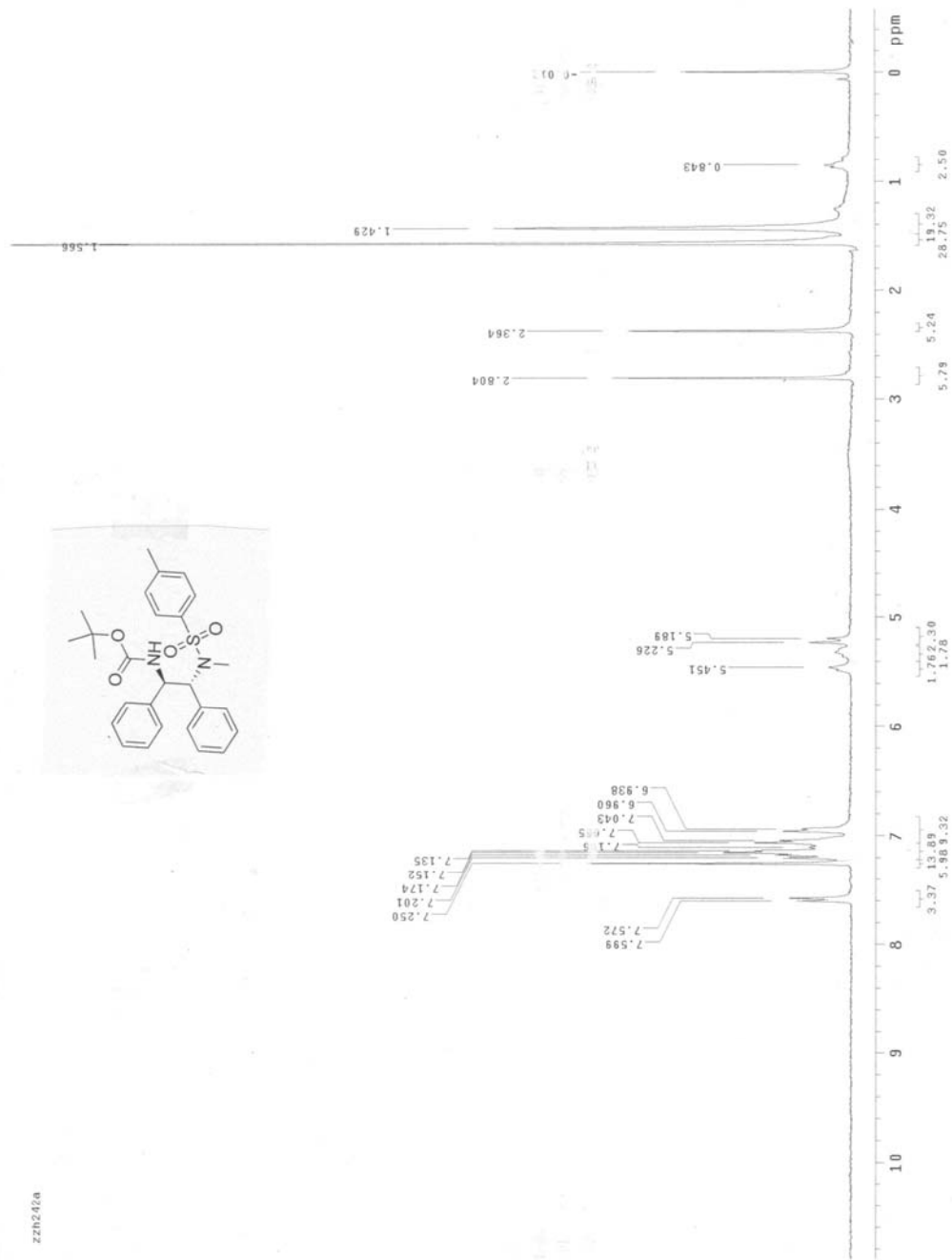
zzh6c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. delay 0.000 sec
Acq. time 1.000 sec
Width 17543.9 Hz
204 repetitions
OBSERVE C13, 5.855576 MHz
PULSE P1, 300.0007172 MHz
Power 40 dB, continuously on
continuously on
WALTZ-16 modulated
D1 time 0.000000 sec
D1 time bracketing 4.0 Hz
FT size 65536
Total time 2 hr, 13 min, 32 sec

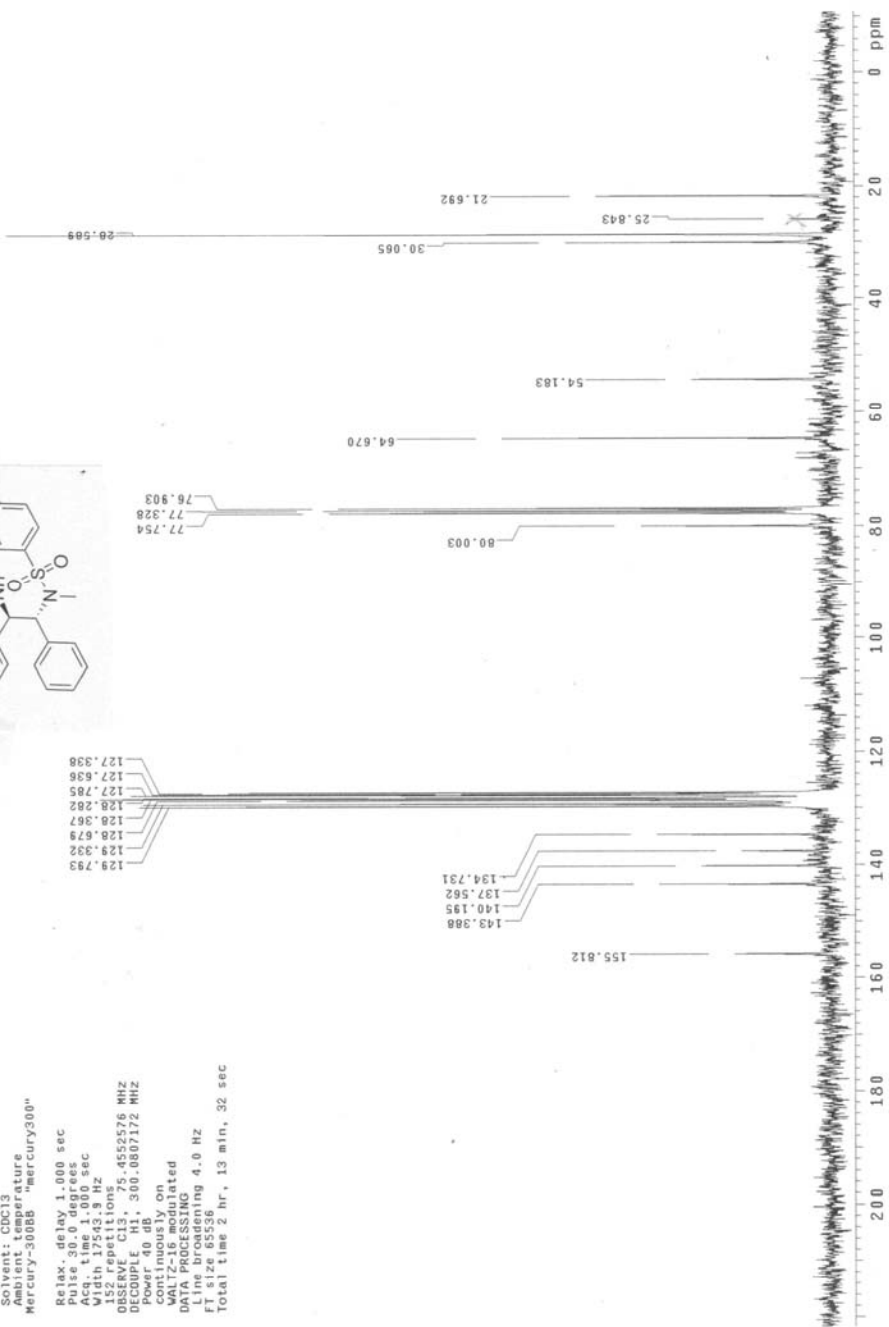
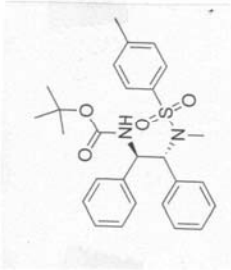


zzh242a

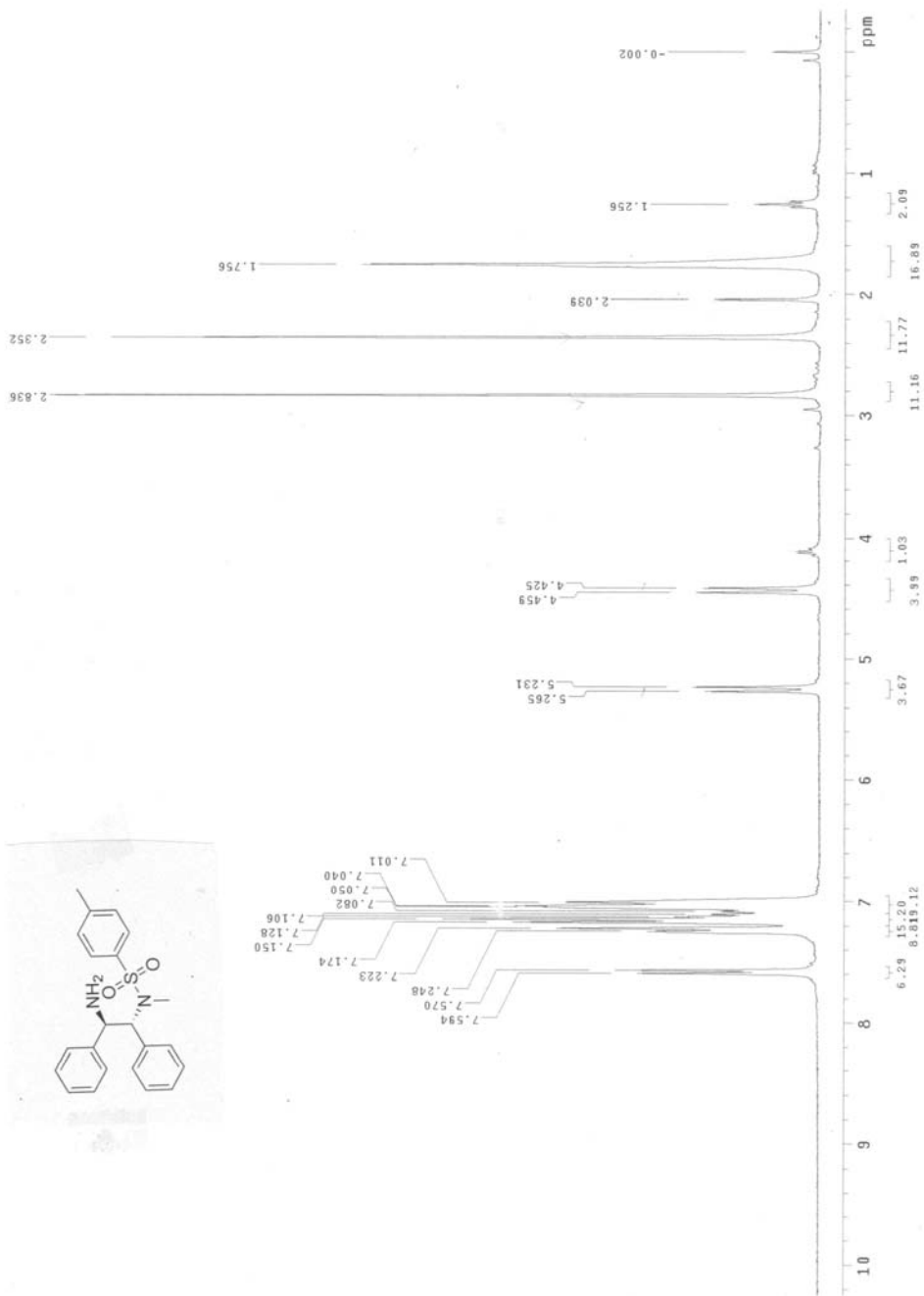
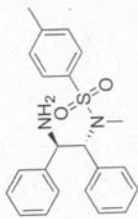


zzh7c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Acq. delay 0.000 sec
Acq. time 1.000 sec
Width 17543.9 Hz
152 repetitions
OBSERVE C13, 75.655576 MHz
OBSERVE H1, 300.007172 MHz
Power 40 db
continuously on
VALTZ-16 modulated
DATA PROCESSING
Reference freq 4.0 Hz
FT size 65536
Total time 2 hr, 13 min, 32 sec



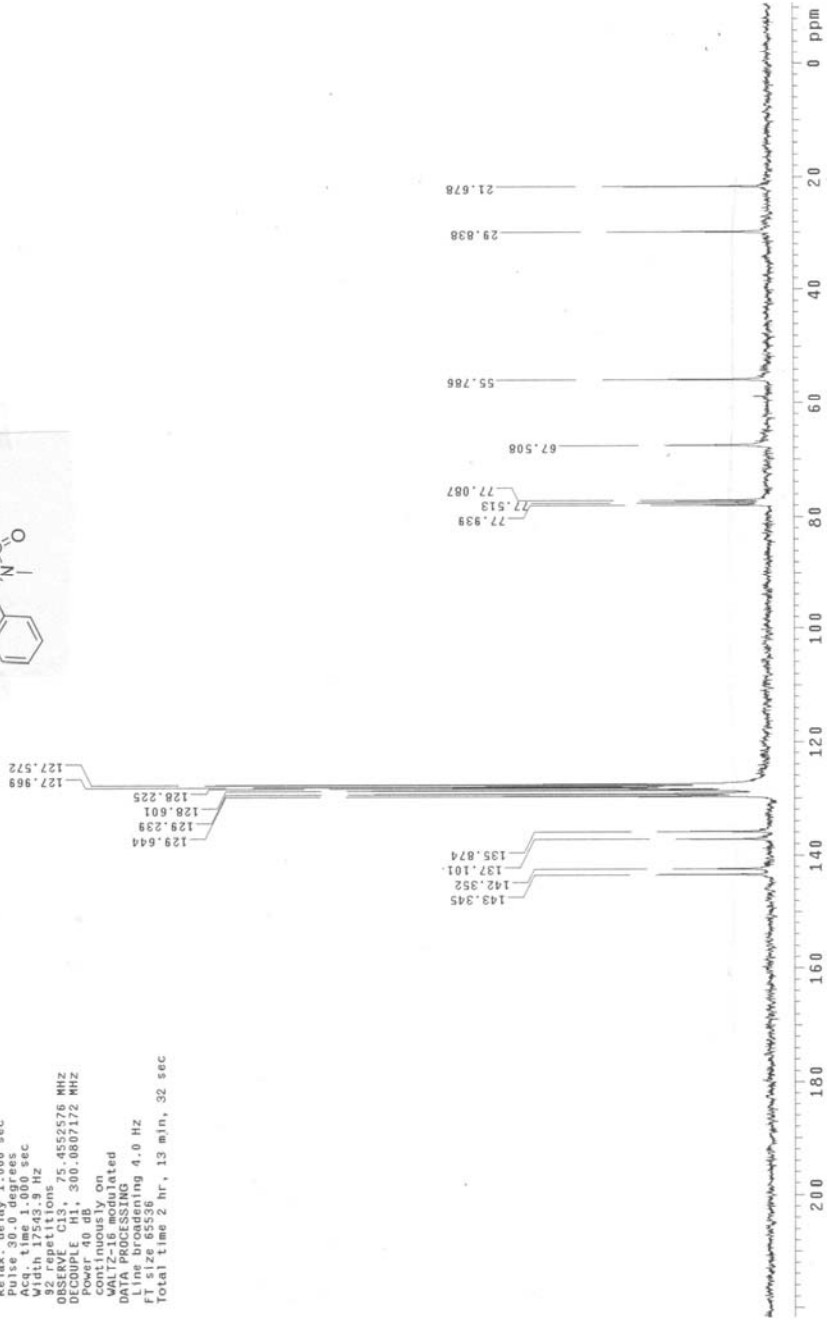
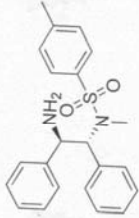
zzh250a



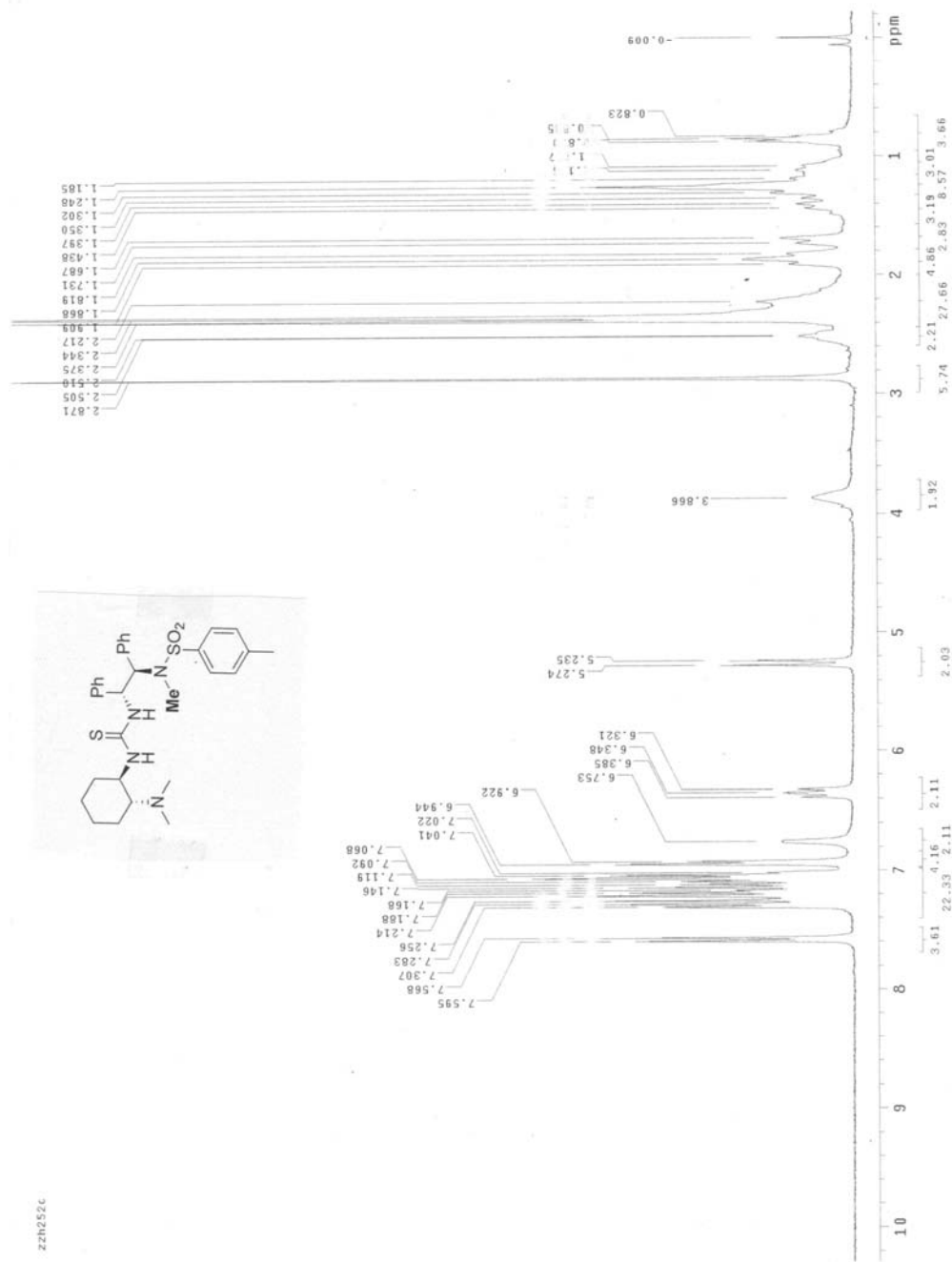
z2h8c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. time 3.000 sec
Acq. date 1.000 sec
Width 17543.9 Hz
32 repetitions 75.4552526 MHz
OBSERVE C13, 300.0007172 MHz
Pulse 12.000 sec
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
F2 size 65536
FT size 65536
Total time 2 hr, 13 min, 32 sec



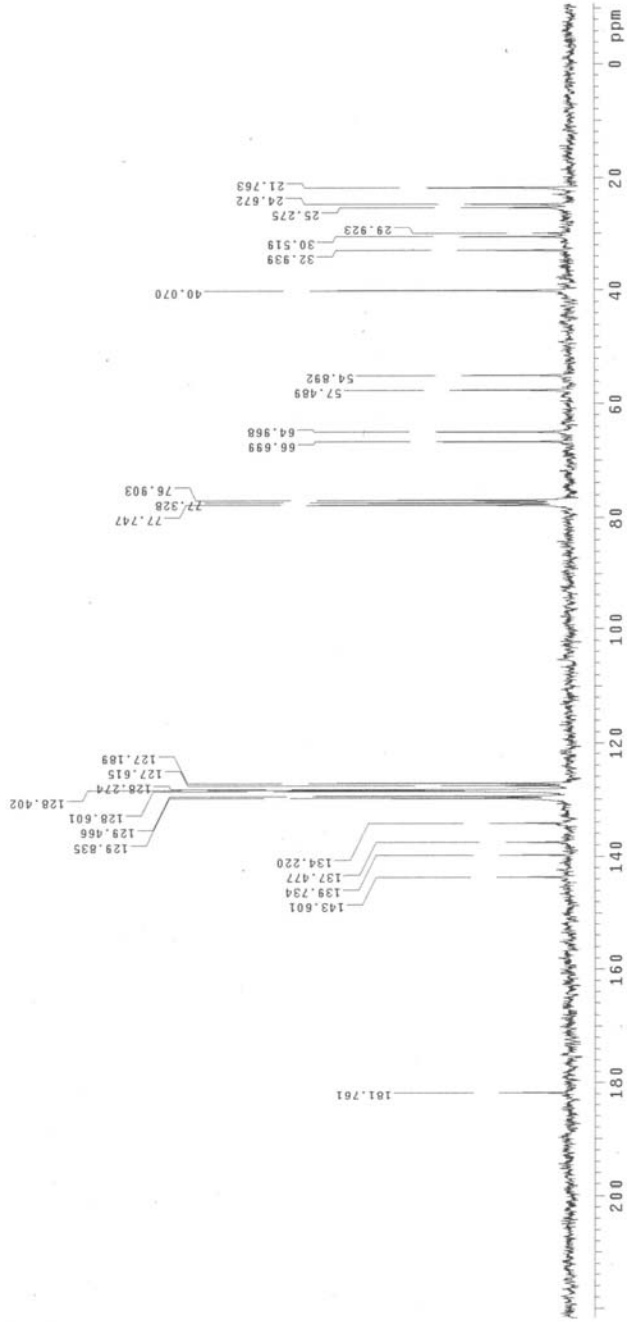
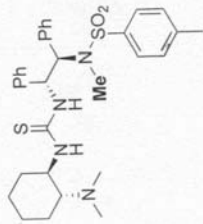
z2h252c



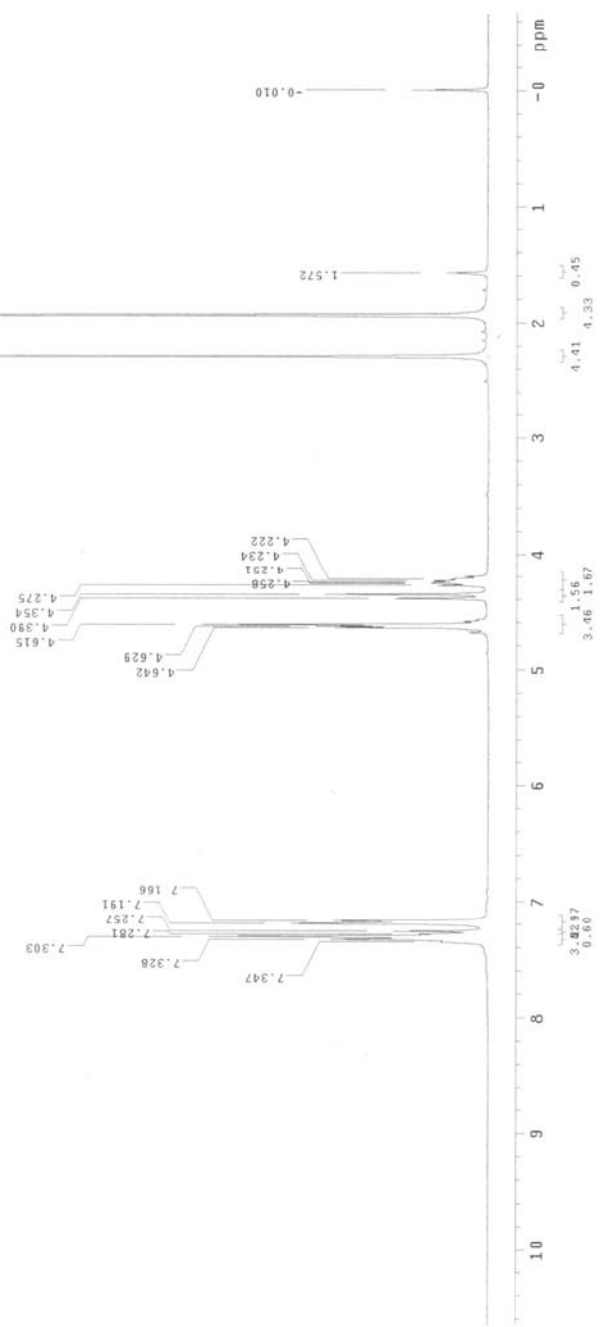
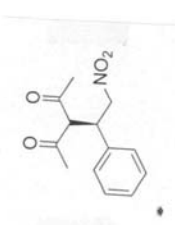
z2h8c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

