Asymmetric Carbon–Carbon Bond Formation γ to a Carbonyl Group: Phosphine-Catalyzed Addition of Nitromethane to Allenes

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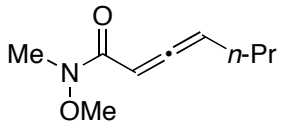
SUPPORTING INFORMATION

Table of Contents

I. Preparation of Allenes

The yields have not been optimized.

Synthesis of Allenes (representative procedure): A 300-mL flask was charged with a phosphorane (14.5 g, 40.0 mmol), evacuated, and back-filled with argon. CH_2Cl_2 (200 mL) and Et_3N (6.1 mL, 40 mmol) were added via syringe, and the solution was cooled to –78 °C. The acid chloride (40.0 mmol) was then added dropwise via syringe over five min. The solution was allowed to warm to room temperature over 3-4 hours, and then the reaction was quenched by the addition of silica gel. After removal of the solvent on a rotary evaporator, the product (adsorbed on silica) was loaded onto a pre-packed column of silica gel and purified via flash chromatography (hexanes/ethyl acetate), which furnished the allene as an oil.



(±)-*N*-Methoxy-*N*-methylhepta-2,3-dienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and pentanoyl chloride via the

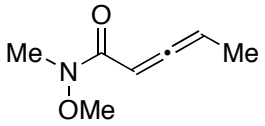
representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 40% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.15 (quintet, J = 2.9 Hz, 1H), 5.64 (q, J = 6.8 Hz, 1H), 3.71 (s, 3H), 3.23 (s, 3H), 2.14-2.09 (m, 2H), 1.49 (sextet, J = 7.4 Hz, 2H), 0.95 (t, J = 7.3 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.2, 166.1, 95.2, 86.3, 61.5, 32.6, 29.7, 22.2, 13.6.

IR (film) 3567, 3291, 3042, 2961, 2935, 2873, 2361, 2339, 1958, 1653, 1463, 1424, 1364 cm^{-1}.

LRMS (ES+) calcd for $C_9H_{16}NO_2$ (M+H$^+$) 170, found 170.



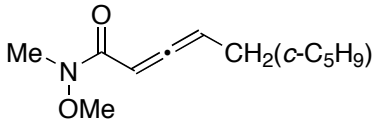
(±)-*N*-Methoxy-*N*-methylpenta-2,3-dienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and propionyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 33% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.20-6.10 (m, 1H), 5.61 (quintet, J = 7.3 Hz, 1H), 3.71 (s, 3H), 3.23 (s, 3H), 1.80-1.76 (m, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.9, 166.1, 90.3, 85.9, 61.8, 32.7, 13.1.

IR (film) 3567, 2974, 2936, 2361, 2339, 1960, 1653, 1457, 1421, 1358 cm^{-1}.

LRMS (ES+) calcd for $C_7H_{12}NO_2$ (M+H$^+$) 142, found 142.



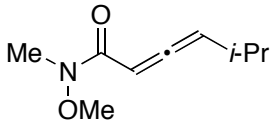
(±)-5-Cyclopentyl-*N*-methoxy-*N*-methylpenta-2,3-dienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and 3-cyclopentyl-propanoyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 41% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.16-6.12 (m, 1H), 5.63 (q, J = 7.3 Hz, 1H), 3.71 (s, 3H), 3.26 (s, 3H), 2.14 (dt, J = 7.2, 2.8 Hz, 2H), 1.95 (septet, J = 7.6 Hz, 1H), 1.83-1.75 (m, 2H), 1.64-1.47 (m, 4H), 1.22-1.14 (2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.6, 166.2, 94.8, 85.9, 61.8, 39.7, 34.2, 32.7, 32.4, 25.4.

IR (film) 3290, 2948, 2867, 2361, 2339, 1654, 1424, 1363 cm^{-1}.

LRMS (ES+) calcd for $C_{12}H_{20}NO_2$ (M+H$^+$) 210, found 210.



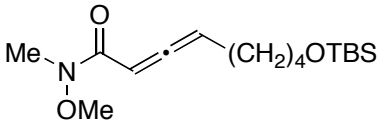
(±)-*N*-Methoxy-*N*,5-dimethylhexa-2,3-dienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and isovaleroyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 44% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.20 (q, *J* = 3.1 Hz, 1H), 5.66 (t, *J* = 6.1 Hz, 1H), 3.71 (s, 3H), 3.23 (s, 3H), 2.52-2.42 (m, 1H), 1.08 (d, *J* = 6.7 Hz, 6H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 211.1, 166.1, 102.6, 87.4, 61.8, 32.6, 27.7, 22.5, 22.3.

IR (film) 3291, 2963, 2937, 2871, 2361, 2339, 1957, 1653, 1465, 1384 cm^{-1}.