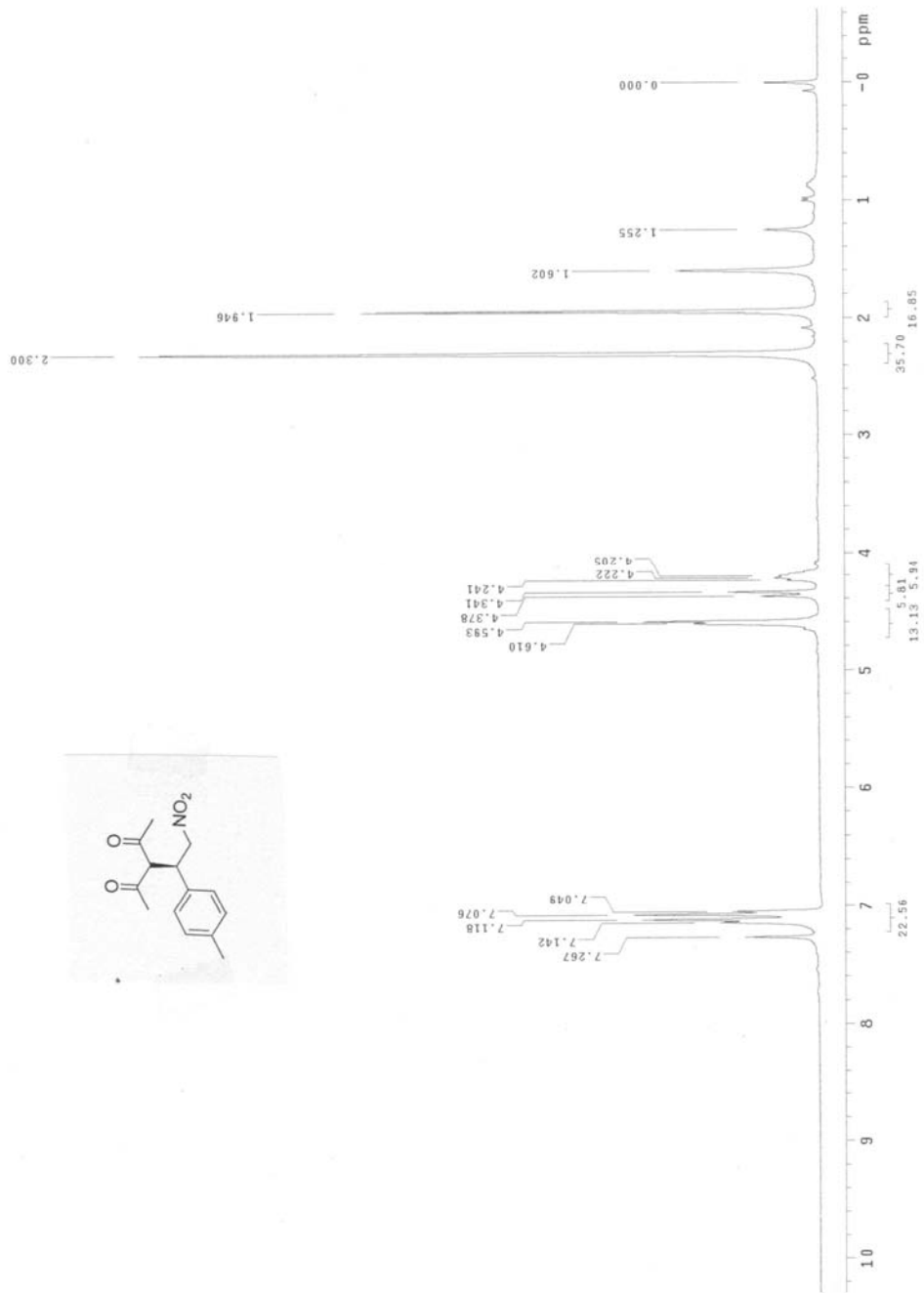
Relax_delay 1.000 sec
Acq_delay 0.000 sec
Acq_time 1.000 sec
Width 17543.9 Hz
188 repetitions
OBSERVE C13 75.4552576 MHz
PULSE 14 596.8607172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
NO 1.0000000000000000
FT size 65536
Total time 2 hr, 13 min, 32 sec



zzh136



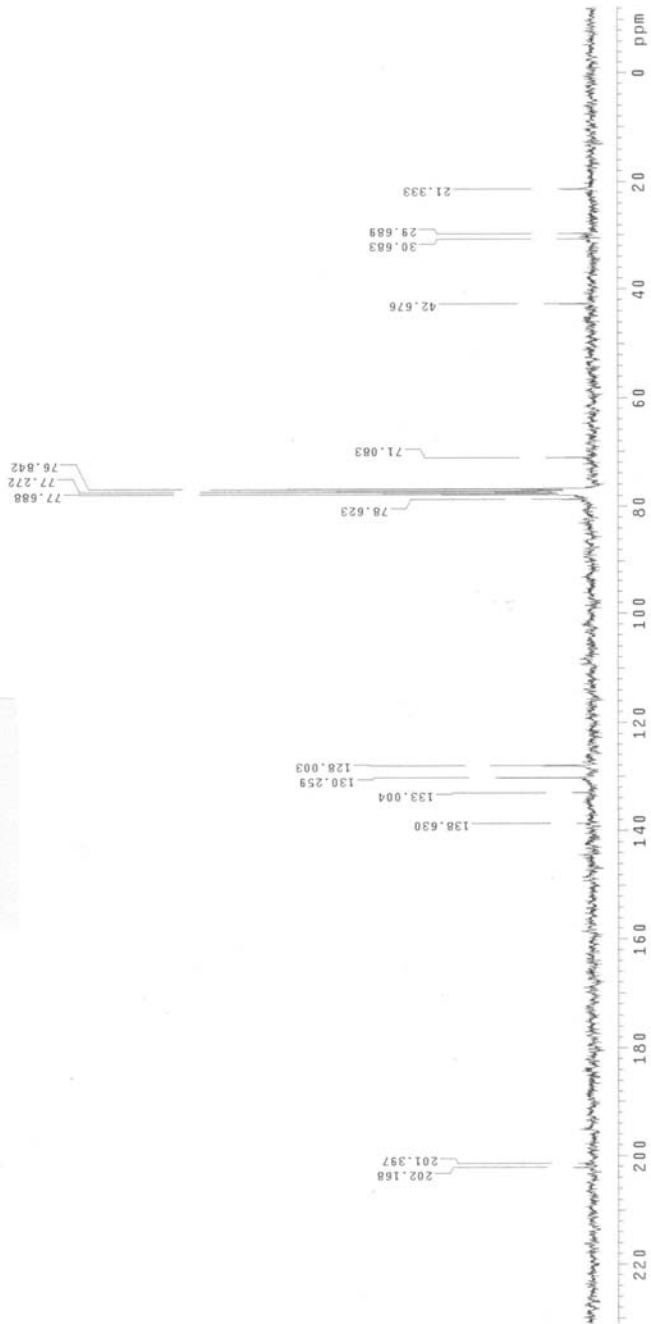
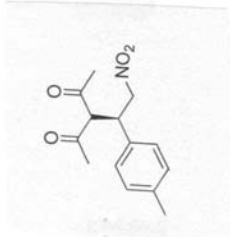
z2h-1b



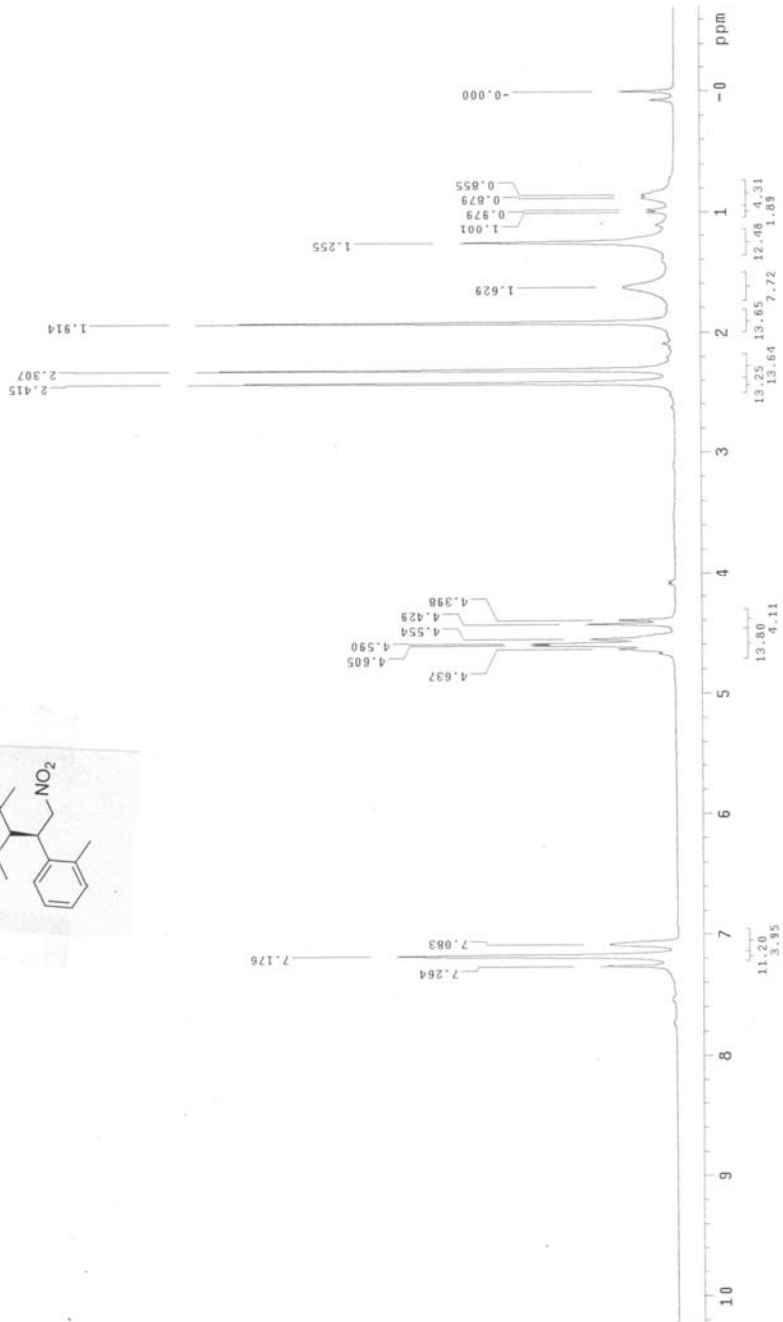
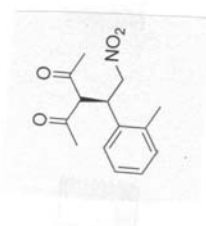
c13-4b

Solvent: CCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. time 0.450 sec
Width 18348.6 Hz
832 repetitions
OBSERVE C13, 75.455256 MHz
PULSE PRG 300.0097172 MHz
Power 50 dB
continuously on
WALTZ-16 modulated
Data PROCESSING
Line PROCESSING 4.0 Hz
FT size 32768
Total time 2 hr, 12 min, 55 sec

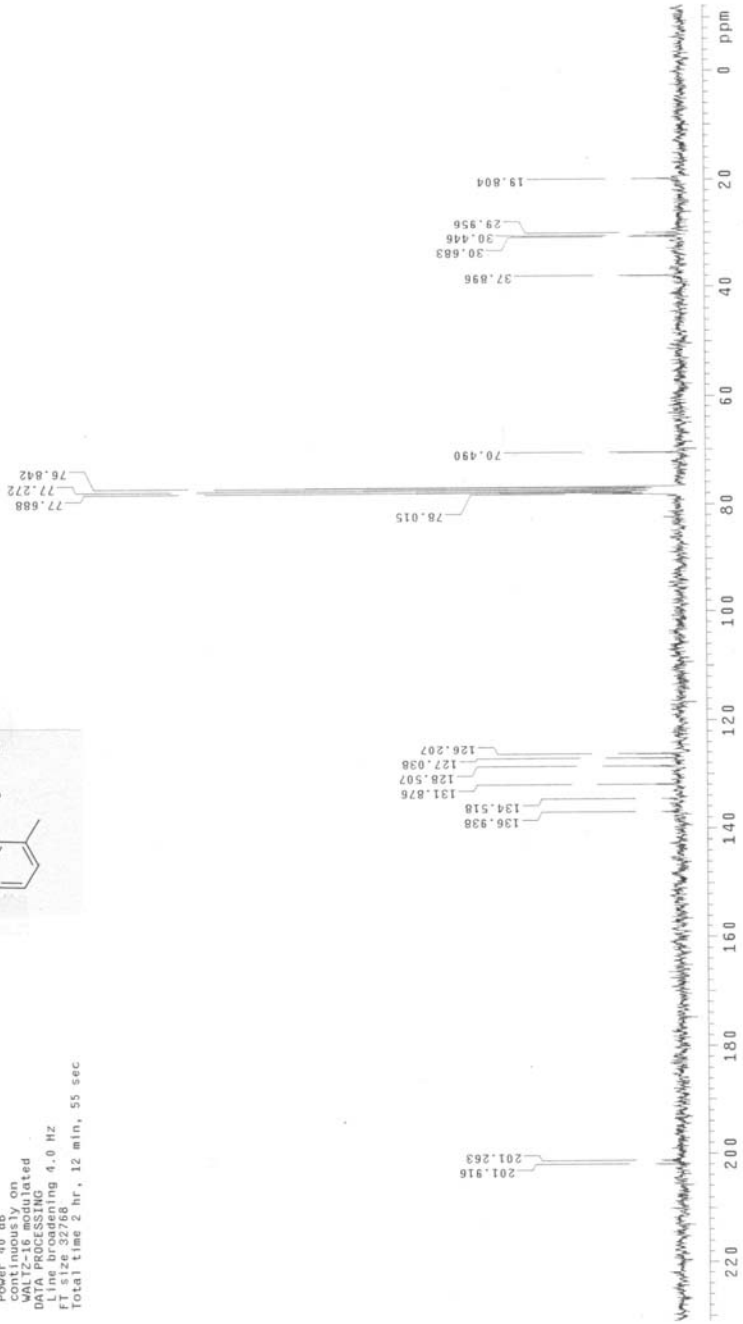
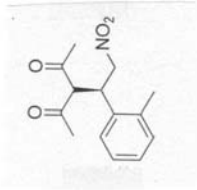


zzh-6c

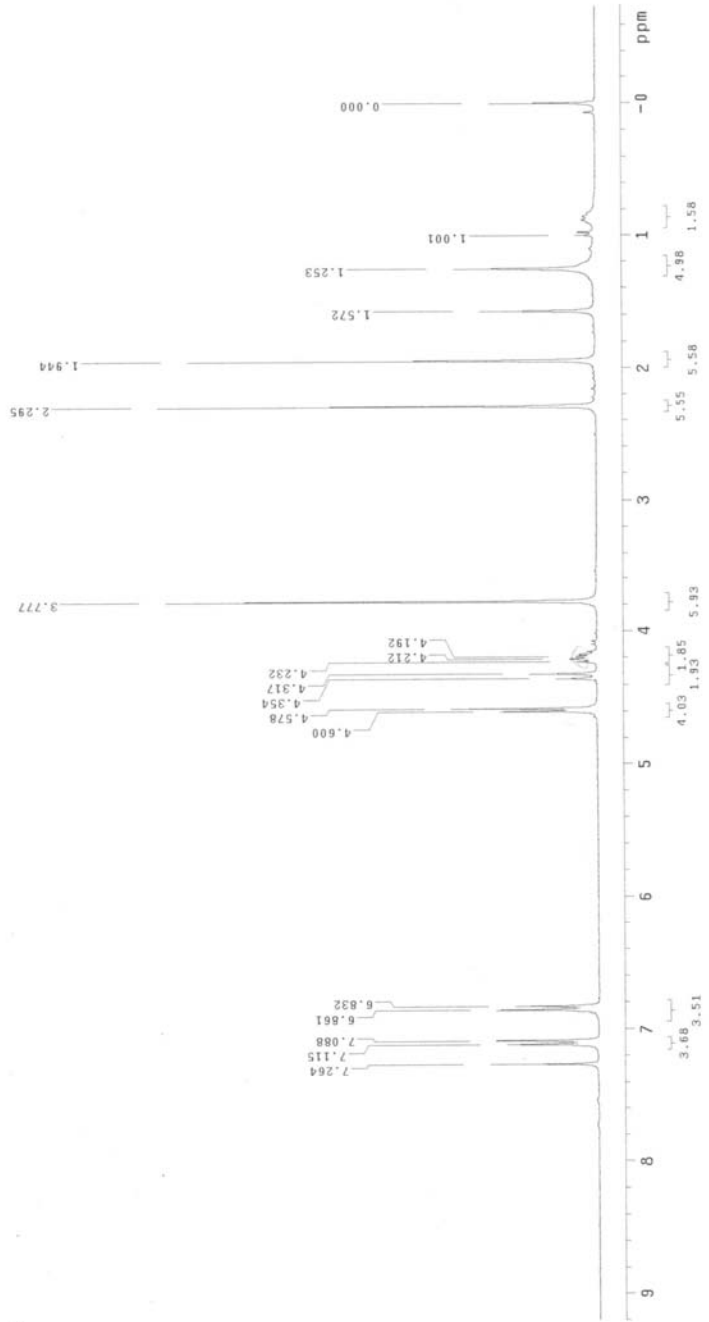
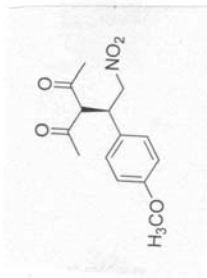


Solvent: CDCl₃
 Ambient Temperature
 Mercury-3000B "mercury200"

Relax. delay: 1.000 sec
 Acq. time: 0.450 sec
 Width: 18248.6 Hz
 576 repetitions
 OSCILLATE CH1, 300.0807172 MHz
 Power: 40 dB
 continuously on
 WALTZ-16 modulated
 Data resolution: 0.1 Hz
 Line broadening: 4.0 Hz
 FT size: 32768
 Total time: 2 hr, 12 min, 55 sec



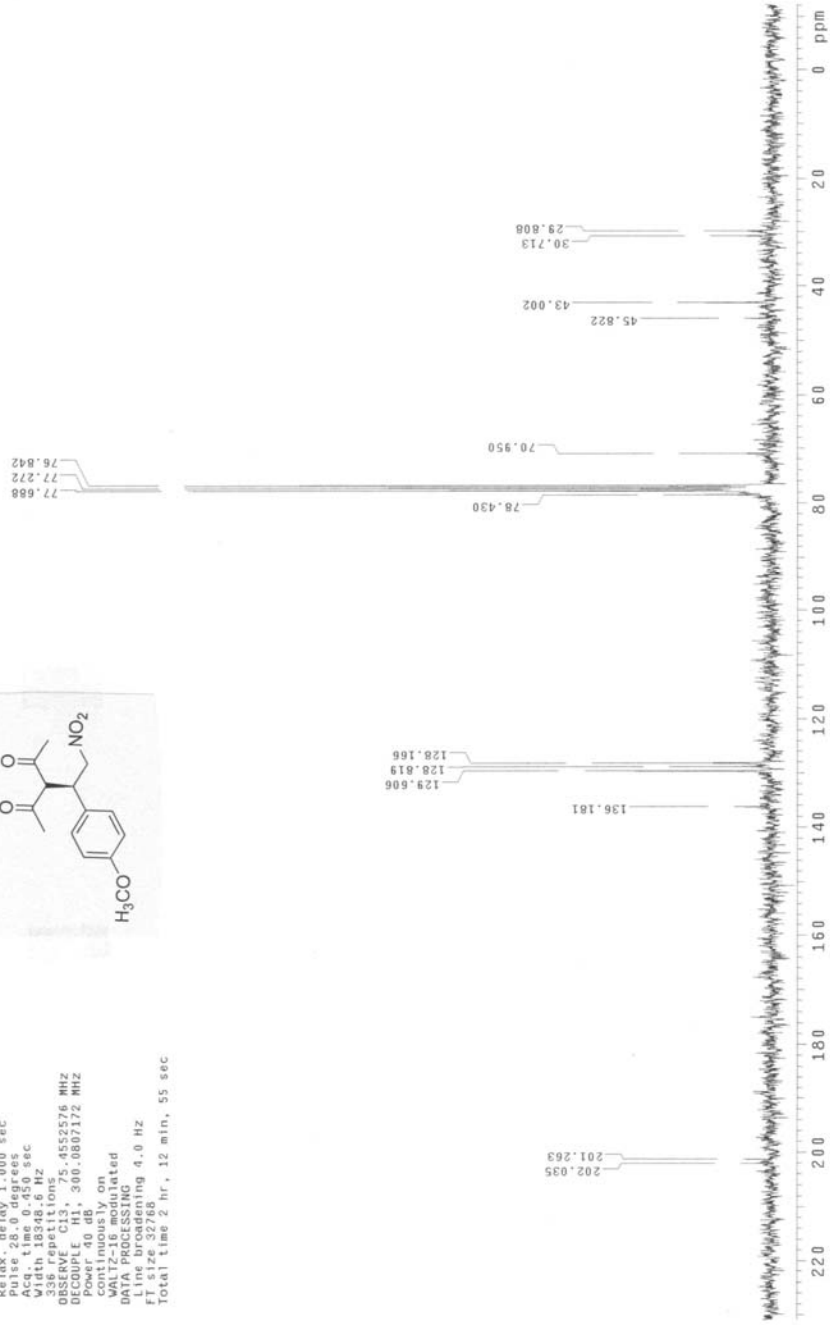
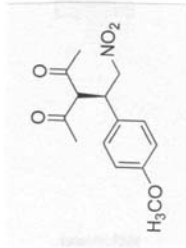
d2



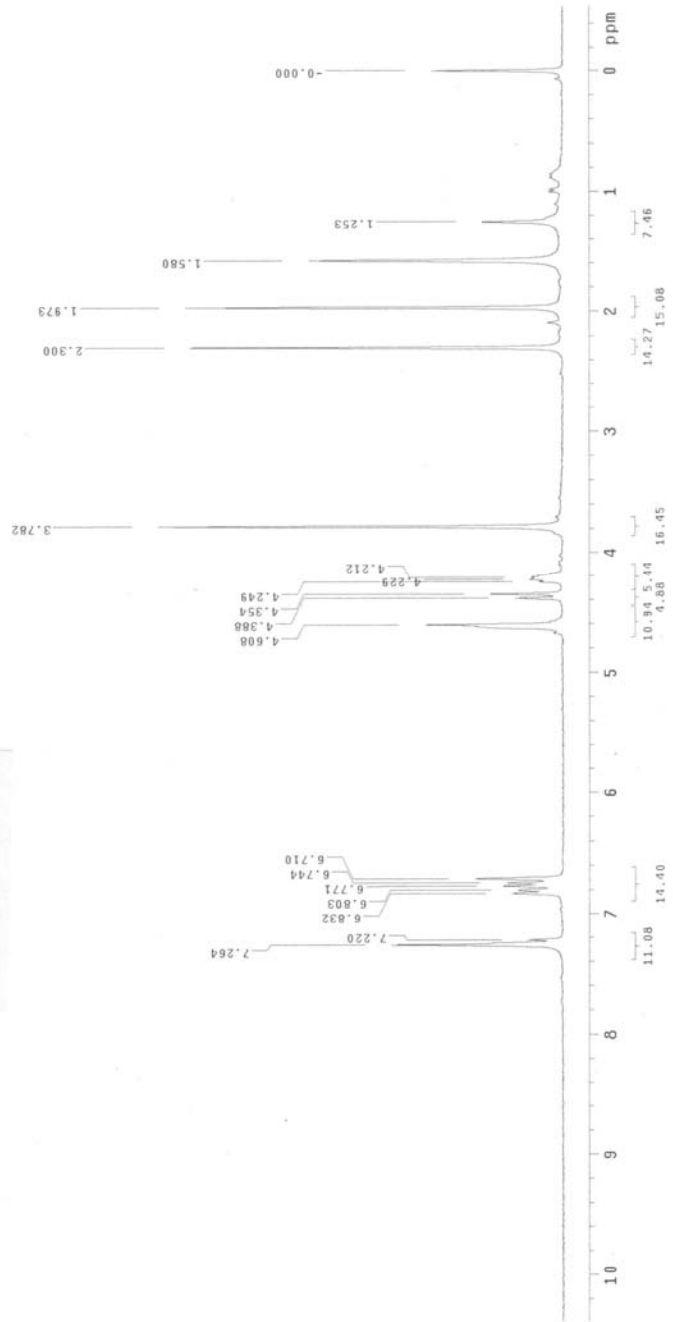
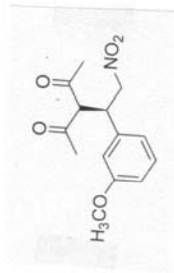
4d

Solvent: CDCl3
Ambient Temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Acq. time 0.450 sec
Width 18348.6 Hz
336 repetitions
OBSERVE C13, 75.4552576 MHz
PULSE PRG zgpg30, 300.0007172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
LISTENING 4.0 HZ
FT size 32768
Total time 2 hr, 12 min, 55 sec



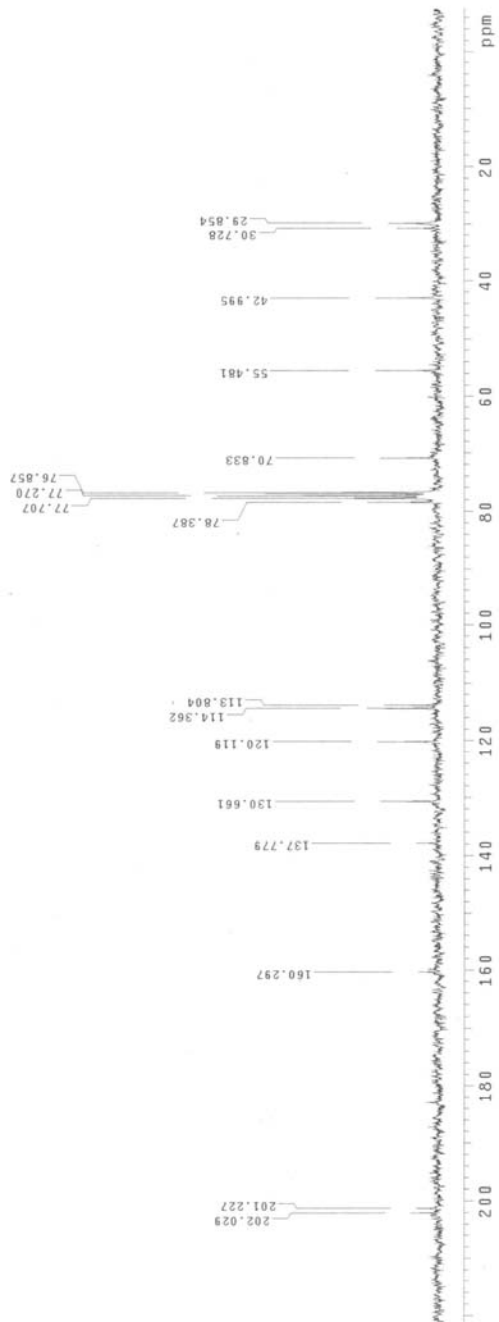
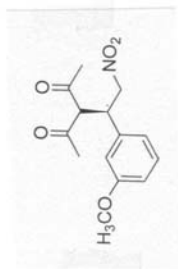
z2h-4e

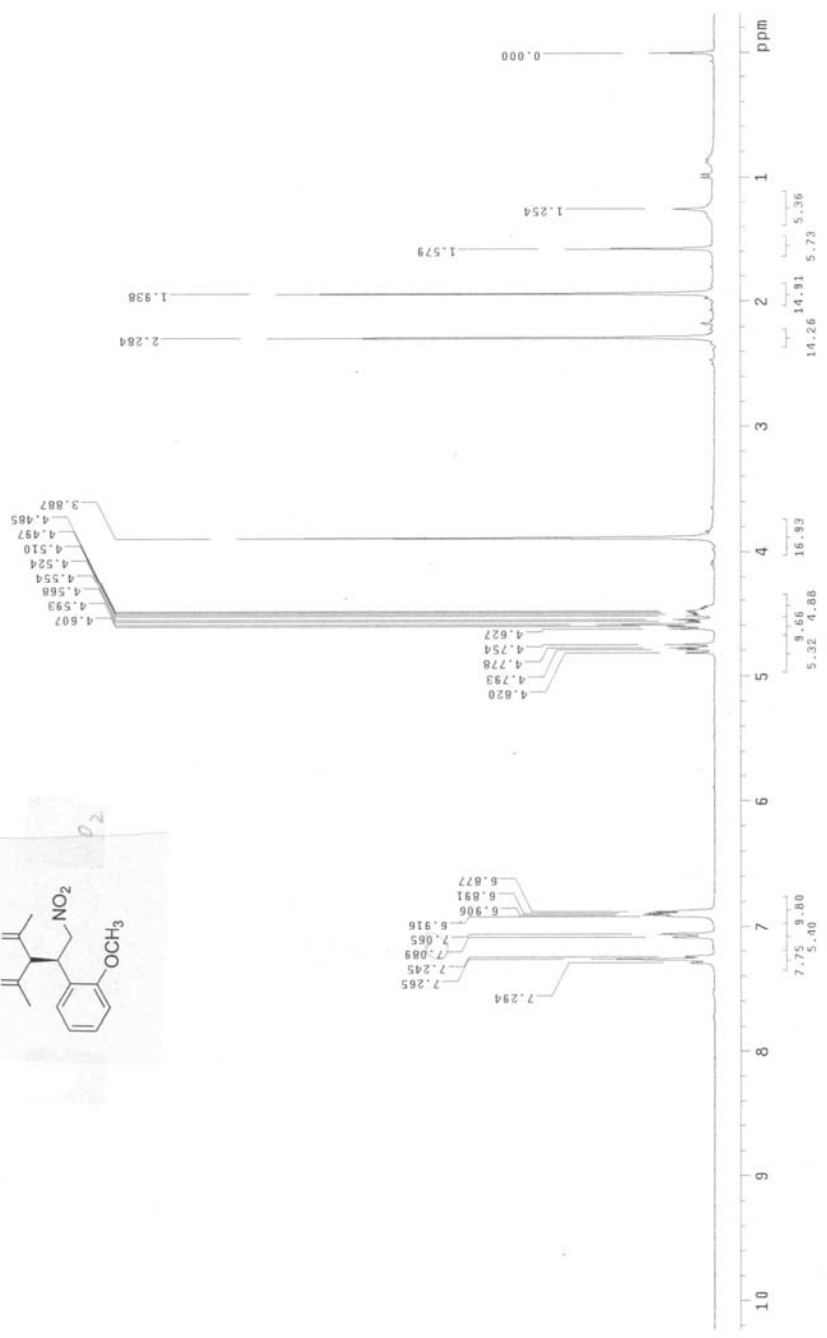
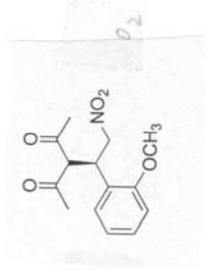


c13-4e

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

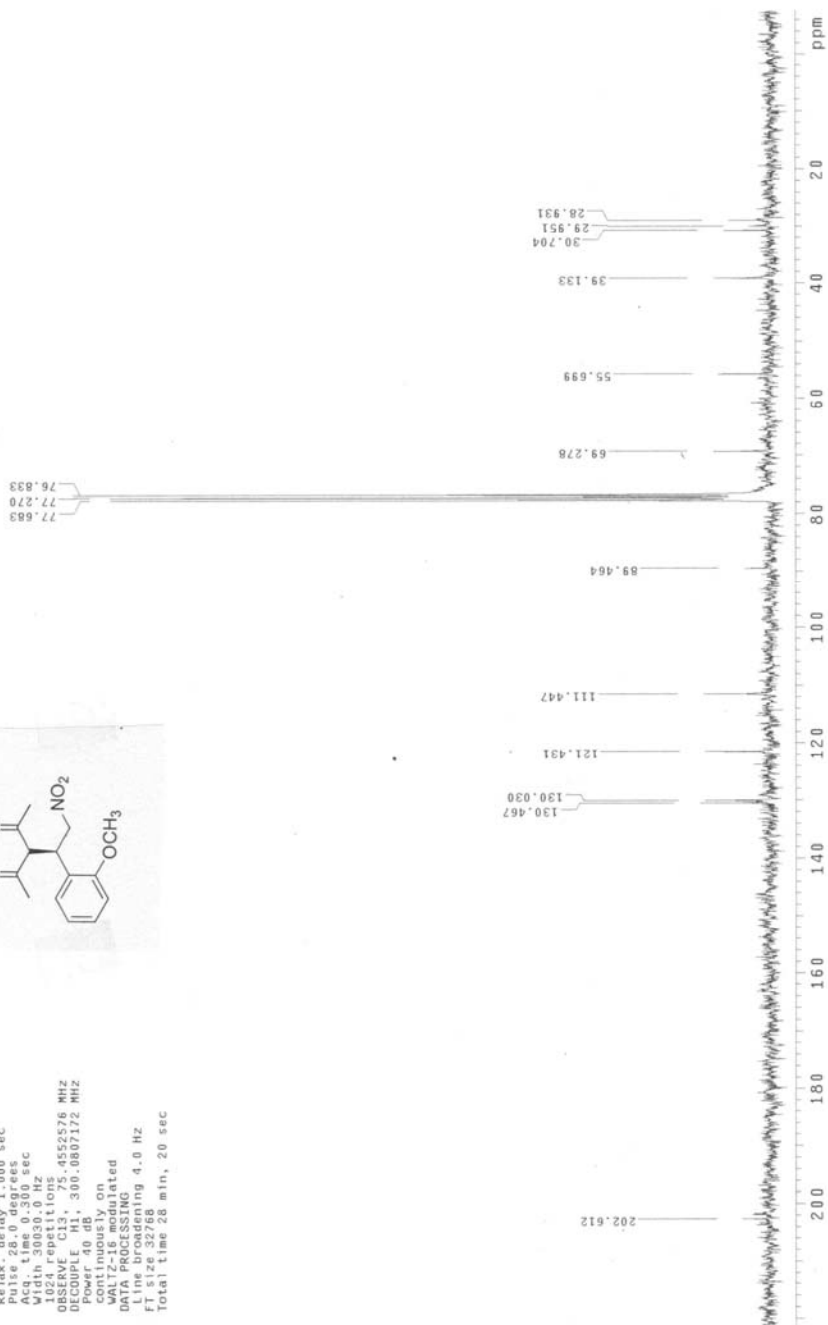
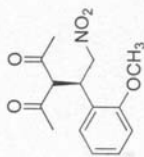
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.300 sec
Width 30030.0 Hz
188 repetitions
Spectral width 32768.0 Hz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
Waltz16 decoupled
DATA ACQUISITION
Line broadening 4.0 Hz
FT size 32768
Total time 28 min, 20 sec



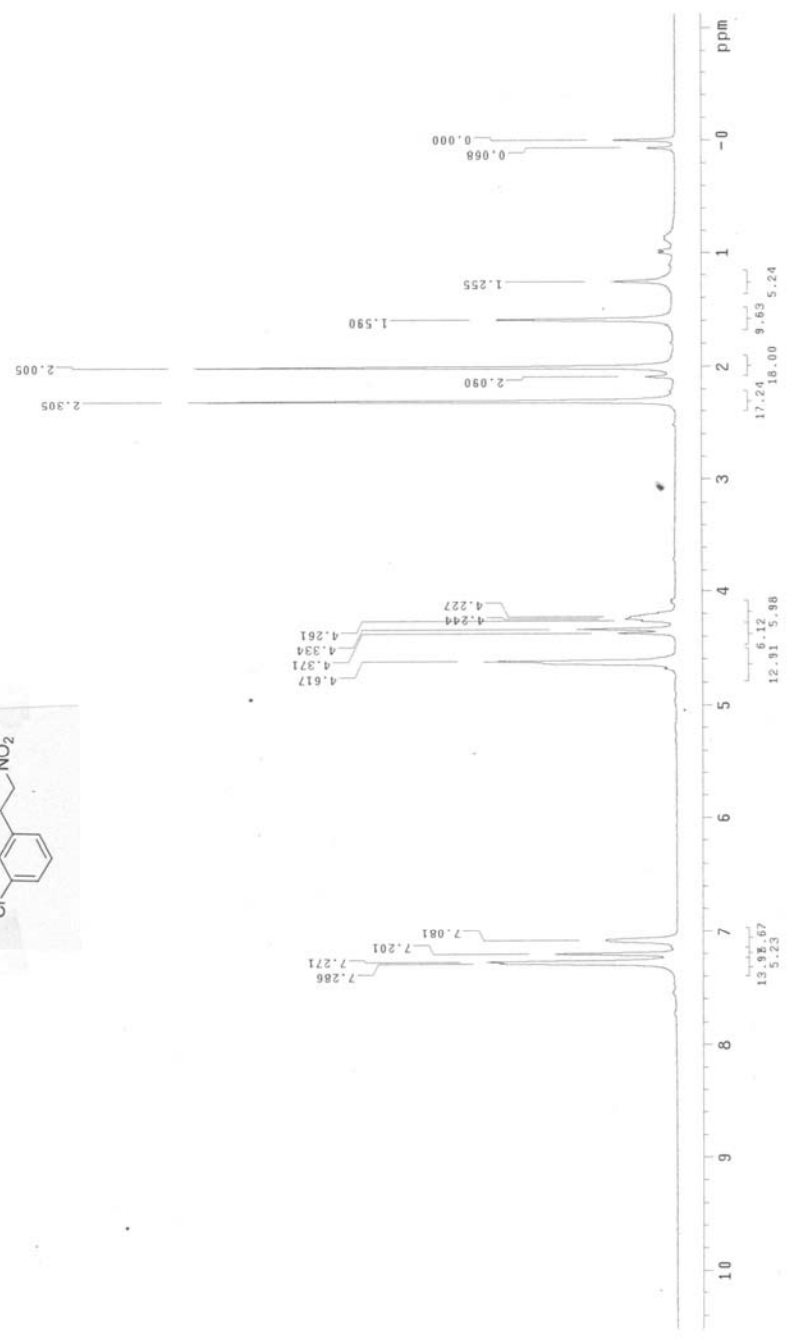
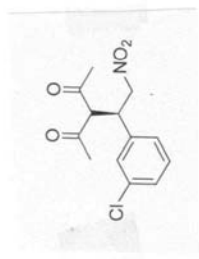


Solvent: CDCl3
 Ambient Temperature
 Mercury-300BBB "mercury300"

Relax. delay 1.000 sec
 Acq. time 0.390 sec
 Width 30030.0 Hz
 1024 repetitions
 OBSERVE F1, 300.8807172 MHz
 DECOUPLE H1, 300.8807172 MHz
 Power 40 dB
 continuously on
 WALTZ16 modulated
 DUMPEM
 Line broadening 4.0 Hz
 FT size 32768
 Total time 28 min, 20 sec

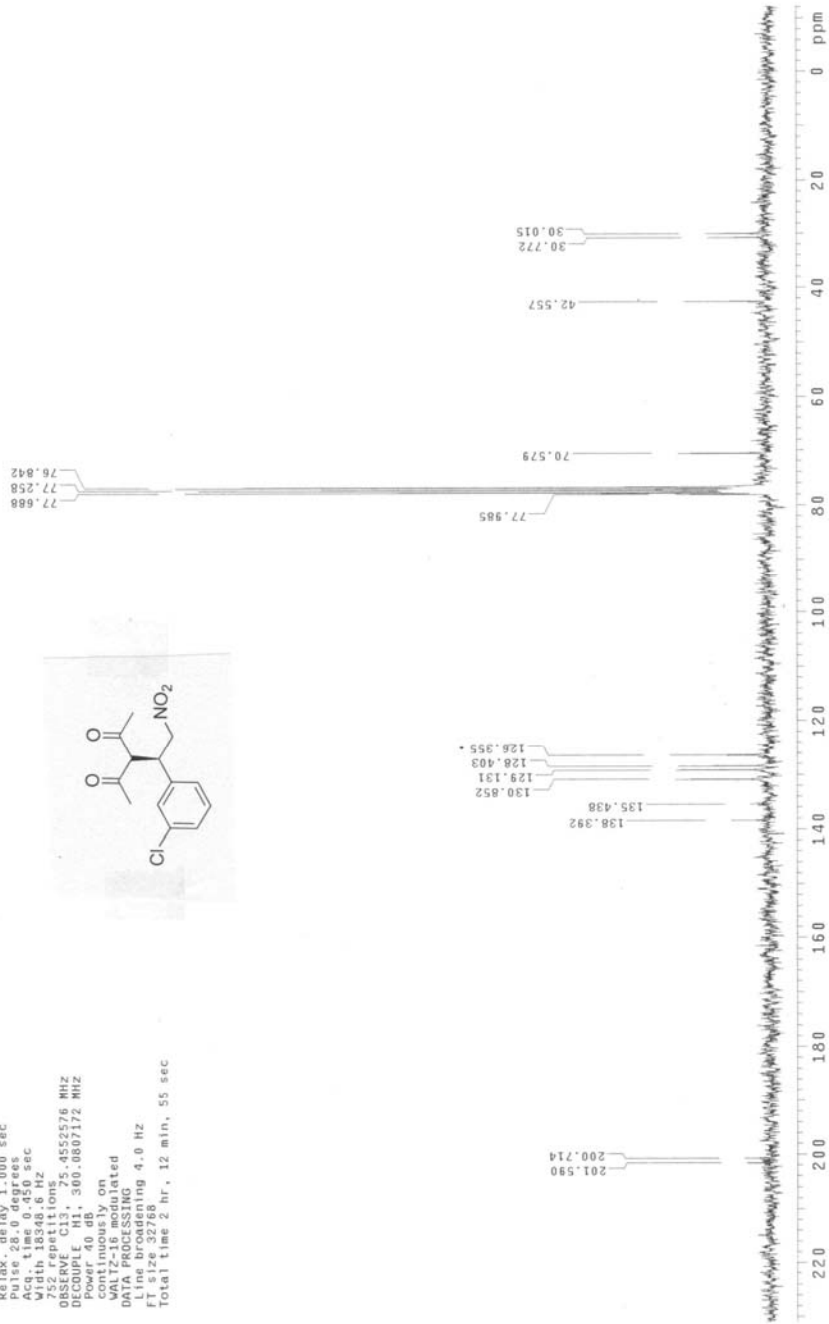
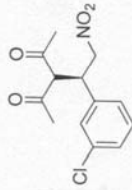


z2h-4g

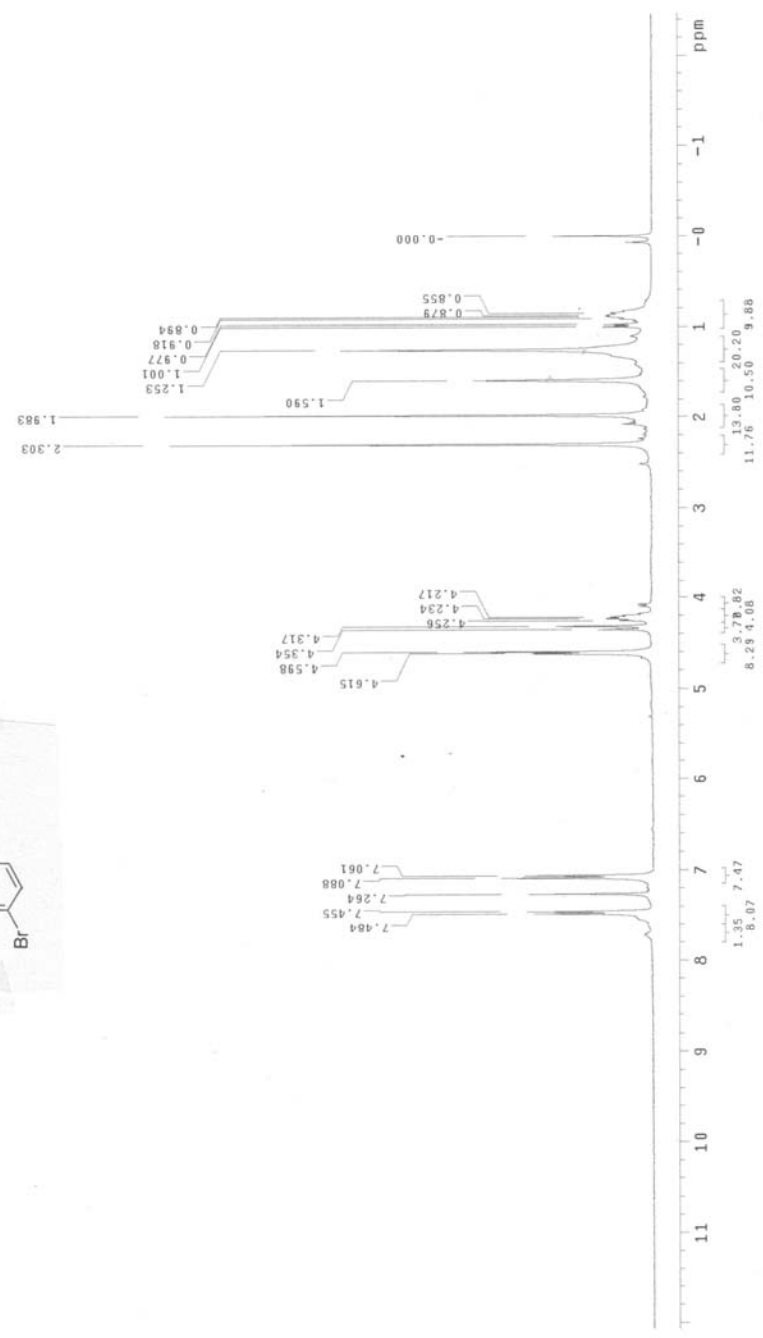
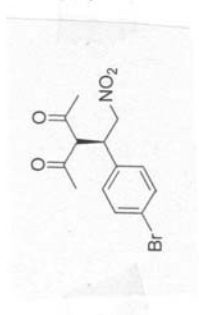


Solvent: CCl₄
 Ambient temperature
 Mercury-300BB "mercury300"

Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Delay 1.000 sec
 Width 18348.6 Hz
 752 repetitions
 OBSERVE C13, 75.4552576 MHz
 DUMP F1, 300.0007172 MHz
 Power 40 db
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING 4.0 Hz
 FT size 32768
 Total time 2 hr, 12 min, 55 sec



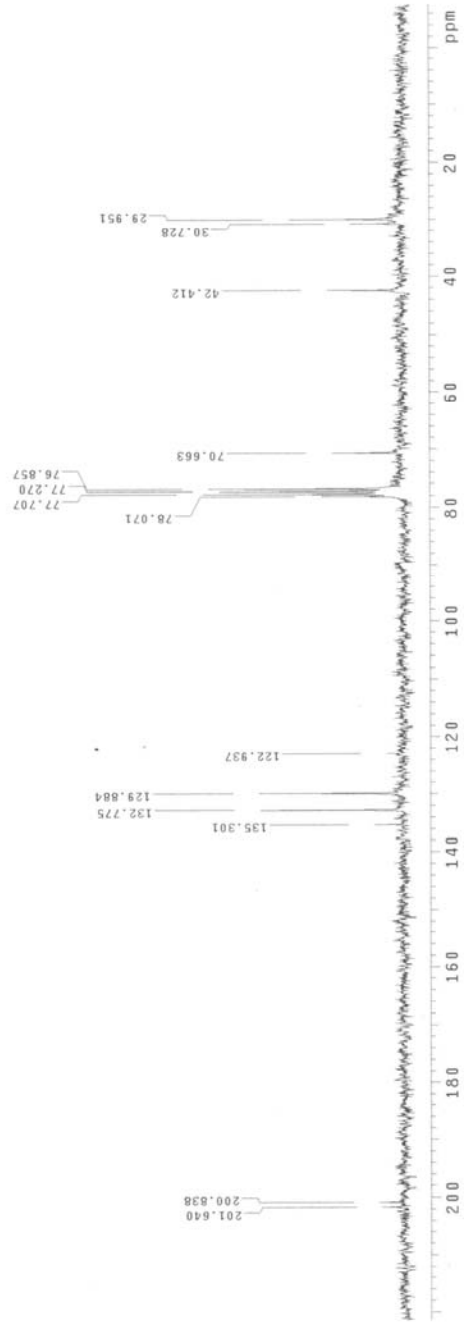
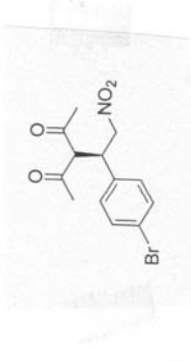
zzh-0h



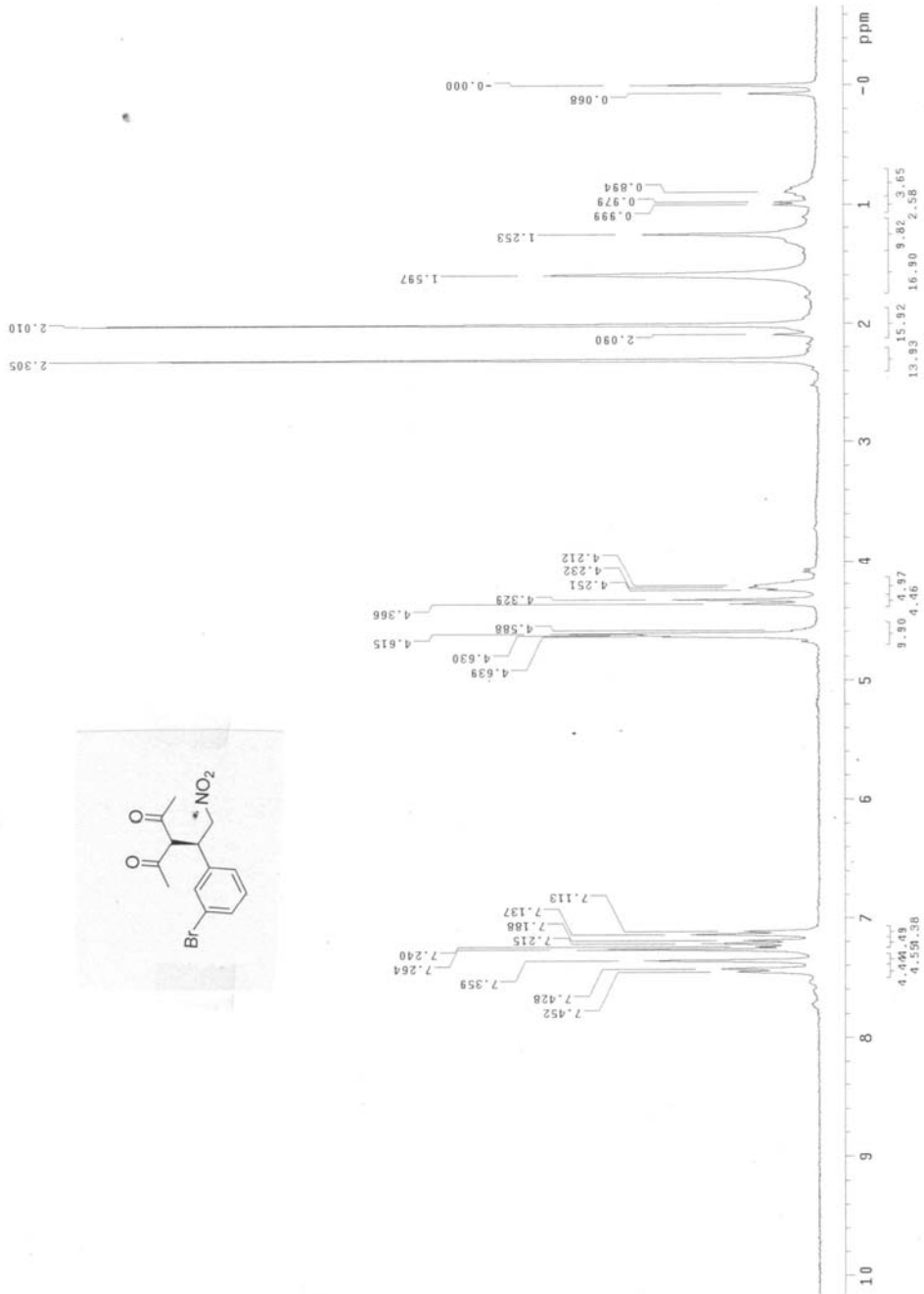
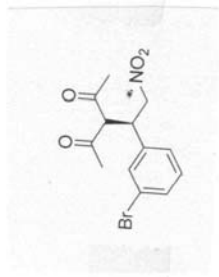
C13-4h

Solvent: CDCl3
Ambient temperature
Mercury-300BBB "mercury300"

Relax. delay 1.000 sec
Acq. time 0.300 sec
Width 30030.0 Hz
372 repetitions
OBSERVE C13, 455676 MHz
PULSE PRG, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
Data Acquisition
Time broadening 4.0 Hz
FT size 32768
Total time 28 min, 20 sec

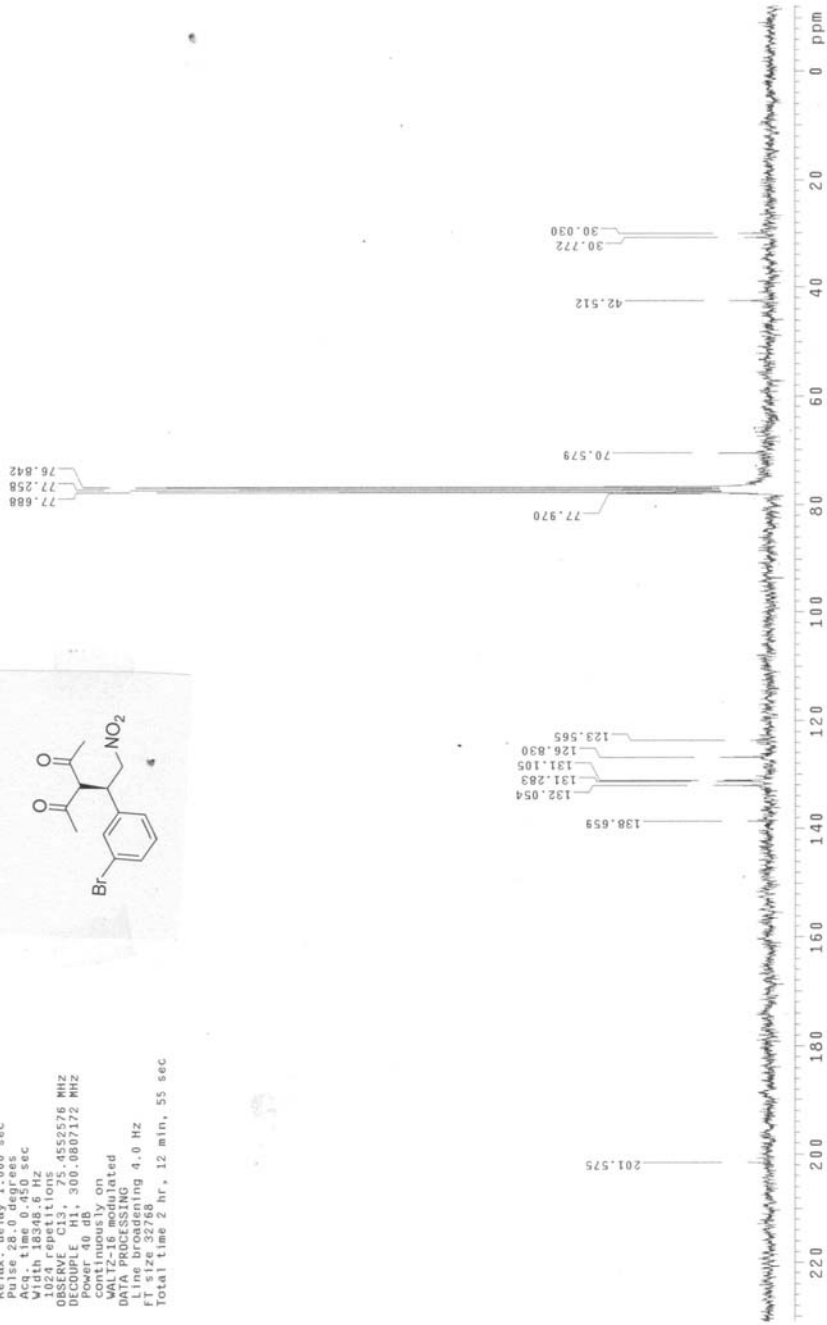
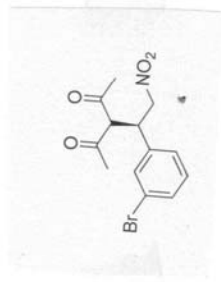


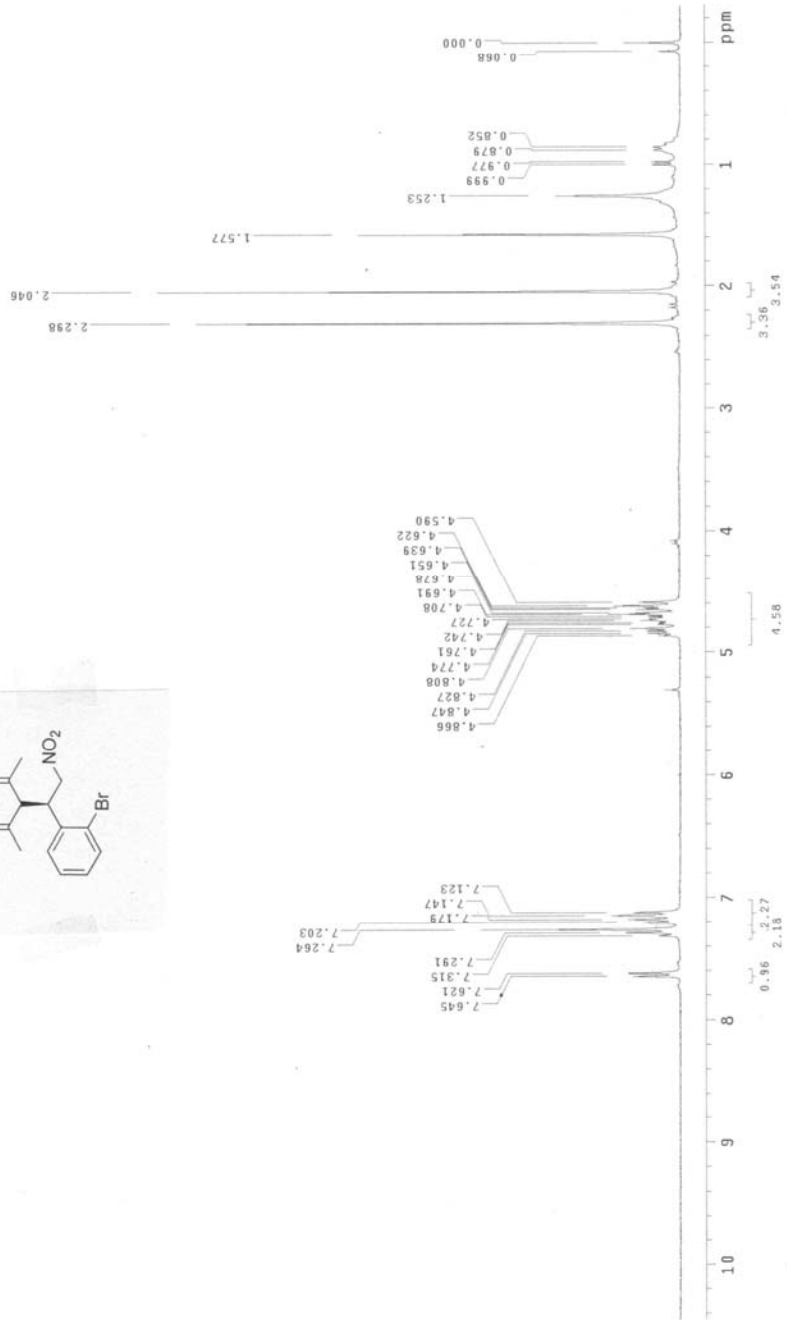
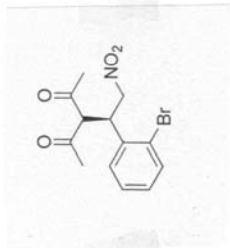
19-3-14-a-41



Solvent: CDCl3
 Ambient temperature
 Mercury-30085 "mercury300"

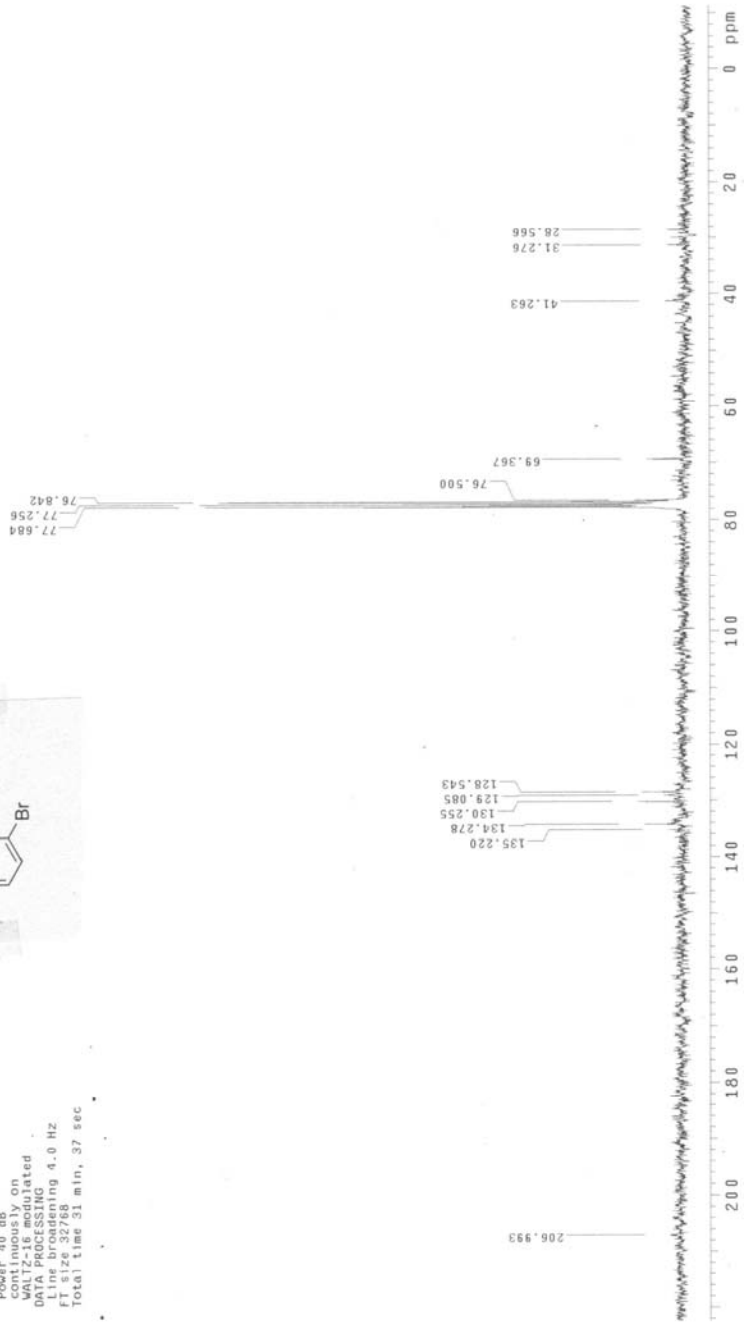
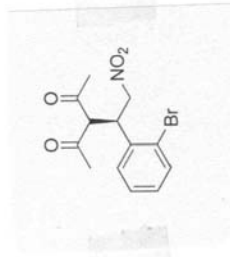
Relax. delay 1.000 sec
 Acq. time 0.450 sec
 Acquisition 0.450 sec
 Width 18348.6 Hz
 1024 repetitions
 OBSERVE C13, 75.455256 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING 4.0 Hz
 FT size 32768
 Total time 2 hr. 12 min. 55 sec



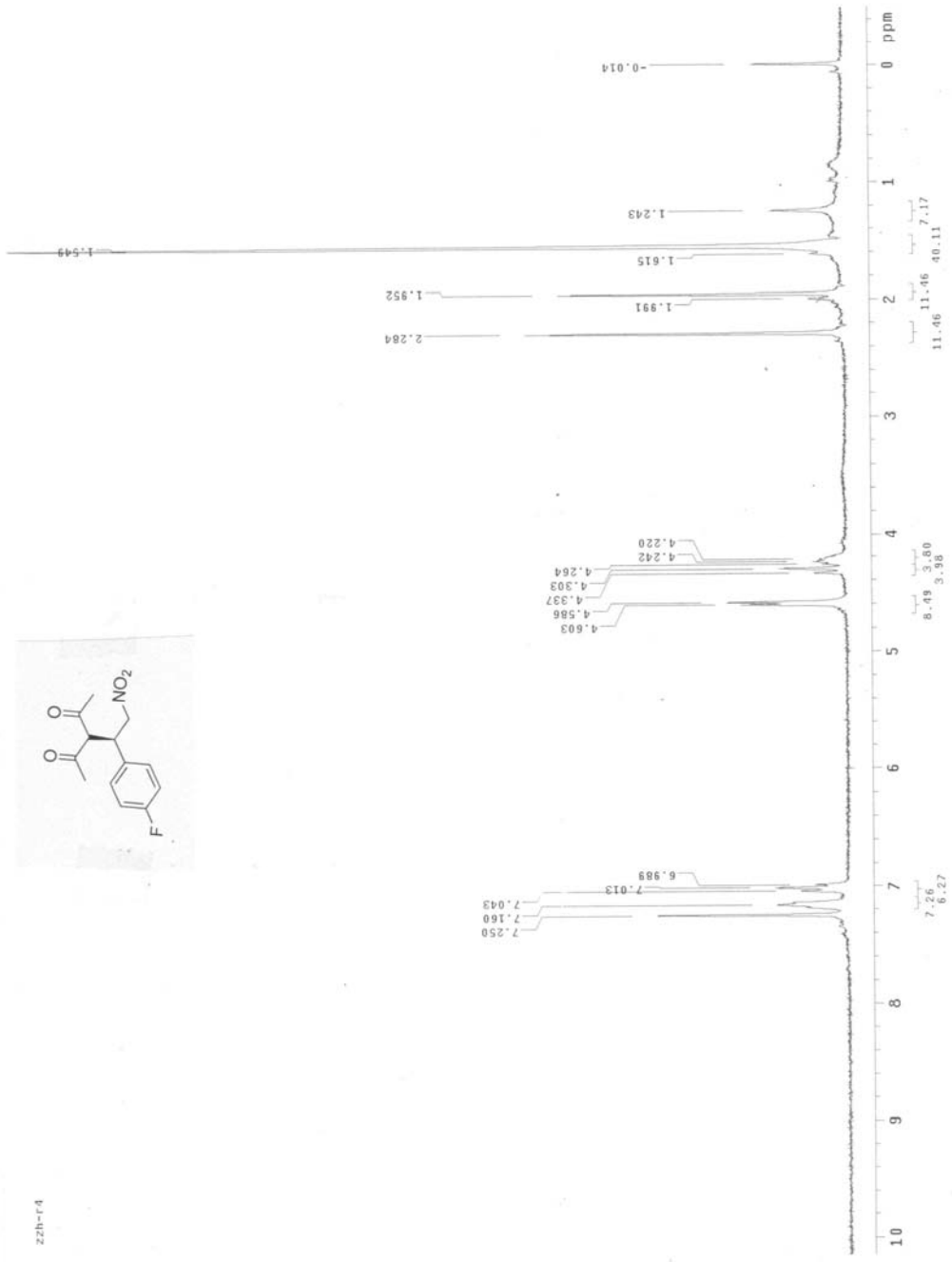
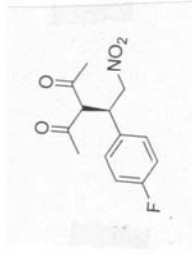


c13-4j

Solvent: CDCl3
Ambient temperature
Mercury-300BB5 "mercury300"
Relax. delay 1.000 sec
Acq. time 0.590 sec
Width 17636.7 Hz
852 repetitions
OBSERVED F1 455036 MHz
DECOUPLE H3 300.0807172 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DUMPER 1000000
Line broadening 4.0 Hz
FT size 32768
Total time 31 min, 37 sec



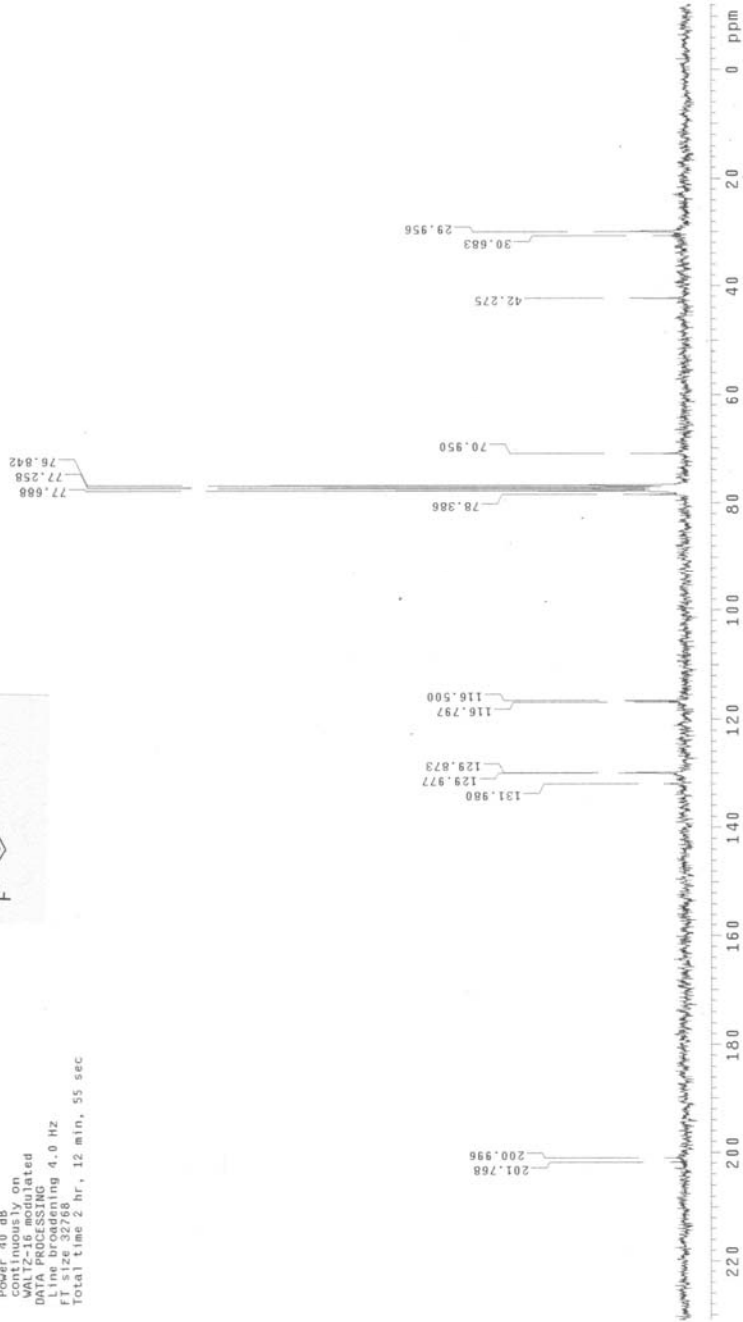
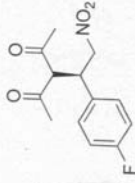
22h-r4



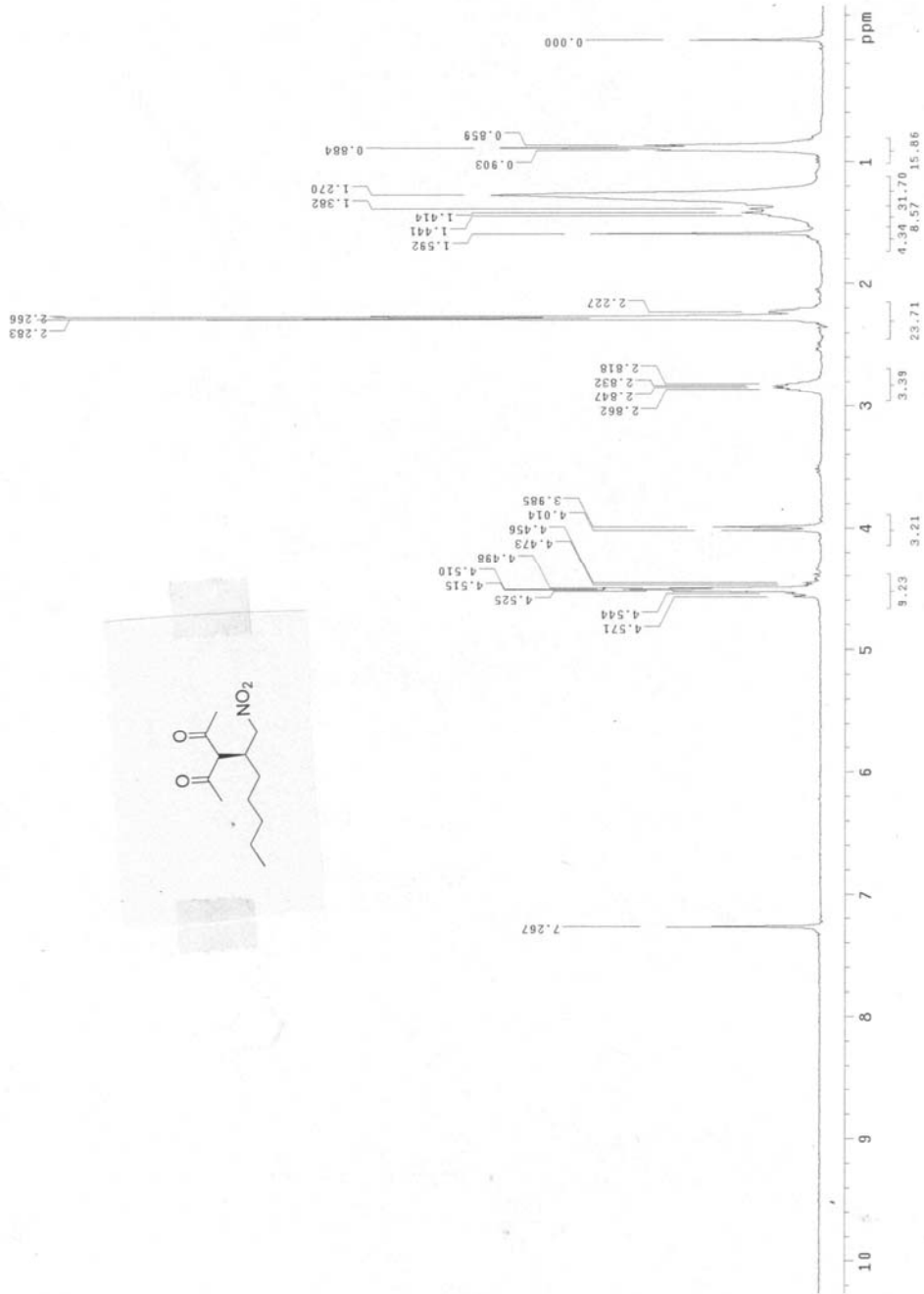
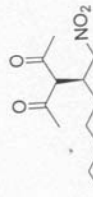
4k

Solvent: ClOCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.000 sec
Width 18348.6 Hz
672 repetitions
OBSERVE Cl3, 75.4552576 MHz
DOPPLER Cl3, 300.0807172 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 size 32768
Total time 2 hr, 12 min, 55 sec



ZZH355b-r

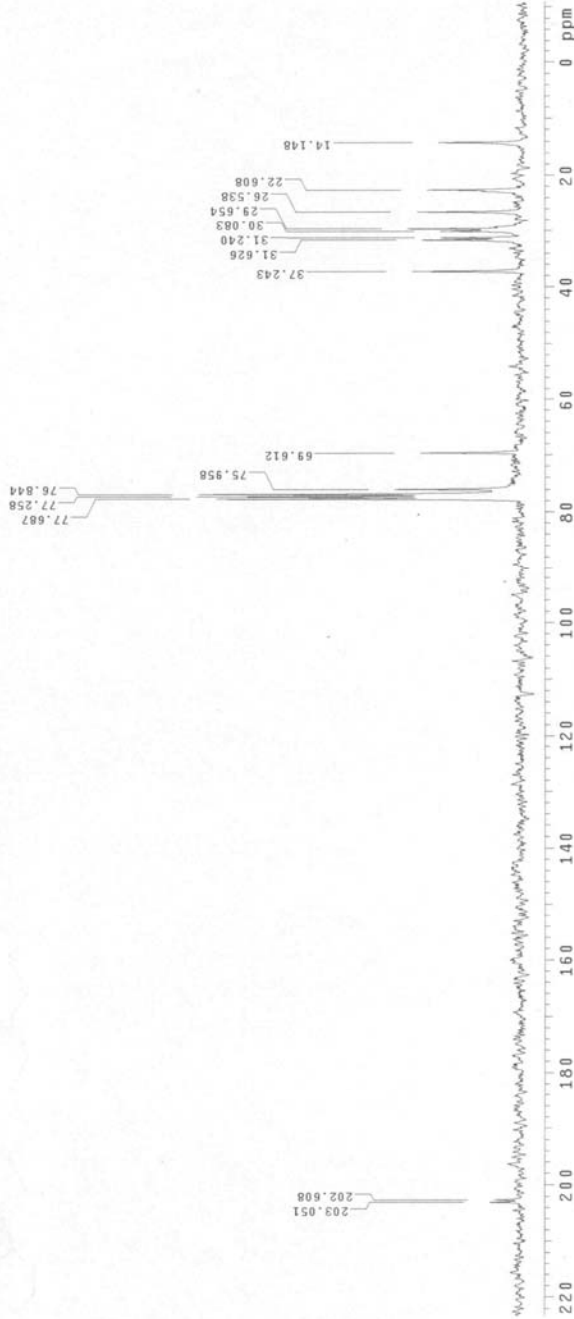
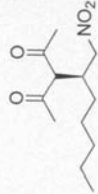


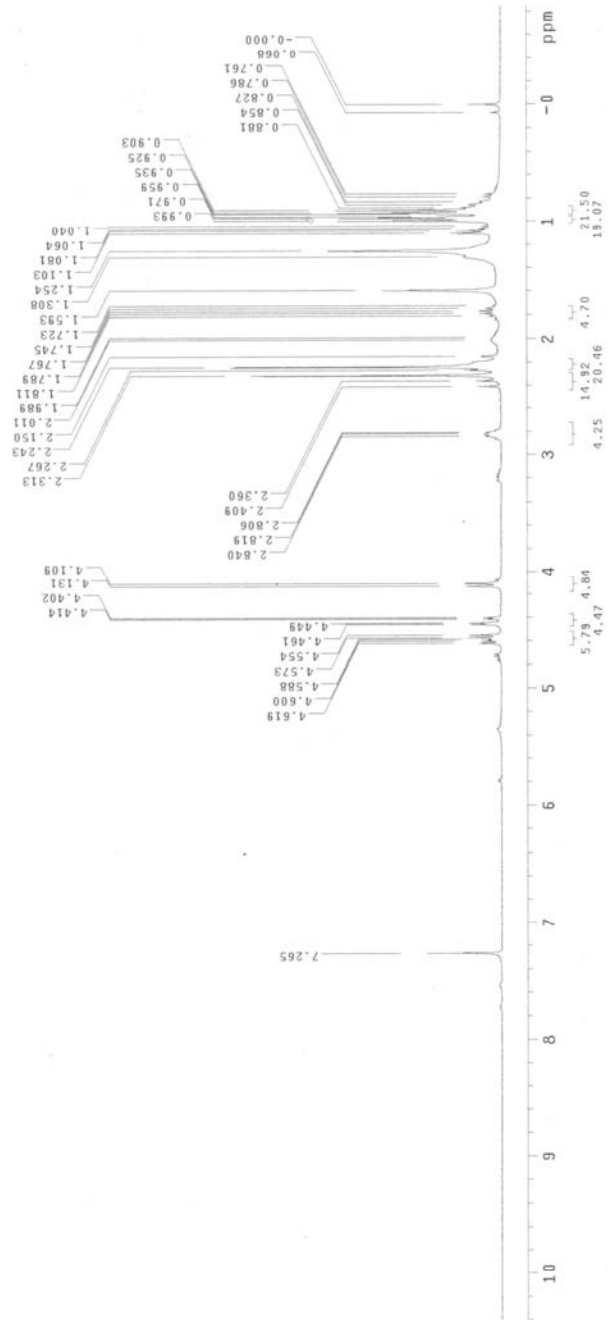
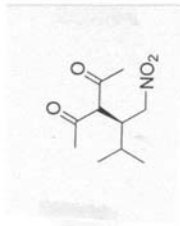
c13220355Dr

4m

Solvent: COCl₂
Ambient temperature
Mercury-3000B "mercury300"

Relax. delay 1.000 sec
Pulse prog zgpg30
Acq. time 0.550 sec
Width 17667.8 Hz
700 repetitions
Observed 455076 MHz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
DUAL F2 ACQUIRED
Data Processing
Line broadening 12.0 Hz
FT size 32768
Total time 2 hr, 6 min, 31 sec

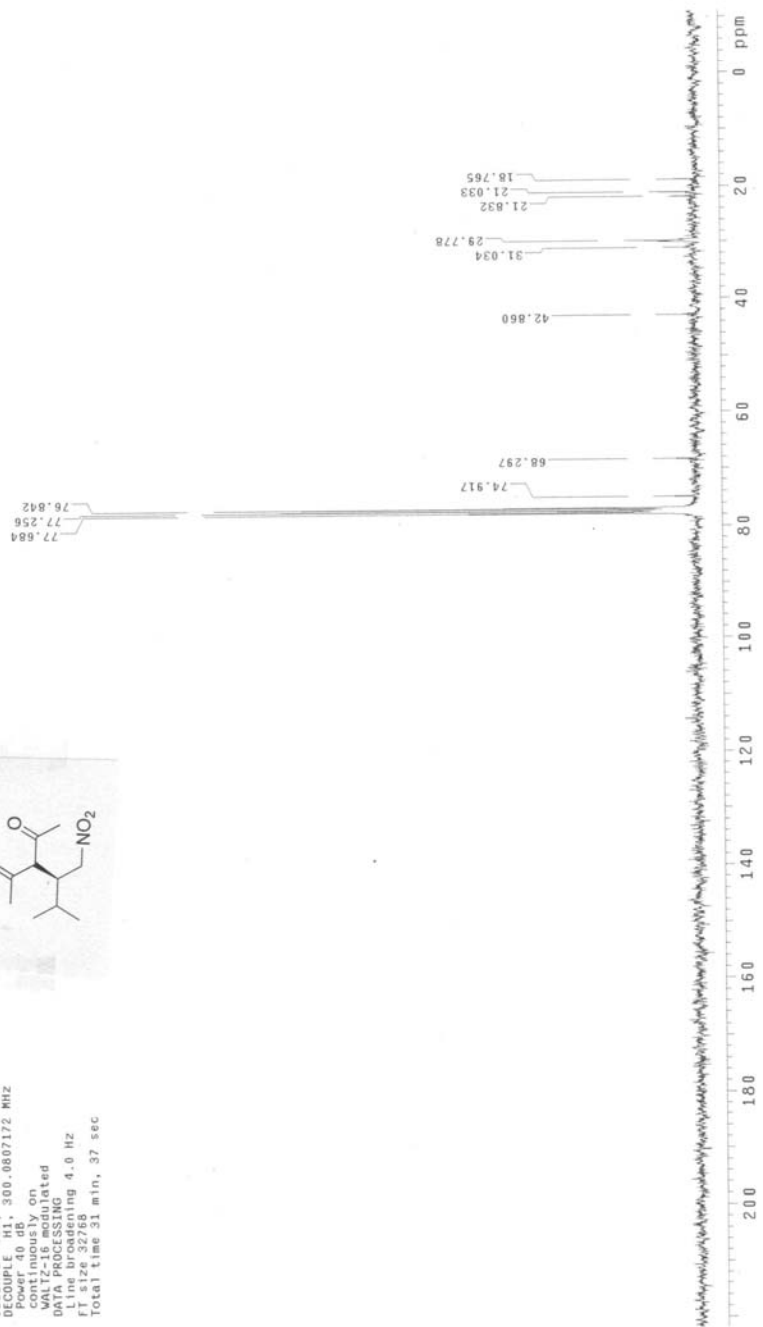
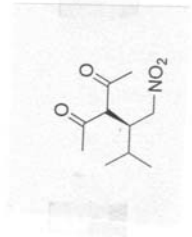




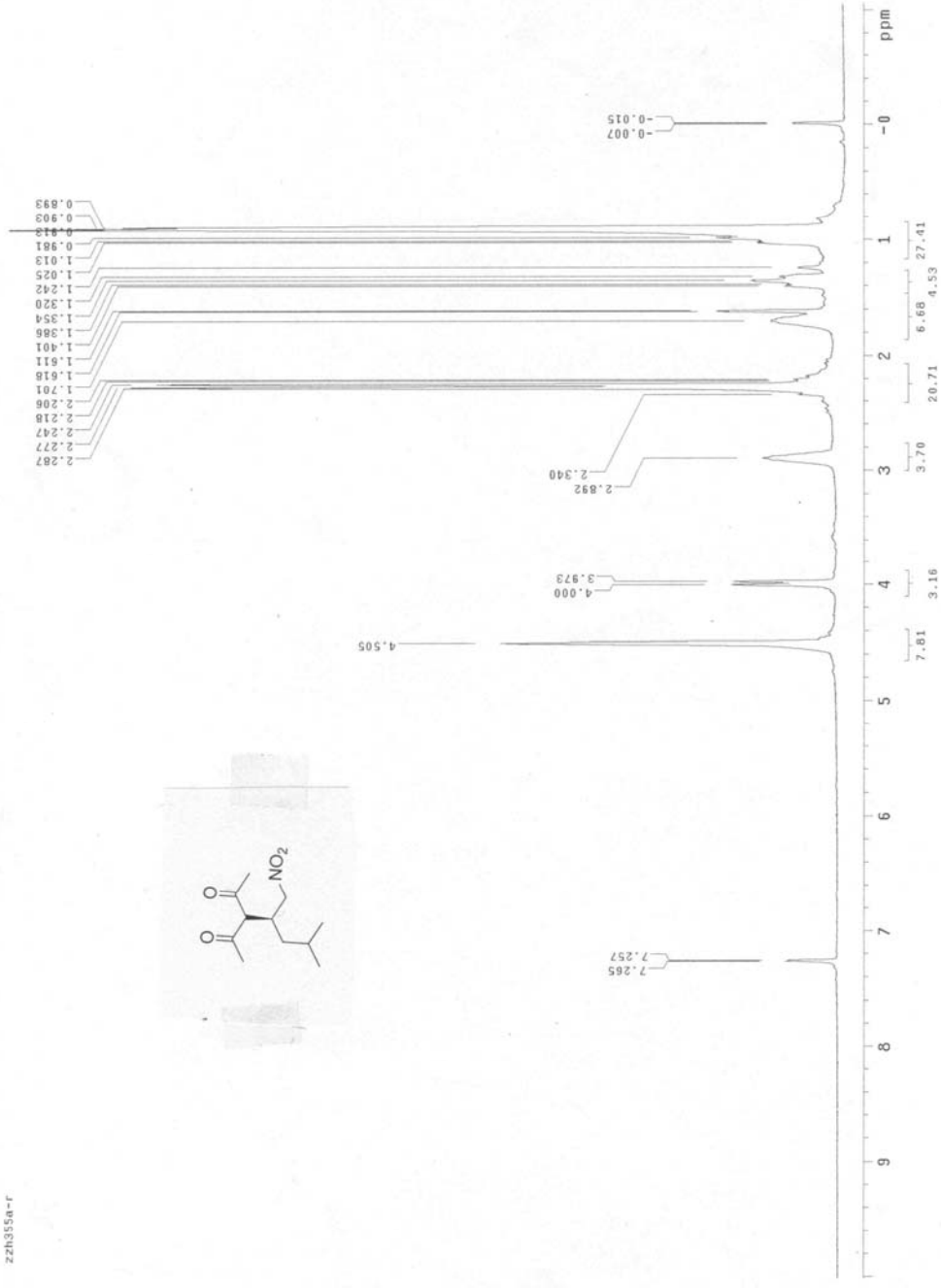
4m-7

Solvent: CDCl3
Ambient temperature
Mercury-30088 "Mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Virt. resolution 1000.000 Hz
1000 FPGs/1000 Hz
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40.00 dB
C13 channel on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
F1 size 32769
Total time 31 min, 37 sec



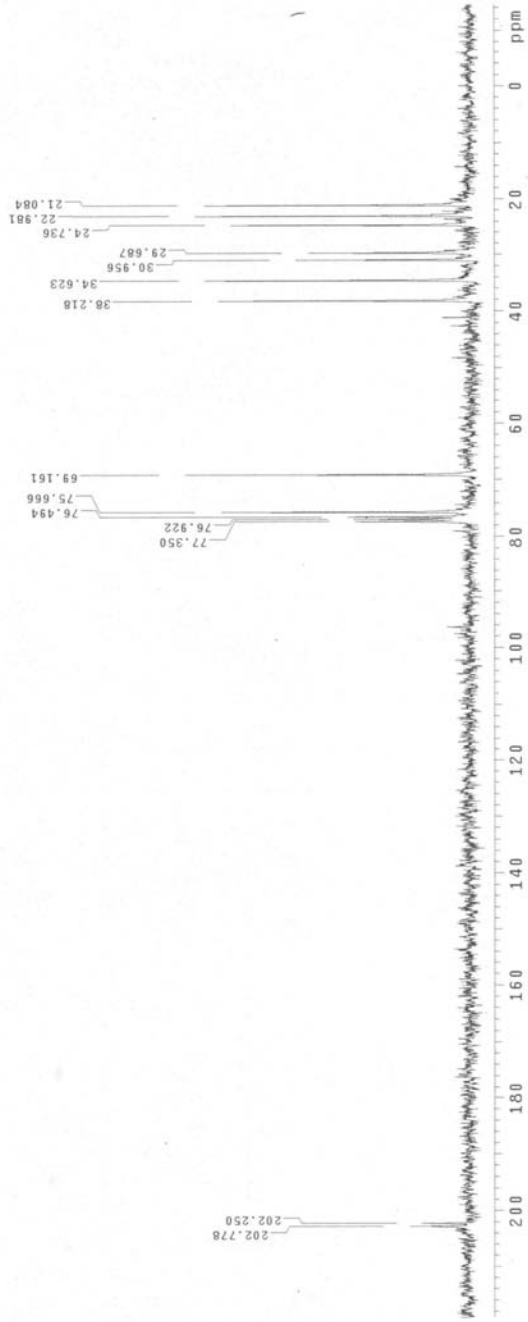
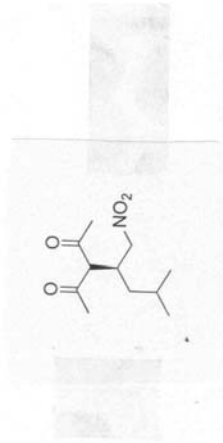
zzh355a-f

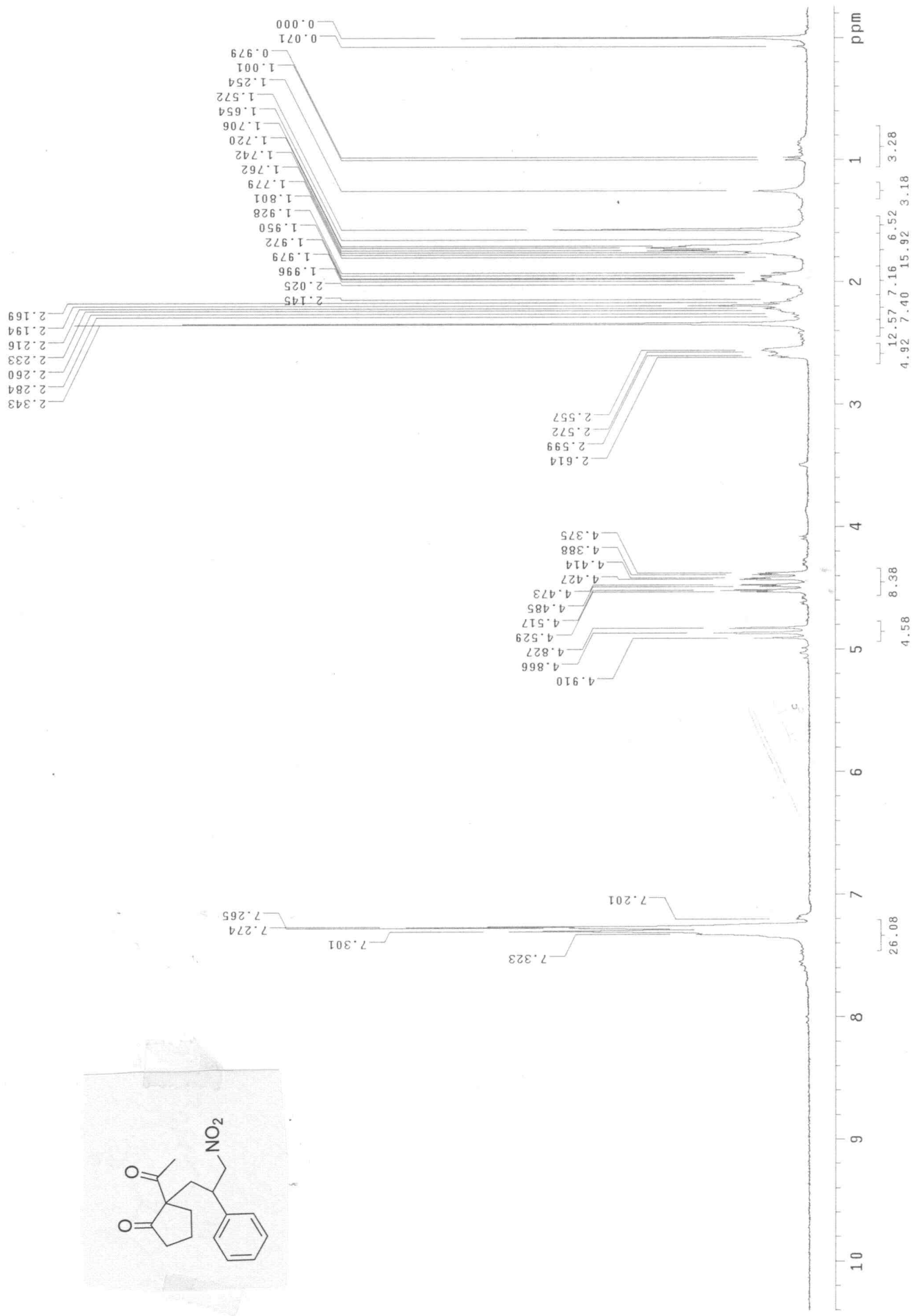
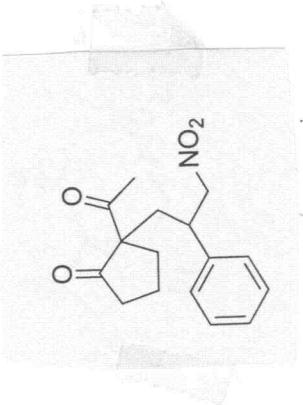


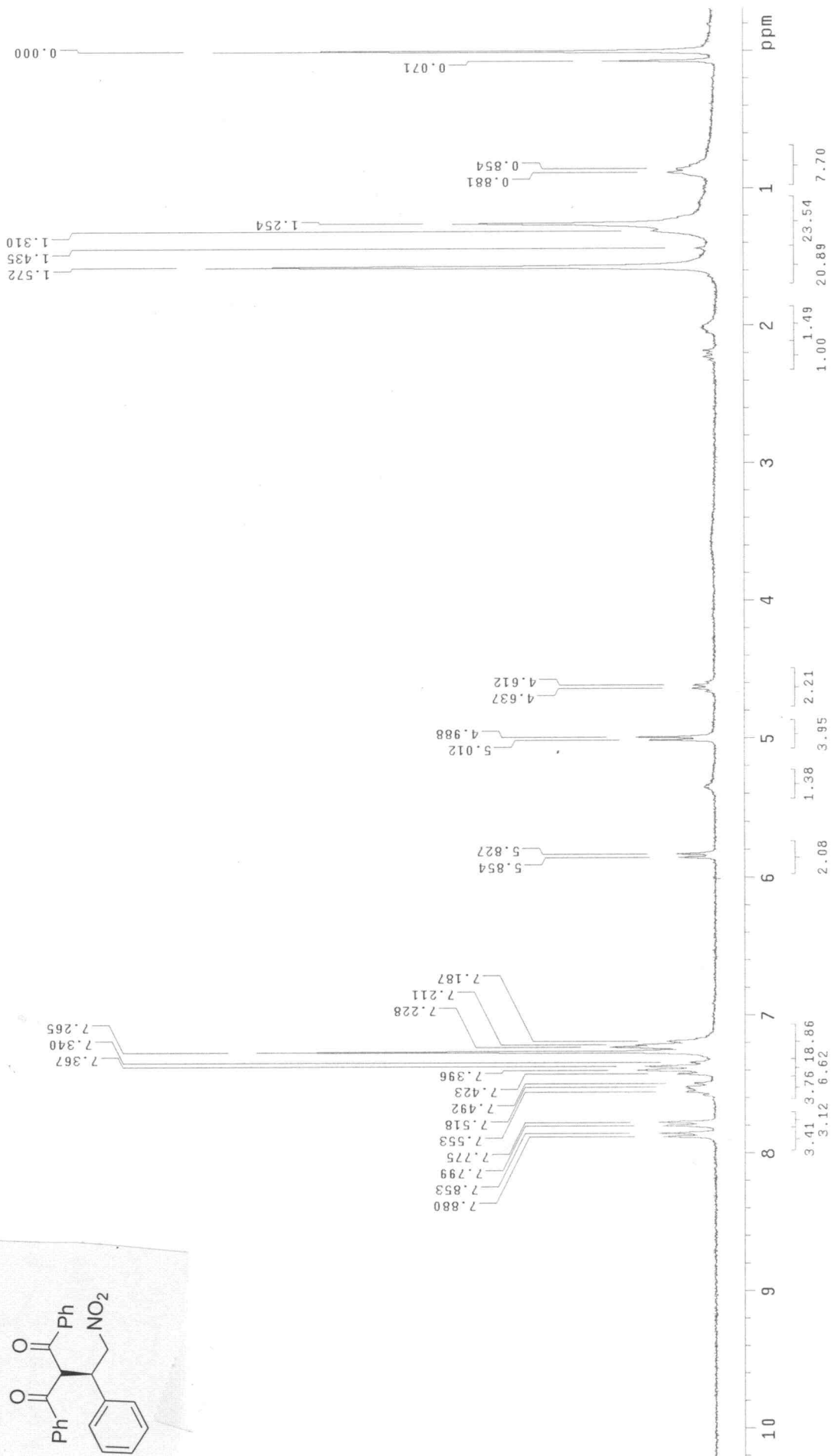
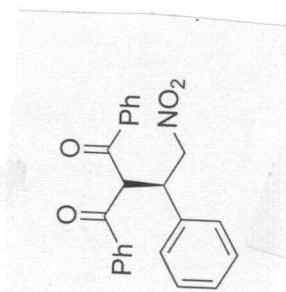
C-22h355a-r

Solvent: CDCl3
Ambient temperature
Mercury-300B5 "mercury300"

Relax. delay 1.000 sec
Acq. time 0.590 sec
Width 17636.7 Hz
128 repetitions
OBSERVED Q13. 75.455576 MHz
DECOUPLE Q13. 309.0897172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
D1 1.00000000 sec
D11 0.00000000 sec
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 3 min, 15 sec



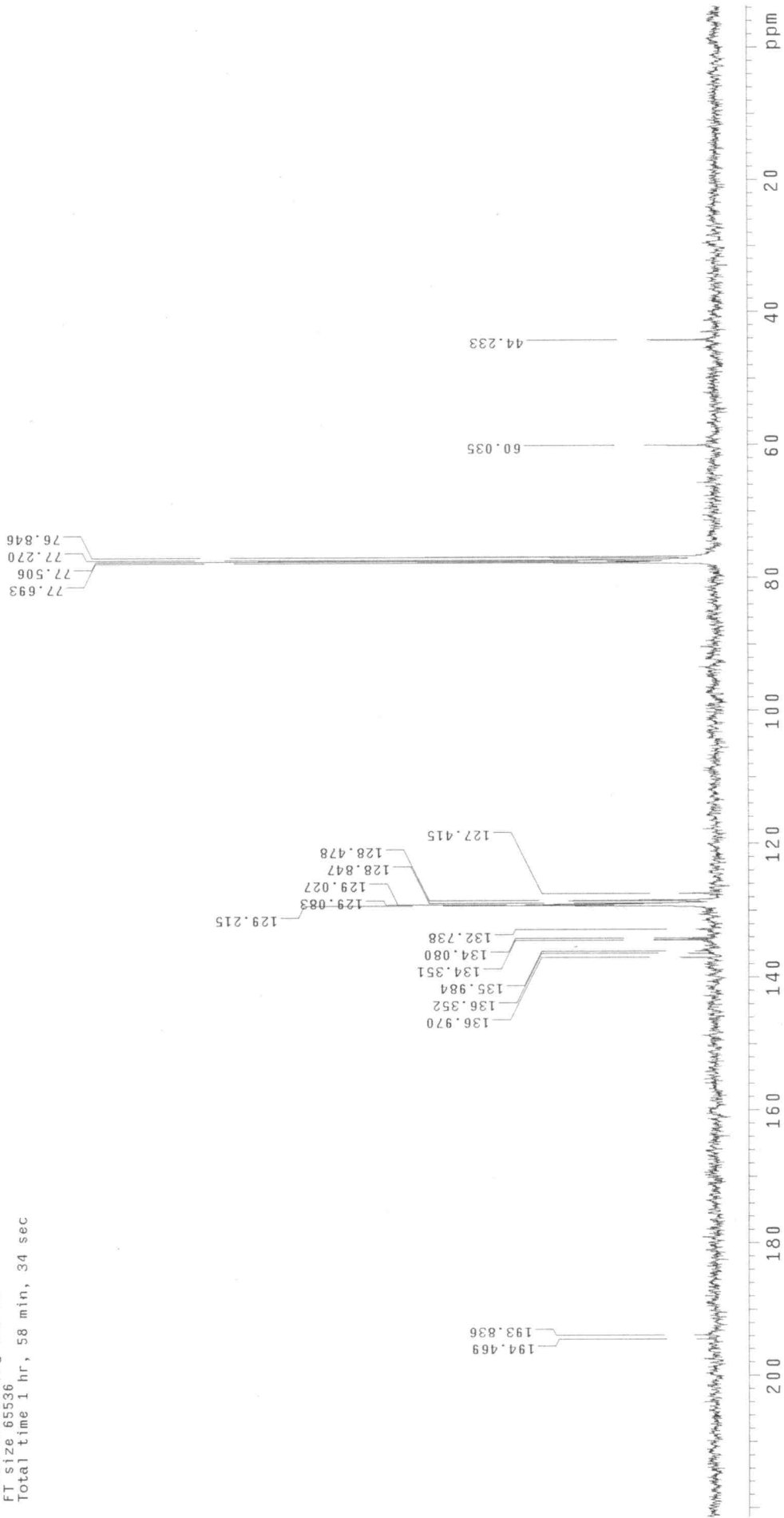
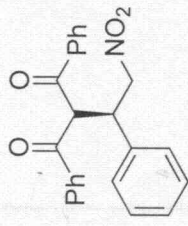




Solvent: CDCl3
 Ambient temperature
 Mercury-300BB "mercury300"

Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 1.000 sec
 Width 17182.1 Hz

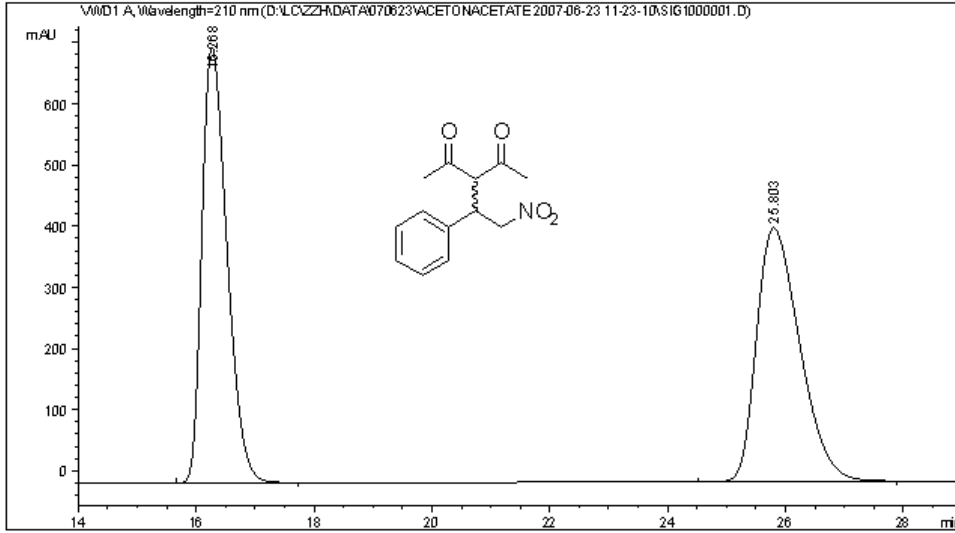
256 repetitions
 OBSERVE C13, 75.4552576 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 65536
 Total time 1 hr, 58 min, 34 sec



```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 6/23/2007 11:26:21 AM    Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\ZZH\data\070623\ACETONACETATE 2007-06-23 11-23-10\ACETONACETATE.M
Last changed    : 6/23/2007 11:17:32 AM by ZZH
Analysis Method : D:\LC\ZZH\DATA\070623\ACETONACETATE 2007-06-23 11-23-10\SIG1000001.D\DA.M (
ACETONACETATE.M)
Last changed    : 11/4/2007 4:11:47 PM by liang gang
                 (modified after loading)
Method Info     : acetanacetate
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

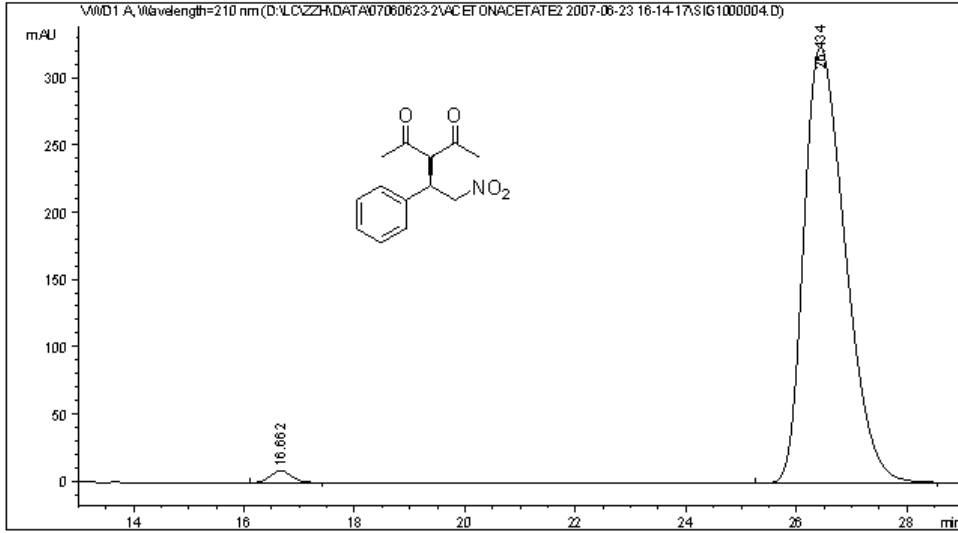
Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 16.268 | BB | 0.4598 | 2.10315e4 | 711.76862 | 49.9311 |
| 2 | 25.803 | BB | 0.7942 | 2.10896e4 | 414.72787 | 50.0689 |

Totals : 4.21211e4 1126.49649

=====
 *** End of Report ***

```
=====  
Acq. Operator   : ZZH                               Seq. Line :    4  
Acq. Instrument : Instrument 1                     Location  : Vial 14  
Injection Date  : 6/23/2007 5:50:28 PM           Inj       :    1  
                                                    Inj Volume: 5 µl  
  
Acq. Method     : D:\LC\ZZH\data\07060623-2\ACETONACETATE2 2007-06-23 16-14-17\ACETONACETATE.M  
Last changed    : 6/23/2007 11:17:32 AM by ZZH  
Analysis Method : D:\LC\ZZH\DATA\07060623-2\ACETONACETATE2 2007-06-23 16-14-17\SIG1000004.D\DA.M  
                (ACETONACETATE.M)  
Last changed    : 8/1/2007 9:26:37 PM by ZZH  
                (modified after loading)  
Method Info     : acetonacetate
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 16.662 | BB | 0.4256 | 242.65547 | 8.86645 | 1.4208 |
| 2 | 26.434 | BB | 0.8101 | 1.68361e4 | 323.96786 | 98.5792 |

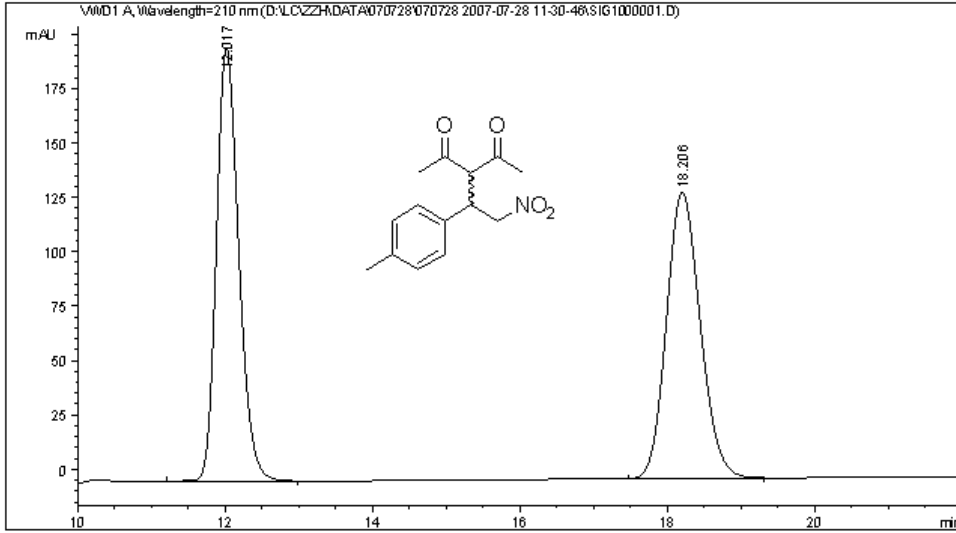
Totals : 1.70787e4 332.83432

=====
*** End of Report ***

Data File D:\LC\ZHZH\DATA\070728\070728 2007-07-28 11-30-46\SIG1000001.D
 Sample Name: ra-zzh255

```

=====
Acq. Operator   : ZZH                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 51
Injection Date  : 7/28/2007 11:32:15 AM            Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\070728\070728 2007-07-28 11-30-46\ASH-210-85-15-30MIN.M
Last changed    : 7/28/2007 11:30:43 AM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\070728\070728 2007-07-28 11-30-46\SIG1000001.D\DA.M (ASH-210-85-15-30MIN.M)
Last changed    : 7/31/2007 8:28:02 PM by ZZH
                  (modified after loading)
Method Info     : ASH-210-85-15-30min
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm

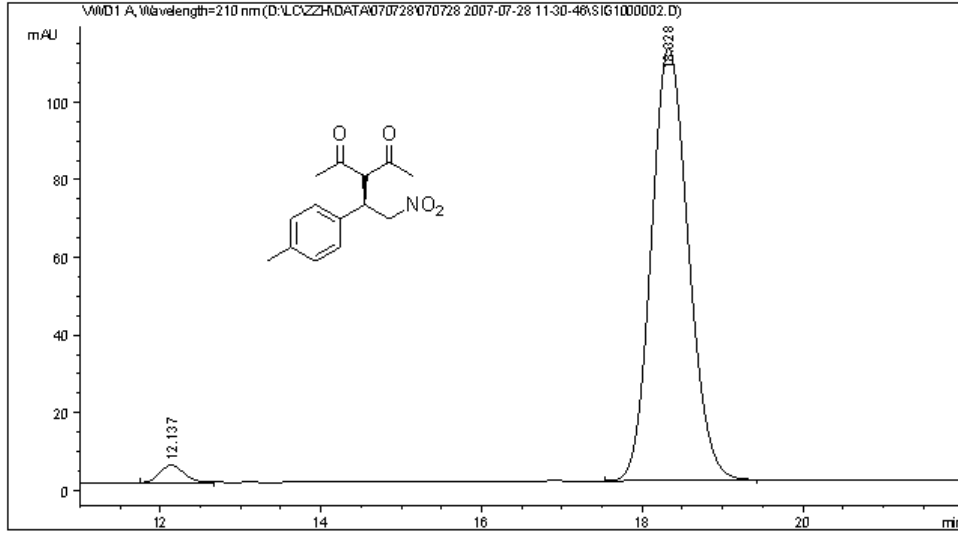
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 12.017 | BB | 0.3258 | 4197.52637 | 199.28558 | 50.0540 |
| 2 | 18.206 | BB | 0.4941 | 4188.47412 | 131.44423 | 49.9460 |

Totals : 8386.00049 330.72981

=====
 *** End of Report ***

Data File D:\LC\ZHZH\DATA\070728\070728 2007-07-28 11-30-46\SIG1000002.D
Sample Name: zzh262a

```
=====
Acq. Operator   : ZZH                               Seq. Line :    2
Acq. Instrument : Instrument 1                       Location  : Vial 52
Injection Date  : 7/28/2007 12:03:47 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\070728\070728 2007-07-28 11-30-46\ASH-210-85-15-30MIN.M
Last changed    : 7/28/2007 11:30:43 AM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\070728\070728 2007-07-28 11-30-46\SIG1000002.D\DA.M (ASH-210-
85-15-30MIN.M)
Last changed    : 7/31/2007 8:31:12 PM by ZZH
                : (modified after loading)
Method Info     : ASH-210-85-15-30min
=====
```



=====
Area Percent Report
=====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 12.137 | BB | 0.3174 | 92.88767 | 4.53884 | 2.5354 |
| 2 | 18.328 | BB | 0.4987 | 3570.77368 | 111.54522 | 97.4646 |

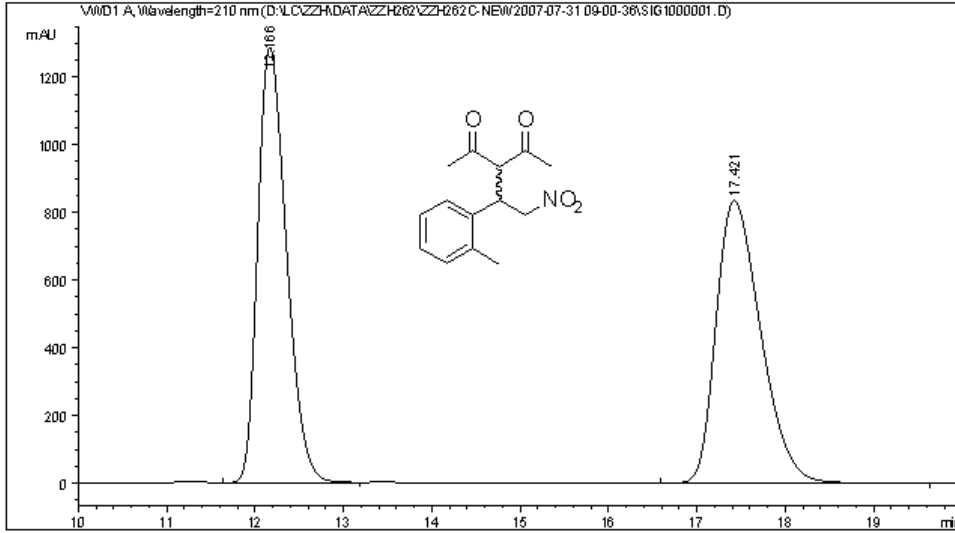
Totals : 3663.66135 116.08406

=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH262\ZZH262C-NEW 2007-07-31 09-00-36\SIG1000001.D
 Sample Name: RC-ZZH262C-NEW

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 77
Injection Date  : 7/31/2007 9:02:08 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262\ZZH262C-NEW 2007-07-31 09-00-36\ASH-85-15-210NM-100MIN.M
Last changed    : 7/30/2007 9:53:20 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262\ZZH262C-NEW 2007-07-31 09-00-36\SIG1000001.D\DA.M (ASH-
85-15-210NM-100MIN.M)
Last changed    : 7/31/2007 8:38:36 PM by ZZH
                 (modified after loading)
Method Info     : ASH-85-15-210nm-100min
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

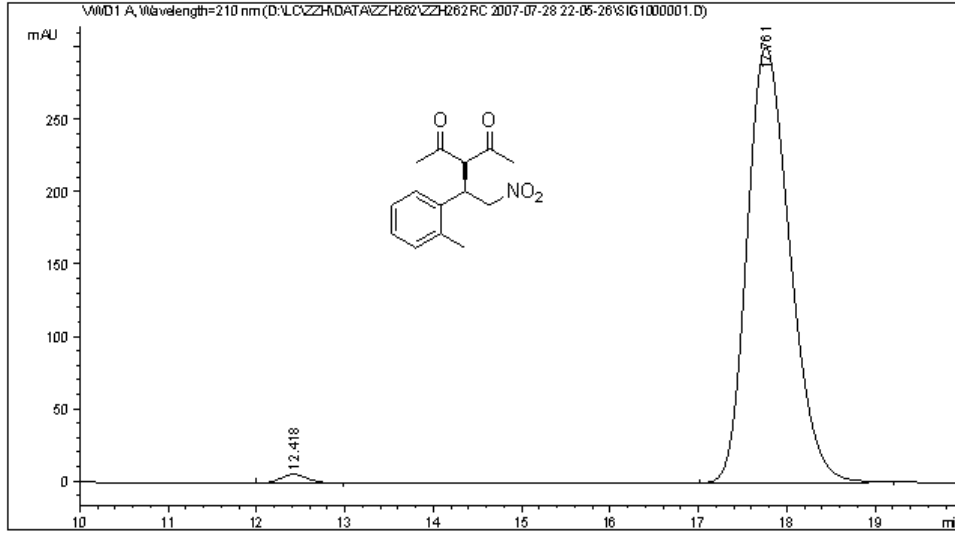
Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 12.166 | VB | 0.3523 | 2.90842e4 | 1287.63086 | 49.8585 |
| 2 | 17.421 | BB | 0.5456 | 2.92493e4 | 834.87976 | 50.1415 |

Totals : 5.83336e4 2122.51062

=====
 *** End of Report ***


```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 63
Injection Date  : 7/28/2007 10:06:54 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method    : D:\LC\ZZH\data\ZZH262\ZZH262RC 2007-07-28 22-05-26\ASH-85-15-210NM-100MIN.M
Last changed   : 7/28/2007 8:33:29 PM by ZZH
Analysis Method: D:\LC\ZZH\DATA\ZZH262\ZZH262RC 2007-07-28 22-05-26\SIG1000001.D\DA.M (ASH-85-
15-210NM-100MIN.M)
Last changed   : 7/31/2007 8:40:20 PM by ZZH
                (modified after loading)
Method Info    : ASH-85-15-210nm-100min
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

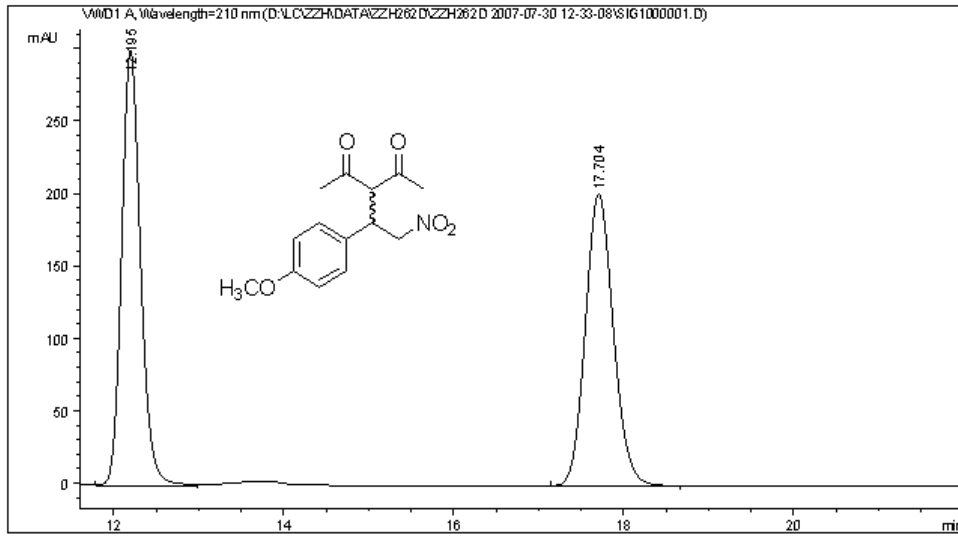
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 12.418 | BB | 0.3275 | 125.21668 | 5.97533 | 1.2232 |
| 2 | 17.761 | BB | 0.5250 | 1.01119e4 | 300.55719 | 98.7768 |

Totals : 1.02372e4 306.53252

=====
*** End of Report ***

Data File D:\LC\ZHZH\DATA\ZZH262D\ZZH262D 2007-07-30 12-33-08\SIG1000001.D
Sample Name: rd-zzh265d

=====
Acq. Operator : ZZH Seq. Line : 1
Acq. Instrument : Instrument 1 Location : Vial 65
Injection Date : 7/30/2007 12:34:37 PM Inj : 1
 Inj Volume: 5 µl
Acq. Method : D:\LC\ZHZH\data\ZZH262D\ZZH262D 2007-07-30 12-33-08\ADH-20-80-210NM-30MIN.M
Last changed : 7/30/2007 12:06:59 PM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\ZZH262D\ZZH262D 2007-07-30 12-33-08\SIG1000001.D\DA.M (ADH-20-
80-210NM-30MIN.M)
Last changed : 7/31/2007 9:25:43 PM by ZZH
 (modified after loading)
Method Info : AD-20/80-210nm-0.8nm-30min
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 12.195 | VB | 0.2384 | 4637.95898 | 300.32980 | 50.1720 |
| 2 | 17.704 | BB | 0.3540 | 4606.15967 | 201.56017 | 49.8280 |

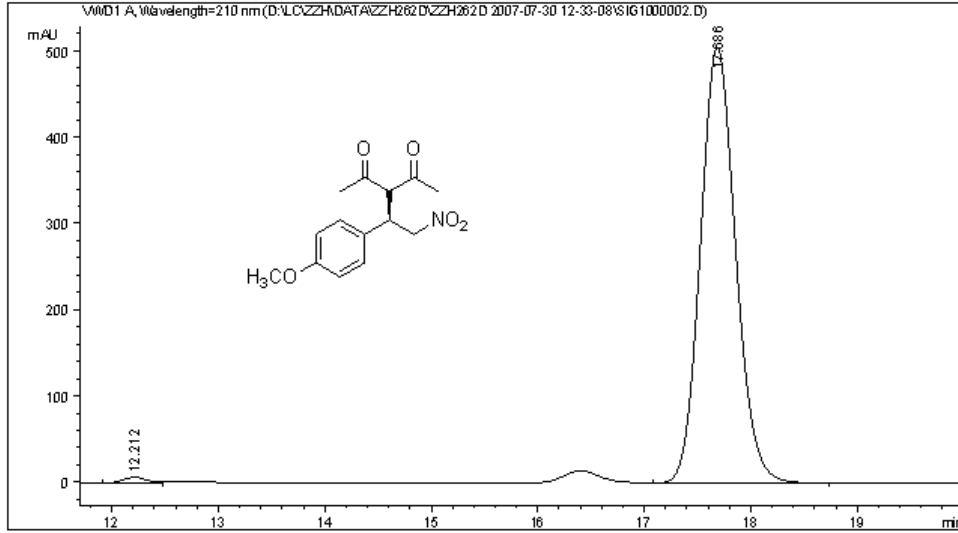
Totals : 9244.11865 501.88997

=====
*** End of Report ***

```

=====
Acq. Operator   : ZZH                               Seq. Line :    2
Acq. Instrument : Instrument 1                       Location  : Vial 66
Injection Date  : 7/30/2007 1:06:18 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZZH262D\ZZH262D 2007-07-30 12-33-08\ADH-20-80-210NM-30MIN.M
Last changed    : 7/30/2007 12:06:59 PM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\ZZH262D\ZZH262D 2007-07-30 12-33-08\SIG1000002.D\DA.M (ADH-20-
80-210NM-30MIN.M)
Last changed    : 7/31/2007 8:46:44 PM by ZZH
                  (modified after loading)
Method Info     : AD-20/80-210nm-0.8nm-30min
=====

```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====

```

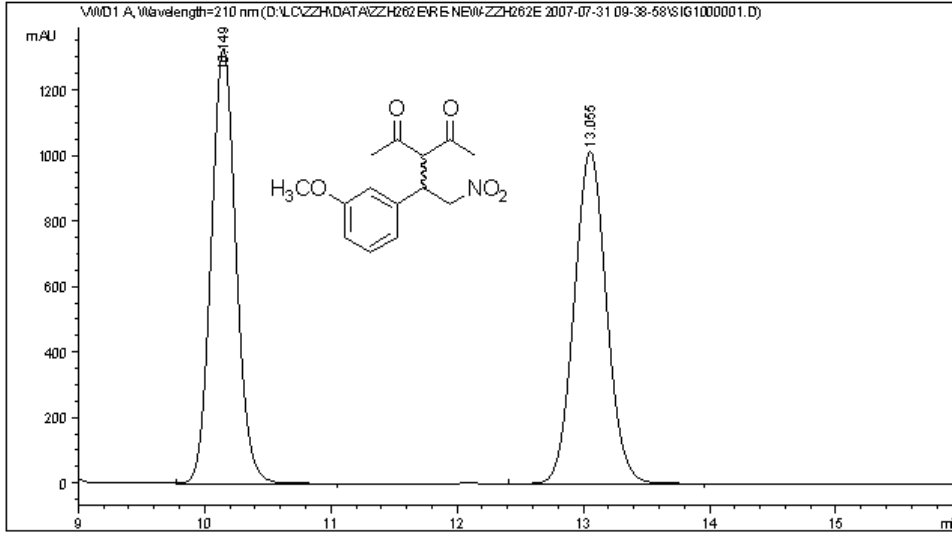
Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 12.212 | BV | 0.2413 | 109.78627 | 6.88587 | 0.9399 |
| 2 | 17.686 | VB | 0.3563 | 1.15714e4 | 504.78906 | 99.0601 |

Totals : 1.16812e4 511.67493

*** End of Report ***

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 78
Injection Date  : 7/31/2007 9:40:31 AM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262E\RE-NEW-ZZH262E 2007-07-31 09-38-58\ADH-85-15-210NM-1ML-100MIN.M
Last changed    : 7/30/2007 5:24:01 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262E\RE-NEW-ZZH262E 2007-07-31 09-38-58\SIG1000001.D\DA.M (
ADH-85-15-210NM-1ML-100MIN.M)
Last changed    : 7/31/2007 9:27:32 PM by ZZH
                 (modified after loading)
Method Info     : ADH-85-15-210NM-1ML-100MIN
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

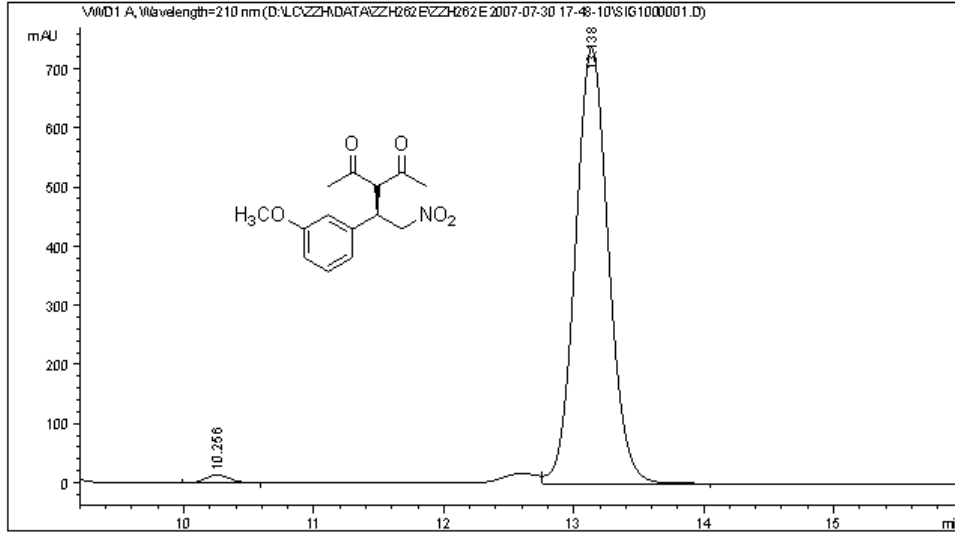
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 10.149 | BB | 0.2115 | 1.81224e4 | 1329.18262 | 50.0965 |
| 2 | 13.055 | VB | 0.2763 | 1.80526e4 | 1013.26898 | 49.9035 |

Totals : 3.61750e4 2342.45160

=====
*** End of Report ***

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 70
Injection Date  : 7/30/2007 5:49:43 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262E\ZZH262E 2007-07-30 17-48-10\ADH-85-15-210NM-1ML-100MIN.
M
Last changed    : 7/30/2007 5:24:01 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262E\ZZH262E 2007-07-30 17-48-10\SIG1000001.D\DA.M (ADH-85-
15-210NM-1ML-100MIN.M)
Last changed    : 7/31/2007 8:49:43 PM by ZZH
(modified after loading)
Method Info     : ADH-85-15-210NM-1ML-100MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

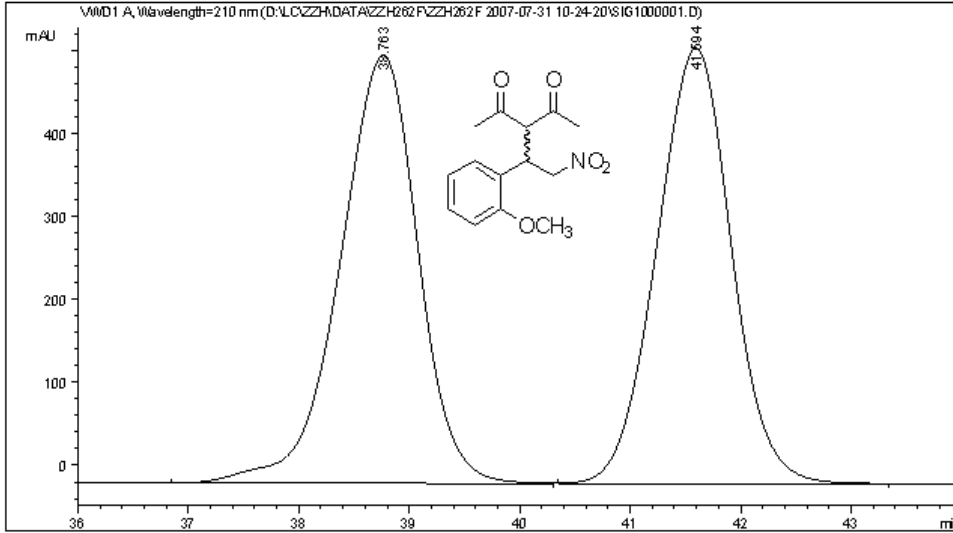
Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|----------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 10.256 | BV | 0.2068 | 181.75406 | 13.60873 | 1.3796 |
| 2 | 13.138 | WB | 0.2727 | 1.29922e4 | 736.61670 | 98.6204 |
| Totals : | | | | 1.31740e4 | 750.22543 | |

=====
 *** End of Report ***

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 75
Injection Date  : 7/31/2007 10:25:49 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262F\ZZH262F 2007-07-31 10-24-20\ADH-97-3-210NM-05ML-100MIN.
M
Last changed    : 7/30/2007 3:05:36 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262F\ZZH262F 2007-07-31 10-24-20\SIG1000001.D\DA.M (ADH-97-
3-210NM-05ML-100MIN.M)
Last changed    : 7/31/2007 8:51:40 PM by ZZH
                 (modified after loading)
Method Info     : ADH-97-3-210NM-05ML/MIN-100MIN
  
```



Area Percent Report

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 38.763 | BB | 0.7534 | 2.53313e4 | 515.93793 | 50.7051 |
| 2 | 41.594 | BB | 0.7310 | 2.46268e4 | 524.76398 | 49.2949 |

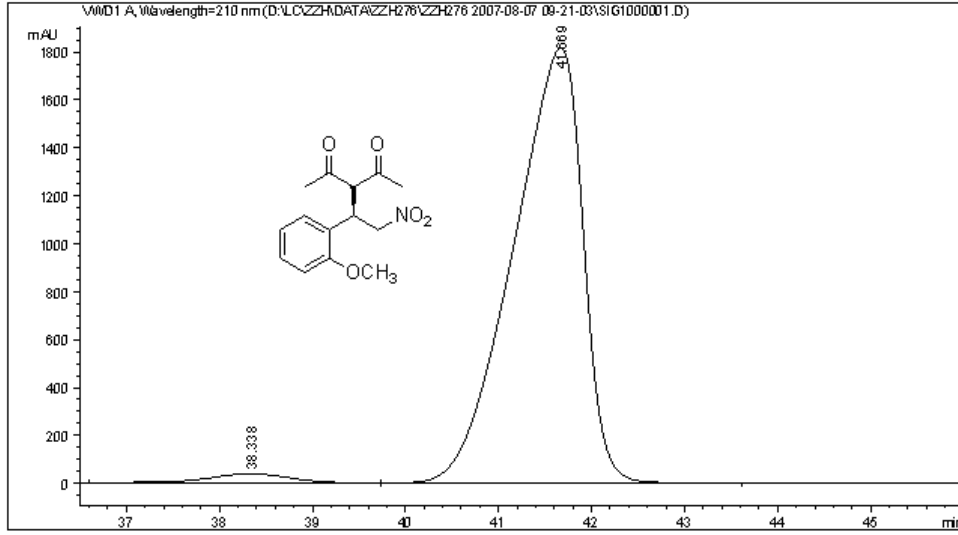
Totals : 4.99582e4 1040.70190

*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH276\ZZH276 2007-08-07 09-21-03\SIG1000001.D
 Sample Name: zzh276a-2

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 90
Injection Date  : 8/7/2007 9:22:30 AM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH276\ZZH276 2007-08-07 09-21-03\ADH-97-3-210NM-05ML-100MIN.M
Last changed    : 7/30/2007 3:05:36 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH276\ZZH276 2007-08-07 09-21-03\SIG1000001.D\DA.M (ADH-97-3-
                210NM-05ML-100MIN.M)
Last changed    : 11/4/2007 4:14:47 PM by liang gang
                (modified after loading)
Method Info     : ADH-97-3-210NM-05ML/MIN-100MIN
=====
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm

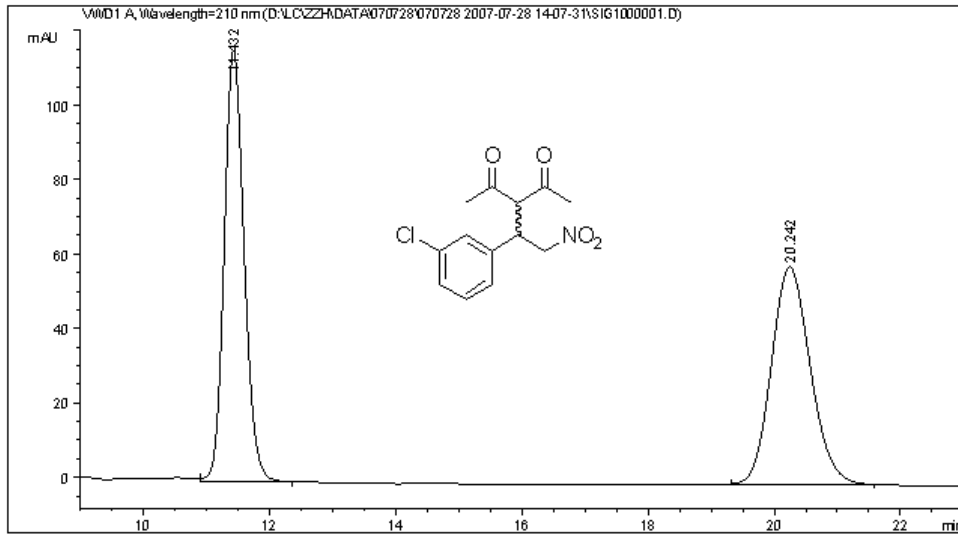
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Area % | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|---------|--------------|---------|
| 1 | 38.338 | BV | 0.9714 | 2424.26953 | 2.5025 | 38.45215 | 2.5025 |
| 2 | 41.669 | VB | 0.7926 | 9.44513e4 | 97.4975 | 1817.75073 | 97.4975 |

Totals : 9.68756e4 1856.20288

=====
 *** End of Report ***

Data File D:\LC\ZHZH\DATA\070728\070728 2007-07-28 14-07-31\SIG1000001.D
Sample Name: ra-zzh263c

```
=====
Acq. Operator   : ZZH                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 53
Injection Date  : 7/28/2007 2:09:03 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\070728\070728 2007-07-28 14-07-31\ASH-75-25-210NM-30MIN.M
Last changed    : 7/28/2007 1:58:22 PM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\070728\070728 2007-07-28 14-07-31\SIG1000001.D\DA.M (ASH-75-25-
                210NM-30MIN.M)
Last changed    : 7/31/2007 8:33:10 PM by ZZH
                (modified after loading)
Method Info     : ASH-75-25-210NM-30MIN.M
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

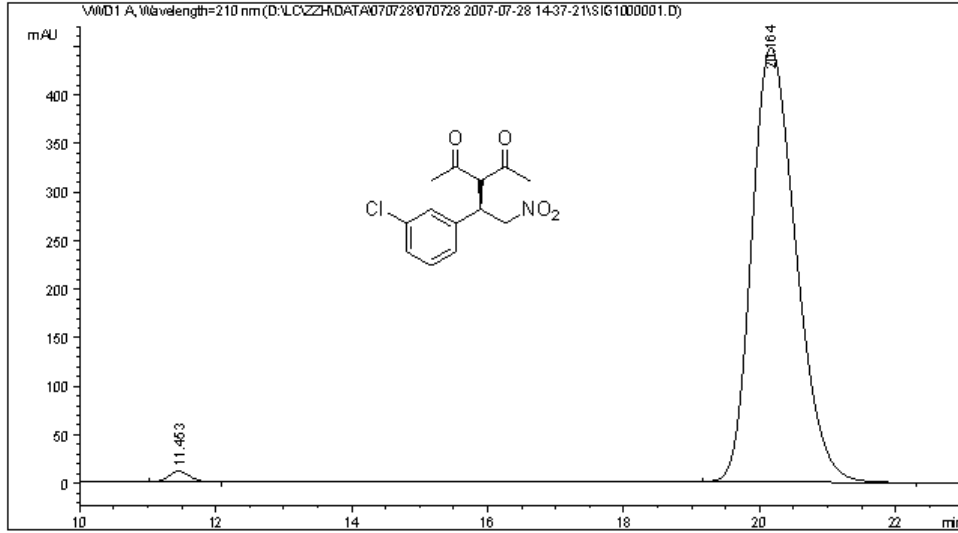
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 11.432 | BB | 0.3391 | 2545.07104 | 115.92770 | 50.4190 |
| 2 | 20.242 | BB | 0.6667 | 2502.77246 | 58.48568 | 49.5810 |

Totals : 5047.84351 174.41338

=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\070728\070728 2007-07-28 14-37-21\SIG1000001.D
Sample Name: zzh262h

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 54
Injection Date  : 7/28/2007 2:39:03 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\070728\070728 2007-07-28 14-37-21\ASH-75-25-210NM-30MIN.M
Last changed    : 7/28/2007 2:36:58 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\070728\070728 2007-07-28 14-37-21\SIG1000001.D\DA.M (ASH-75-25-
210NM-30MIN.M)
Last changed    : 7/31/2007 8:34:36 PM by ZZH
                 (modified after loading)
Method Info     : ASH-75-25-210NM-30MIN.M
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

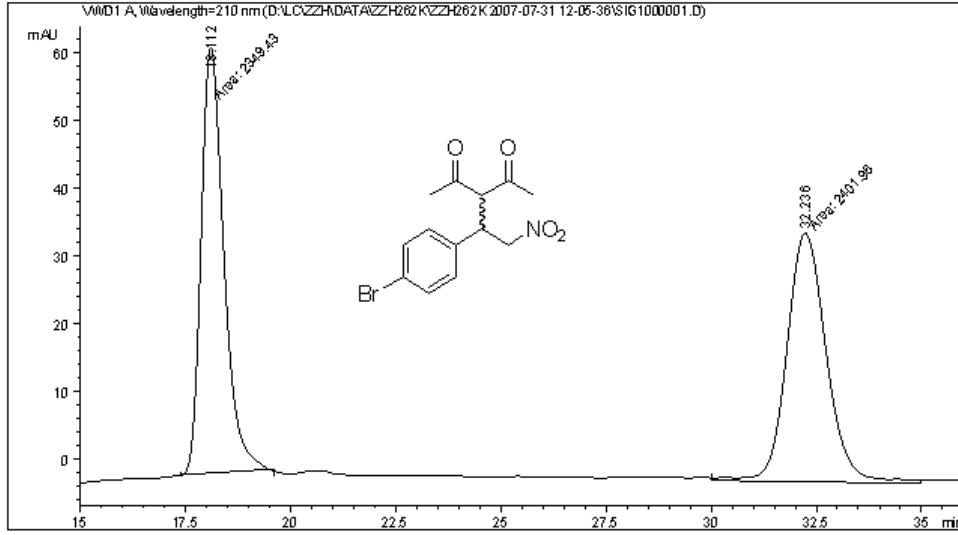
Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 11.453 | BB | 0.3351 | 239.99431 | 11.10519 | 1.1788 |
| 2 | 20.164 | BB | 0.7023 | 2.01200e4 | 447.41919 | 98.8212 |

Totals : 2.03600e4 458.52438

=====
*** End of Report ***

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 81
Injection Date  : 7/31/2007 12:06:55 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZZH262K\ZZH262K 2007-07-31 12-05-36\ASH-85-15-210NM-1ML-40MIN.M
Last changed    : 7/31/2007 12:02:08 PM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\ZZH262K\ZZH262K 2007-07-31 12-05-36\SIG1000001.D\DA.M (ASH-85-15-210NM-1ML-40MIN.M)
Last changed    : 7/31/2007 9:02:18 PM by ZZH
                 (modified after loading)
Method Info     : ASH-85-15-210NM-1ML-40MIN
=====
```



=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=210 nm

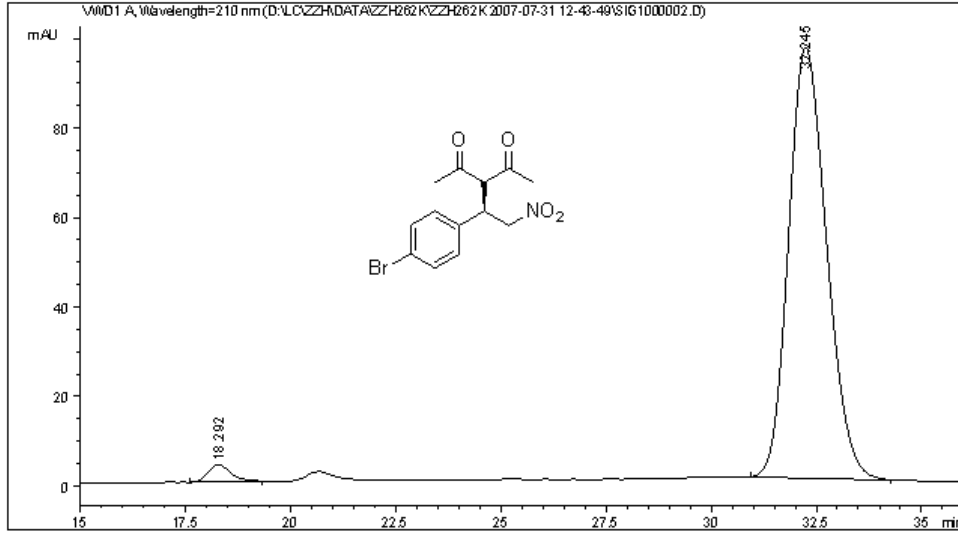
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 18.112 | MM | 0.6237 | 2349.42749 | 62.78243 | 49.4471 |
| 2 | 32.236 | MM | 1.0909 | 2401.96460 | 36.69532 | 50.5529 |

Totals : 4751.39209 99.47775

=====
*** End of Report ***

```

=====
Acq. Operator   : ZZH                               Seq. Line :    2
Acq. Instrument : Instrument 1                       Location  : Vial 82
Injection Date  : 7/31/2007 1:26:39 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method    : D:\LC\ZHZH\data\ZZH262K\ZZH262K 2007-07-31 12-43-49\ASH-85-15-210NM-1ML-40MIN.M
Last changed   : 7/31/2007 12:02:08 PM by ZZH
Analysis Method: D:\LC\ZHZH\DATA\ZZH262K\ZZH262K 2007-07-31 12-43-49\SIG1000002.D\DA.M (ASH-85-15-210NM-1ML-40MIN.M)
Last changed   : 7/31/2007 9:03:11 PM by ZZH
                (modified after loading)
Method Info    : ASH-85-15-210NM-1ML-40MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

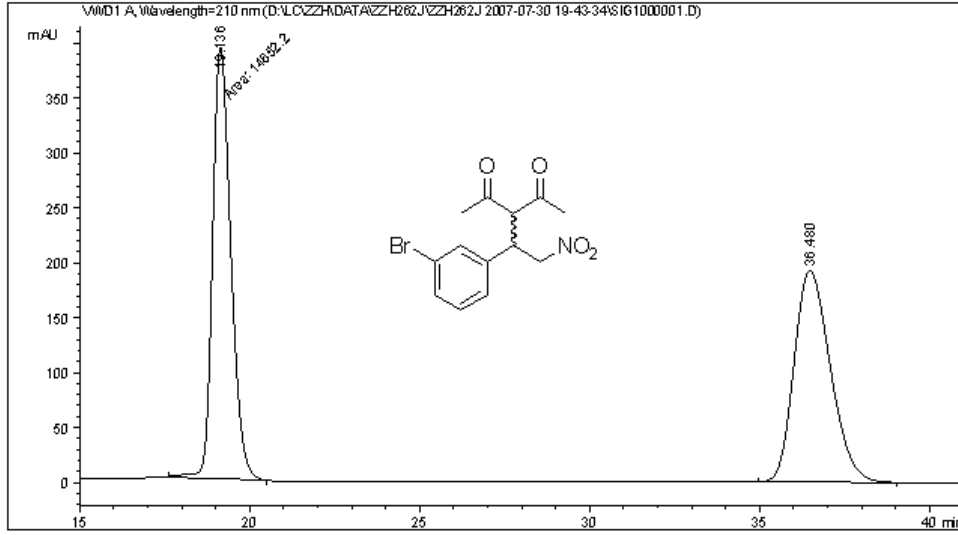
Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 18.292 | BB | 0.5871 | 150.65996 | 3.91662 | 2.4273 |
| 2 | 32.245 | BB | 0.9695 | 6056.11523 | 96.31187 | 97.5727 |

Totals : 6206.77519 100.22849

=====
 *** End of Report ***

=====
Acq. Operator : ZZH Seq. Line : 1
Acq. Instrument : Instrument 1 Location : Vial 73
Injection Date : 7/30/2007 7:44:57 PM Inj : 1
 Inj Volume : 5 µl
Acq. Method : D:\LC\ZZH\data\ZZH262J\ZZH262J 2007-07-30 19-43-34\ASH-85-15-210NM-100MIN.M
Last changed : 7/28/2007 8:33:29 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262J\ZZH262J 2007-07-30 19-43-34\SIG1000001.D\DA.M (ASH-85-
15-210NM-100MIN.M)
Last changed : 7/31/2007 8:59:07 PM by ZZH
 (modified after loading)
Method Info : ASH-85-15-210nm-100min
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

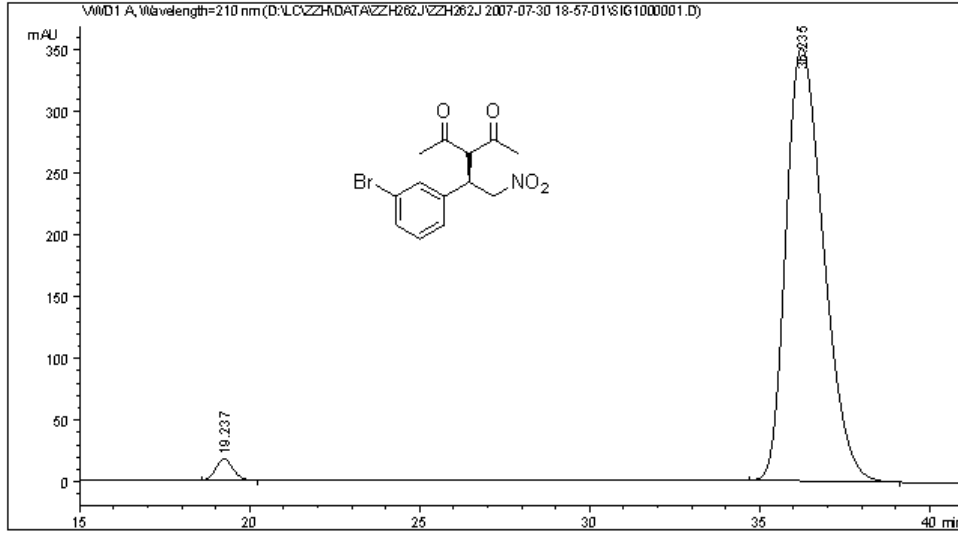
Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 19.136 | MM | 0.6219 | 1.46522e4 | 392.70370 | 50.6306 |
| 2 | 36.480 | BB | 1.1569 | 1.42872e4 | 192.85721 | 49.3694 |

Totals : 2.89395e4 585.56091

=====
*** End of Report ***

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 74
Injection Date  : 7/30/2007 6:58:40 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262J\ZZH262J 2007-07-30 18-57-01\ASH-85-15-210NM-100MIN.M
Last changed    : 7/28/2007 8:33:29 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262J\ZZH262J 2007-07-30 18-57-01\SIG1000001.D\DA.M (ASH-85-
15-210NM-100MIN.M)
Last changed    : 7/31/2007 8:57:26 PM by ZZH
                 (modified after loading)
Method Info     : ASH-85-15-210nm-100min
=====
```



=====
Area Percent Report
=====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 19.237 | BB | 0.5430 | 605.48718 | 17.32972 | 2.1978 |
| 2 | 36.235 | BB | 1.1943 | 2.69438e4 | 351.48270 | 97.8022 |

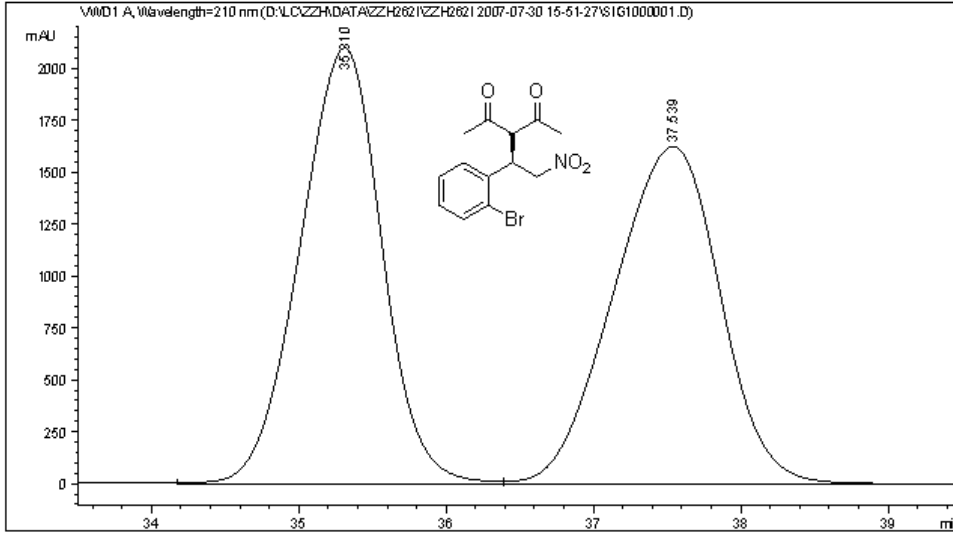
Totals : 2.75493e4 368.81242

=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH262I\ZZH262I 2007-07-30 15-51-27\SIG1000001.D
 Sample Name: zzh262i-0

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 67
Injection Date  : 7/30/2007 3:53:01 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH262I\ZZH262I 2007-07-30 15-51-27\ADH-97-3-210NM-05ML-100MIN.
M
Last changed    : 7/30/2007 3:05:36 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH262I\ZZH262I 2007-07-30 15-51-27\SIG1000001.D\DA.M (ADH-97-
3-210NM-05ML-100MIN.M)
Last changed    : 7/31/2007 8:54:23 PM by ZZH
                 (modified after loading)
Method Info     : ADH-97-3-210NM-05ML/MIN-100MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm

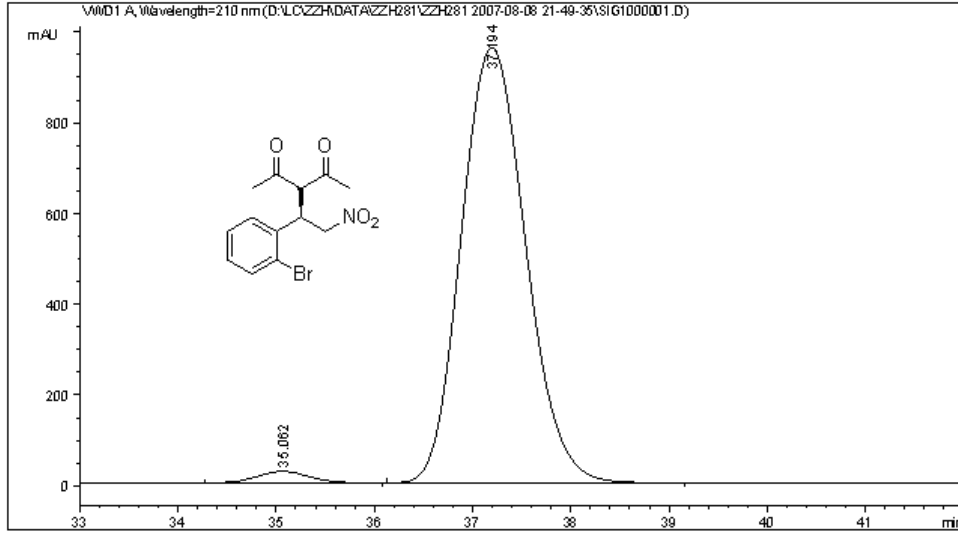
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 35.310 | EV | 0.6045 | 8.12097e4 | 2097.88965 | 49.7992 |
| 2 | 37.539 | WBA | 0.7990 | 8.18648e4 | 1624.70959 | 50.2008 |

Totals : 1.63075e5 3722.59924

=====
 *** End of Report ***

Data File D:\LC\ZHZH\DATA\ZZH281\ZZH281 2007-08-08 21-49-35\SIG1000001.D
Sample Name: zzh281i

```
=====
Acq. Operator   : ZZH                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 5
Injection Date  : 8/8/2007 9:51:03 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZHZH\data\ZZH281\ZZH281 2007-08-08 21-49-35\ADH-97-3-210NM-05ML-100MIN.M
Last changed    : 7/30/2007 3:05:36 PM by ZZH
Analysis Method : D:\LC\ZHZH\DATA\ZZH281\ZZH281 2007-08-08 21-49-35\SIG1000001.D\DA.M (ADH-97-3-
                210NM-05ML-100MIN.M)
Last changed    : 11/4/2007 4:20:48 PM by liang gang
                (modified after loading)
Method Info     : ADH-97-3-210NM-05ML/MIN-100MIN
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 35.062 | BB | 0.5733 | 925.64673 | 25.16572 | 2.0726 |
| 2 | 37.194 | BB | 0.7210 | 4.37359e4 | 959.69708 | 97.9274 |

Totals : 4.46615e4 984.86280

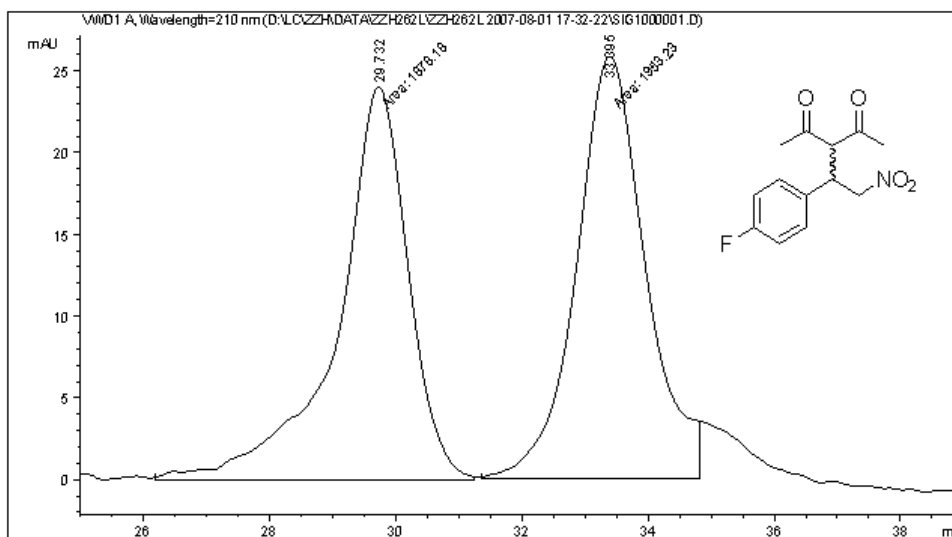
=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH262L\ZZH262L 2007-08-01 17-32-22\SIG1000001.D
Sample Name: RL-ZZH262L

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 79
Injection Date  : 8/1/2007 5:33:54 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method    : D:\LC\ZZH\data\ZZH262L\ZZH262L 2007-08-01 17-32-22\ODH-90-10-1ML-210NM-40MIN.M
Last changed   : 7/31/2007 7:54:49 PM by ZZH
Analysis Method: D:\LC\ZZH\DATA\ZZH262L\ZZH262L 2007-08-01 17-32-22\SIG1000001.D\DA.M (ODH-90-10-1ML-210NM-40MIN.M)
Last changed   : 8/1/2007 6:43:40 PM by ZZH
                                           (modified after loading)
Method Info    : ODH-90-10-1ML-210NM-40MIN
=====

```



=====
Area Percent Report
=====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 29.732 | MM | 1.2985 | 1878.17847 | 24.10779 | 49.0205 |
| 2 | 33.395 | MF | 1.2610 | 1953.23242 | 25.81499 | 50.9795 |

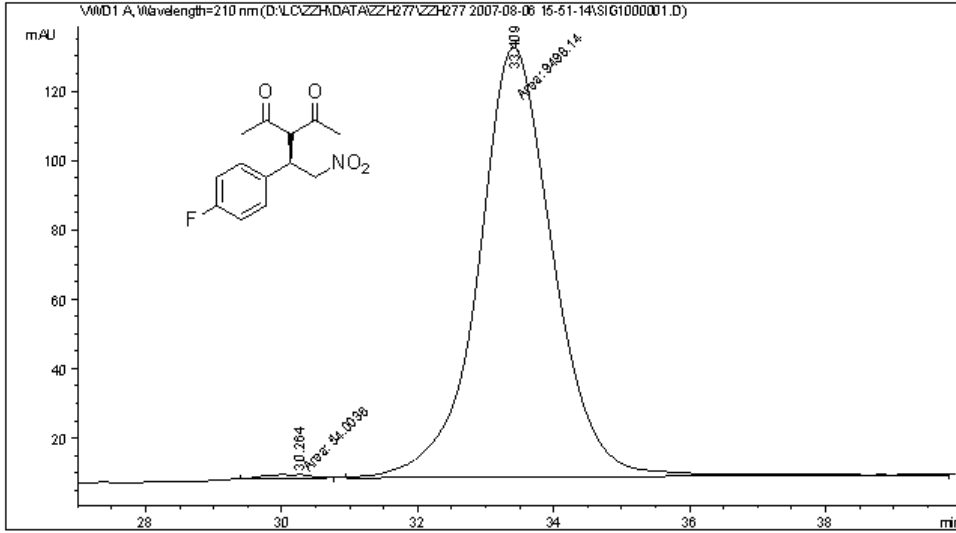
Totals : 3831.41089 49.92278

=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH277\ZZH277 2007-08-06 15-51-14\SIG1000001.D
 Sample Name: zzh277c-262f-2

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 96
Injection Date  : 8/6/2007 3:52:37 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH277\ZZH277 2007-08-06 15-51-14\ODH-90-10-1ML-210NM-40MIN.M
Last changed    : 7/31/2007 7:54:49 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH277\ZZH277 2007-08-06 15-51-14\SIG1000001.D\DA.M (ODH-90-10-1ML-210NM-40MIN.M)
Last changed    : 10/30/2007 4:35:17 PM by ZZH
                                           (modified after loading)
Method Info     : ODH-90-10-1ML-210NM-40MIN
=====
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

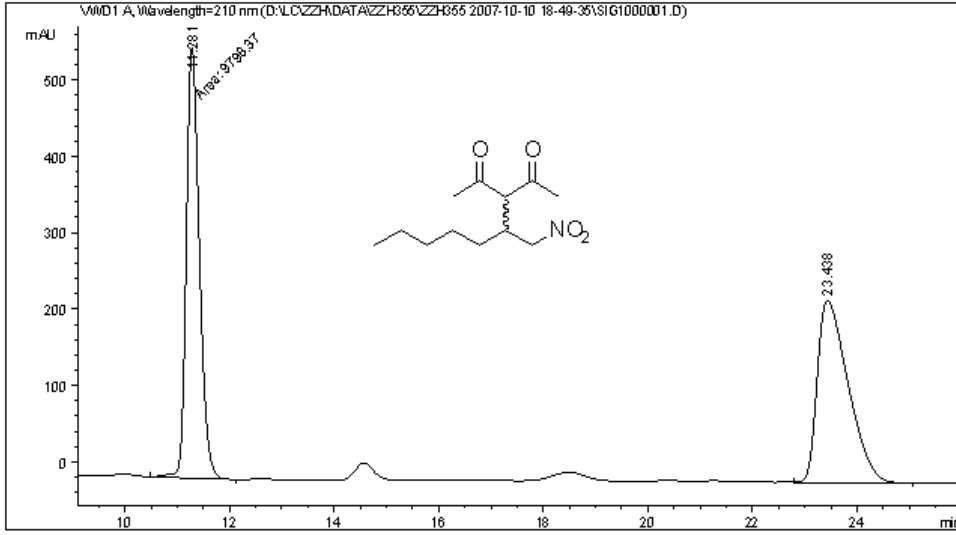
Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 30.264 | MM | 0.8722 | 54.00359 | 1.03196 | 0.5654 |
| 2 | 33.409 | MM | 1.2784 | 9498.13965 | 123.82489 | 99.4346 |

Totals : 9552.14324 124.85685

=====
 *** End of Report ***

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 12
Injection Date  : 10/10/2007 6:51:00 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH355\ZZH355 2007-10-10 18-49-35\ASH-95-5-08-210NM-30MIN.M
Last changed    : 7/30/2007 2:02:51 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 18-49-35\SIG1000001.D\DA.M (ASH-95-5-08-210NM-30MIN.M)
Last changed    : 10/29/2007 7:53:42 PM by ZZH
                 (modified after loading)
Method Info     : ASH-95-5-0.8-210nm-30min
=====
```



=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=210 nm

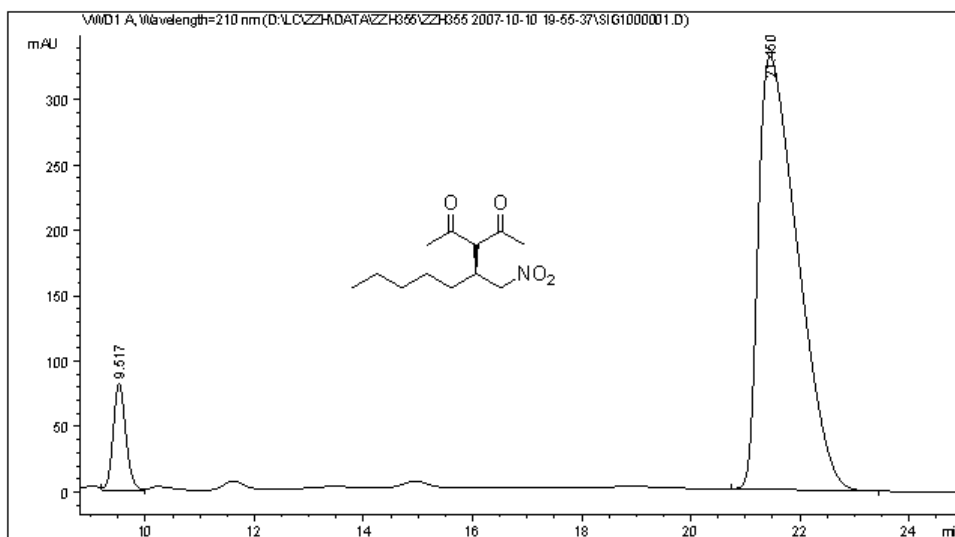
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 11.281 | MM | 0.2896 | 9798.36816 | 563.86017 | 50.1249 |
| 2 | 23.438 | BB | 0.6308 | 9749.53613 | 238.72713 | 49.8751 |

Totals : 1.95479e4 802.58730

=====
*** End of Report ***

```

=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 13
Injection Date  : 10/10/2007 7:57:01 PM    Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method    : D:\LC\ZZH\data\ZZH355\ZZH355 2007-10-10 19-55-37\ASH-95-5-08-210NM-30MIN.M
Last changed   : 7/30/2007 2:02:51 PM by ZZH
Analysis Method: D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 19-55-37\SIG1000001.D\DA.M (ASH-95-5-08-210NM-30MIN.M)
Last changed   : 10/29/2007 7:57:19 PM by ZZH
                                                (modified after loading)
Method Info    : ASH-95-5-0.8-210nm-30min
    
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
    
```

Signal 1: VWD1 A, Wavelength=210 nm

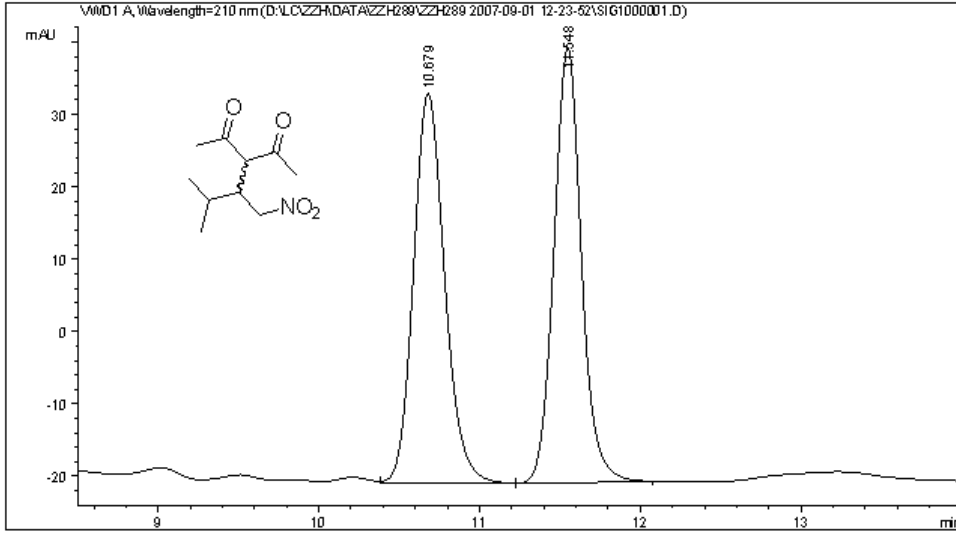
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 9.517 | VV | 0.2489 | 1323.33765 | 81.61952 | 7.3910 |
| 2 | 21.450 | BB | 0.7805 | 1.65814e4 | 331.27127 | 92.6090 |

Totals : 1.79047e4 412.89079

=====
 *** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH289\ZZH289 2007-09-01 12-23-52\SIG1000001.D
Sample Name: ZZH289-R

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 11
Injection Date  : 9/1/2007 12:25:18 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH289\ZZH289 2007-09-01 12-23-52\ADH-95-5-210NM-1ML-100MIN.M
Last changed    : 7/30/2007 2:44:59 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH289\ZZH289 2007-09-01 12-23-52\SIG1000001.D\DA.M (ADH-95-5-
210NM-1ML-100MIN.M)
Last changed    : 10/29/2007 8:01:30 PM by ZZH
                  (modified after loading)
Method Info     : ADH-95-5-210NM-1ML/MIN-100MIN
=====
```



=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=210 nm

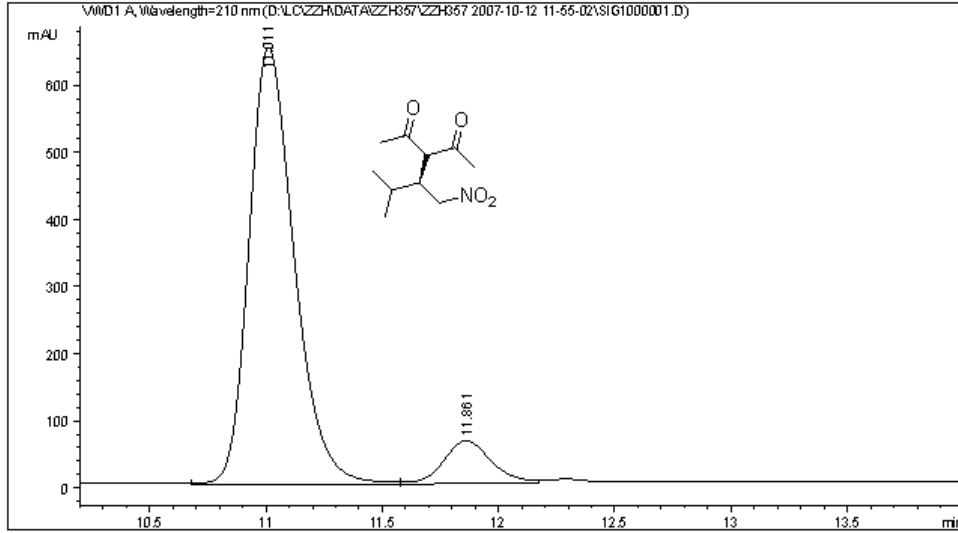
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|-----------|--------------|---------|
| 1 | 10.679 | VV | 0.2071 | 728.49011 | 53.91901 | 50.4404 |
| 2 | 11.548 | VB | 0.1825 | 715.76837 | 60.16836 | 49.5596 |

Totals : 1444.25848 114.08737

=====
*** End of Report ***

```

=====
Acq. Operator   : ZZH                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 19
Injection Date  : 10/12/2007 11:56:25 AM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method    : D:\LC\ZZH\data\ZZH357\ZZH357 2007-10-12 11-55-02\ADH-95-5-210NM-1ML-100MIN.M
Last changed   : 7/30/2007 2:44:59 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH357\ZZH357 2007-10-12 11-55-02\SIG1000001.D\DA.M (ADH-95-5-210NM-1ML-100MIN.M)
Last changed   : 11/4/2007 4:27:41 PM by liang gang
                (modified after loading)
Method Info    : ADH-95-5-210NM-1ML/MIN-100MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm

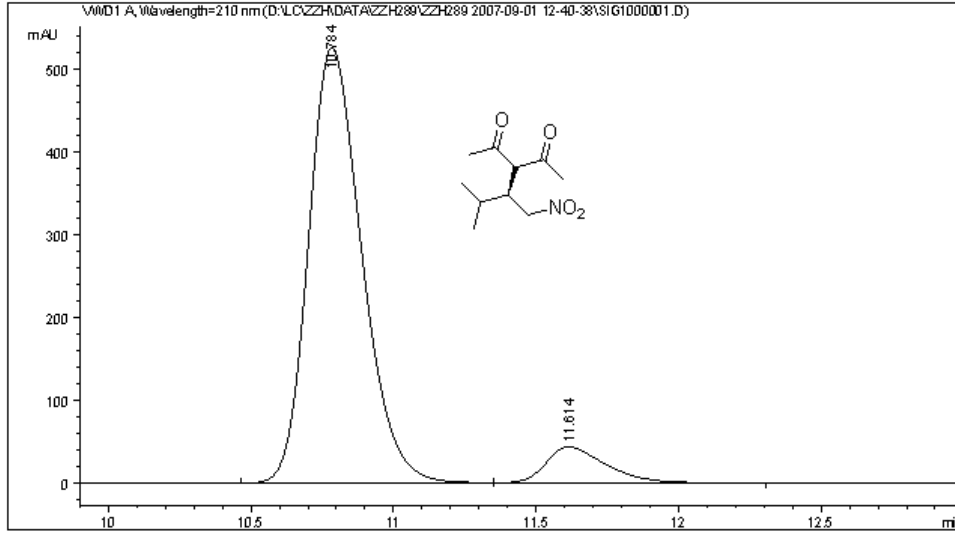
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 11.011 | VV | 0.2093 | 8841.07129 | 651.50189 | 90.5172 |
| 2 | 11.861 | VV | 0.2195 | 926.21155 | 64.09734 | 9.4828 |

Totals : 9767.28284 715.59923

=====
 *** End of Report ***

=====

| | | | |
|-----------------|--|------------|-----------|
| Acq. Operator | : ZZH | Seq. Line | : 1 |
| Acq. Instrument | : Instrument 1 | Location | : Vial 12 |
| Injection Date | : 9/1/2007 12:42:03 PM | Inj | : 1 |
| | | Inj Volume | : 5 µl |
| Acq. Method | : D:\LC\ZZH\data\ZZH289\ZZH289 2007-09-01 12-40-38\ADH-95-5-210NM-LML-100MIN.M | | |
| Last changed | : 7/30/2007 2:44:59 PM by ZZH | | |
| Analysis Method | : D:\LC\ZZH\DATA\ZZH289\ZZH289 2007-09-01 12-40-38\SIG1000001.D\DA.M (ADH-95-5-210NM-LML-100MIN.M) | | |
| Last changed | : 10/29/2007 8:03:05 PM by ZZH | | |
| | (modified after loading) | | |
| Method Info | : ADH-95-5-210NM-LML/MIN-100MIN | | |



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

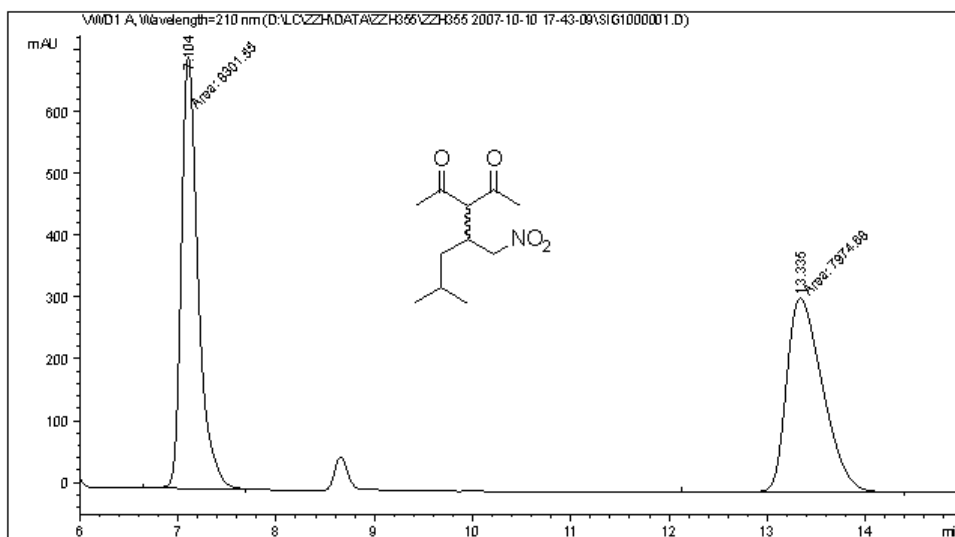
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 10.784 | BV | 0.2006 | 6813.55469 | 526.04761 | 91.3651 |
| 2 | 11.614 | VB | 0.2211 | 643.94690 | 43.77002 | 8.6349 |

Totals : 7457.50159 569.81762

=====
*** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 17-43-09\SIG1000001.D
 Sample Name: ZZH355B-R

```
=====
Acq. Operator   : ZZH                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 12
Injection Date  : 10/10/2007 5:45:02 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH355\ZZH355 2007-10-10 17-43-09\ASH-10-90-210MM-1ML-100MIN.M
Last changed    : 8/28/2007 4:06:26 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 17-43-09\SIG1000001.D\DA.M (ASH-10-90-
                210MM-1ML-100MIN.M)
Last changed    : 10/29/2007 8:19:42 PM by ZZH
                (modified after loading)
Method Info     : ASH-10-90-210MM-1ML-100MIN
=====
```



=====
 Area Percent Report
 =====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=210 nm

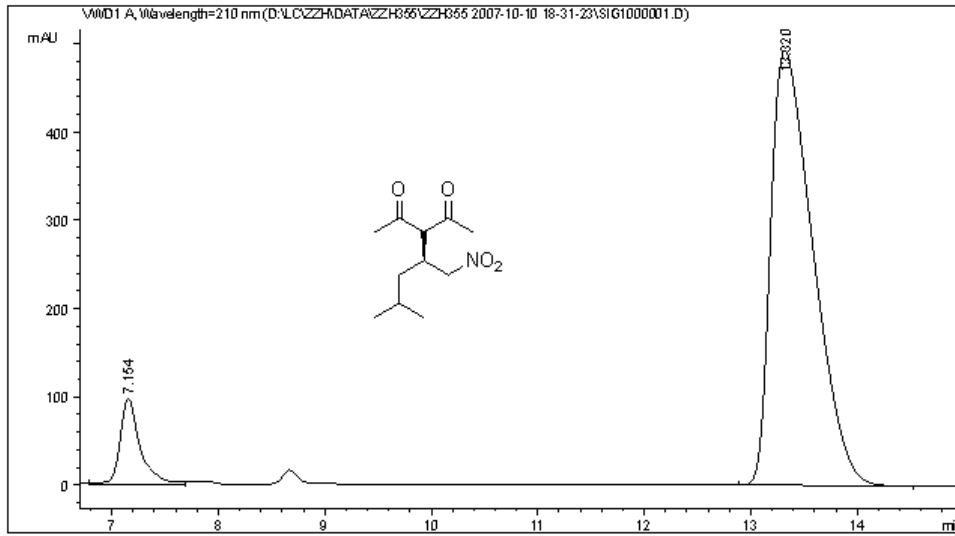
| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 7.104 | MM | 0.1980 | 8301.55371 | 698.69305 | 51.0042 |
| 2 | 13.335 | MM | 0.4247 | 7974.67578 | 312.94482 | 48.9958 |

```
Totals :                   1.62762e4  1011.63788
```

=====
 *** End of Report ***

Data File D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 18-31-23\SIG1000001.D
Sample Name: ZZH355

```
=====
Acq. Operator   : ZZH                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 13
Injection Date  : 10/10/2007 6:32:48 PM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH355\ZZH355 2007-10-10 18-31-23\ASH-10-90-210NM-1ML-100MIN.M
Last changed    : 8/28/2007 4:06:26 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH355\ZZH355 2007-10-10 18-31-23\SIG1000001.D\DA.M (ASH-10-90-
210NM-1ML-100MIN.M)
Last changed    : 10/29/2007 8:22:12 PM by ZZH
                : (modified after loading)
Method Info     : ASH-10-90-210NM-1ML-100MIN
=====
```



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

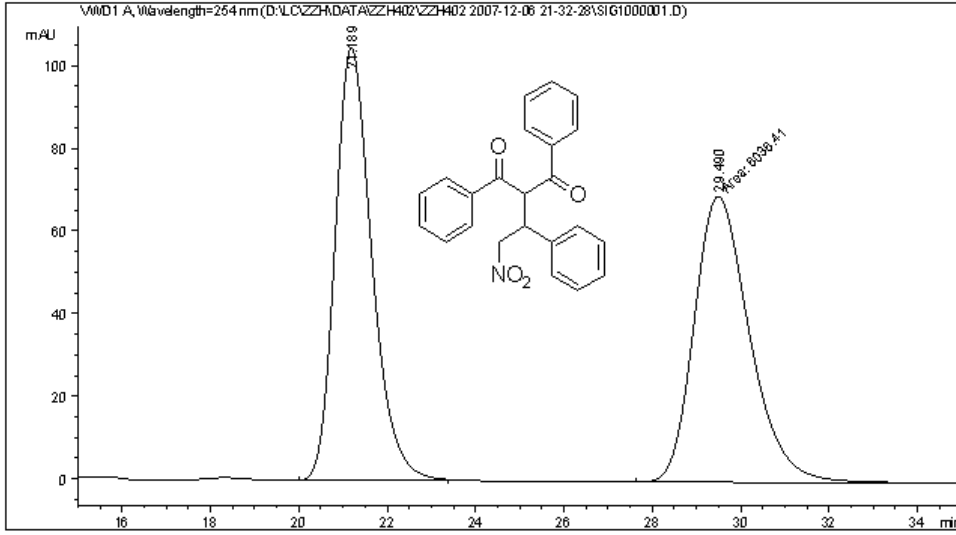
Signal 1: VWD1 A, Wavelength=210 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU | Height [mAU] | Area % |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1 | 7.154 | VV | 0.1986 | 1326.14685 | 98.04235 | 8.9244 |
| 2 | 13.320 | BB | 0.4254 | 1.35337e4 | 492.61053 | 91.0756 |

Totals : 1.48598e4 590.65289

=====
*** End of Report ***

=====
Acq. Operator : zzh Seq. Line : 1
Acq. Instrument : Instrument 1 Location : Vial 53
Injection Date : 12/6/2007 9:33:52 PM Inj : 1
 Inj Volume: 5 µl
Acq. Method : D:\LC\ZZH\data\ZZH402\ZZH402 2007-12-06 21-32-28\ASH-20-80-1ML-254NM-100MIN.M
Last changed : 9/10/2007 4:51:35 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH402\ZZH402 2007-12-06 21-32-28\SIG1000001.D\DA.M (ASH-20-80-
1ML-254NM-100MIN.M)
Last changed : 12/6/2007 10:10:54 PM by zzh
 (modified after loading)
Method Info : ASH-20-80-1ML-254NM-100MIN
=====



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 21.189 | BB | 0.8878 | 6037.31201 | 104.77172 | 50.0037 |
| 2 | 29.490 | MM | 1.4531 | 6036.41211 | 69.23789 | 49.9963 |

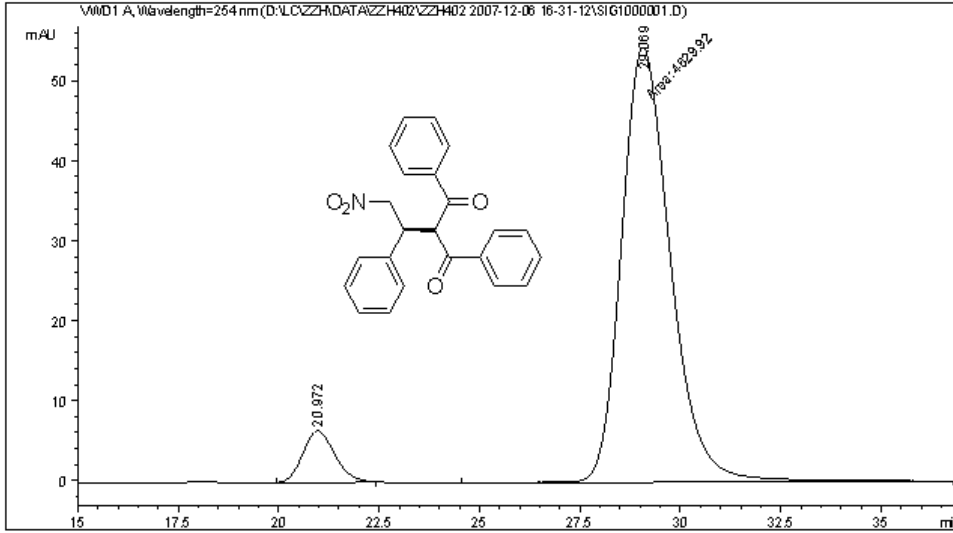
Totals : 1.20737e4 174.00961

=====
*** End of Report ***
=====

```

=====
Acq. Operator   : zzh                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 52
Injection Date  : 12/6/2007 4:32:35 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method    : D:\LC\ZZH\data\ZZH402\ZZH402 2007-12-06 16-31-12\ASH-20-80-1ML-254NM-100MIN.M
Last changed   : 9/10/2007 4:51:35 PM by ZZH
Analysis Method: D:\LC\ZZH\DATA\ZZH402\ZZH402 2007-12-06 16-31-12\SIG1000001.D\DA.M (ASH-20-80-1ML-254NM-100MIN.M)
Last changed   : 12/6/2007 5:15:34 PM by zzh
                (modified after loading)
Method Info    : ASH-20-80-1ML-254NM-100MIN
=====

```



=====
Area Percent Report
=====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: VWD1 A, Wavelength=254 nm

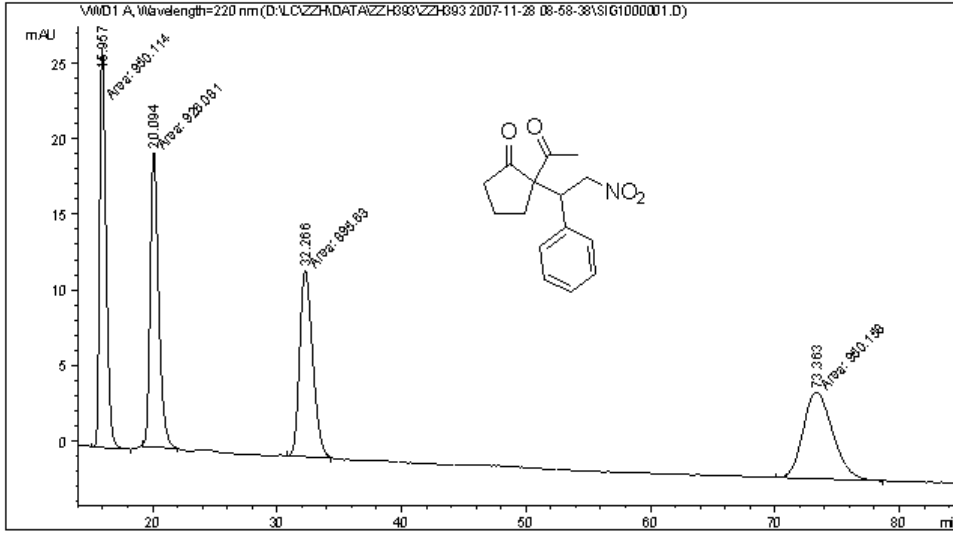
| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 20.972 | BB | 0.8447 | 359.93262 | 6.44947 | 7.2133 |
| 2 | 29.069 | MM | 1.4188 | 4629.91748 | 54.38589 | 92.7867 |

Totals : 4989.85010 60.83536

=====
*** End of Report ***

```

=====
Acq. Operator   : zzh                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 11
Injection Date  : 11/28/2007 9:00:00 AM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\ZZH\data\ZZH393\ZZH393 2007-11-28 08-58-38\ODH-20-80-1ML-220NM-100MIN.M
Last changed    : 8/10/2007 12:03:16 PM by ZZH
Analysis Method : D:\LC\ZZH\DATA\ZZH393\ZZH393 2007-11-28 08-58-38\SIG1000001.D\DA.M (ODH-20-80-1ML-220NM-100MIN.M)
Last changed    : 12/6/2007 5:21:25 PM by zzh
                                           (modified after loading)
Method Info     : ODH-20-80-1ML-220NM-100MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=220 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 15.957 | MM | 0.5977 | 950.11414 | 26.49490 | 25.5271 |
| 2 | 20.094 | MM | 0.7911 | 926.08112 | 19.50961 | 24.8814 |
| 3 | 32.266 | MM | 1.2123 | 895.62970 | 12.31298 | 24.0632 |
| 4 | 73.363 | MM | 2.7602 | 950.15771 | 5.73724 | 25.5283 |

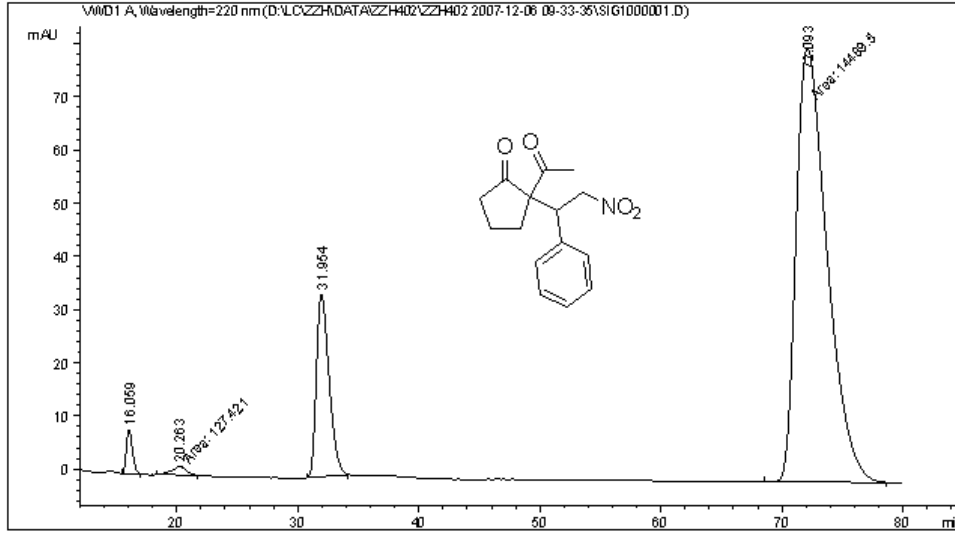
Totals : 3721.98267 64.05473

Data File D:\LC\ZZH\DATA\ZZH402\ZZH402 2007-12-06 09-33-35\SIG1000001.D
 Sample Name: zzh402b

```

=====
Acq. Operator   : zzh                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 46
Injection Date  : 12/6/2007 9:34:59 AM    Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method    : D:\LC\ZZH\data\ZZH402\ZZH402 2007-12-06 09-33-35\ODH-20-80-1ML-220NM-80MIN.M
Last changed   : 12/6/2007 9:15:55 AM by zzh
Analysis Method: D:\LC\ZZH\DATA\ZZH402\ZZH402 2007-12-06 09-33-35\SIG1000001.D\DA.M (ODH-20-80-1ML-220NM-80MIN.M)
Last changed   : 12/6/2007 5:24:03 PM by zzh
                                           (modified after loading)
Method Info    : ODH-20-80-1ML-220NM-80MIN
  
```



=====
 Area Percent Report
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=220 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area % |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1 | 16.059 | BB | 0.5415 | 292.12708 | 8.33351 | 1.6778 |
| 2 | 20.263 | MM | 1.3126 | 127.42136 | 1.61794 | 0.7318 |
| 3 | 31.954 | BB | 1.1169 | 2502.65552 | 34.33321 | 14.3734 |
| 4 | 72.093 | MM | 2.9546 | 1.44895e4 | 81.73413 | 83.2170 |

Totals : 1.74117e4 126.01879