LRMS (ES+) calcd for $C_9H_{16}NO_2$ ($M+H^+$) 170, found 170.



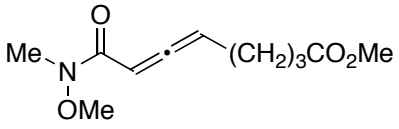
(±)-8-(*tert*-Butyldimethylsilyloxy)-*N*-methoxy-*N*-methylocta-2,3-dienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and 6-(*tert*-butyldimethylsilyloxy)hexanoyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 10% yield).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.09-6.06 (m, 1H), 5.57 (t, *J* = 6.7 Hz, 1H), 3.63 (s, 3H), 3.56-3.52 (m, 2H), 3.16 (s, 3H), 2.15-2.05 (m, 2H), 1.60-1.38 (m, 4H), 0.81 (s, 9H), –0.04 (s, 6H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.2, 166.1, 95.5, 86.5, 63.0, 61.8, 32.7, 32.3, 27.4, 26.1, 25.3, 18.5, –5.1.

IR (film) 3308, 2935, 2857, 2361, 2340, 1959, 1658, 1472 cm^{-1}.

LRMS (ES+) calcd for $C_{16}H_{32}NO_3Si$ ($M+H^+$) 314, found 314.



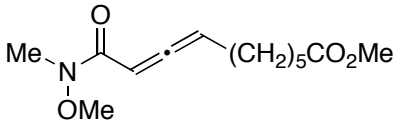
(±)-Methyl 8-(methoxy(methyl)amino)-8-oxoocta-5,6-dienoate. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and methyl adipoyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 25% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.18 (quintet, *J* = 2.9 Hz, 1H), 5.63 (q, *J* = 6.7 Hz, 1H), 3.71 (s, 3H), 3.66 (s, 3H), 3.23 (s, 3H), 2.39 (t, *J* = 7.4 Hz, 2H), 2.18 (qd, *J* = 7.1, 3.0 Hz, 2H), 1.80 (quintet, *J* = 7.4 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.3, 173.9, 165.9, 94.7, 86.9, 61.9, 51.7, 33.3, 32.8, 27.1, 24.1.

IR (film) 3282, 2951, 2361, 2339, 1959, 1734, 1653, 1639, 1457 cm^{-1}.

LRMS (ES+) calcd for $C_{11}H_{18}NO_4$ (M+H$^+$) 228, found 228.



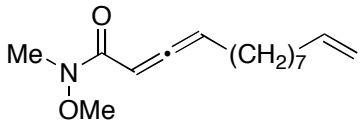
(±)-Methyl 10-(methoxy(methyl)amino)-10-oxodeca-7,8-dienoate. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and methyl 8-chloro-8-oxooctanoate chloride[1] via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 21% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.18-6.14 (m, 1H), 5.52 (q, *J* = 2.3 Hz, 1H), 3.61 (s, 3H), 3.55 (s, 3H), 3.13 (s, 3H), 2.20 (t, *J* = 7.4 Hz, 2H), 2.06-2.01 (m, 2H), 1.56-1.49 (m, 2H), 1.42-1.35 (m, 2H), 1.31-1.24 (m, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.2, 174.2, 166.0, 95.3, 86.5, 61.8, 51.5, 34.0, 32.7, 28.6, 28.5, 27.4, 24.8.

IR (film) 3288, 2937, 2859, 2361, 2338, 1958, 1734, 1653 cm^{-1}.

LRMS (ES+) calcd for $C_{13}H_{22}NO_4$ (M+H$^+$) 256, found 256.



(±)-*N*-Methoxy-*N*-methyltrideca-2,3,12-trienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and 10-undecenoyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 39% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.15 (quintet, *J* = 2.9 Hz, 1H), 5.80 (qt, *J* = 10.3, 6.7 Hz, 1H), 5.64 (q, *J* = 6.9 Hz, 1H), 4.98 (dq, *J* = 17.1, 1.6 Hz, 1H), 4.92 (dquintet, *J* = 10.2, 1.2 Hz, 1H), 3.71 (s, 3H), 3.24 (s, 3H), 2.13 (qd, *J* = 7.0, 3.0 Hz, 2H), 2.05-2.00 (m, 2H), 1.46 (quintet, *J* = 6.4 Hz, 2H), 1.40-1.27 (m, 8H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.3, 166.1, 139.4, 114.3, 95.7, 86.5, 61.9, 34.0, 29.4, 29.3, 29.23, 29.21, 29.12, 29.10, 27.8.

IR (film) 3075, 2927, 2855, 2361, 2338, 1959, 1653, 1464, 1423, 1362 cm^{-1}.

LRMS (ES+) calcd for $C_{15}H_{26}NO_2$ (M+H$^+$) 252, found 252.

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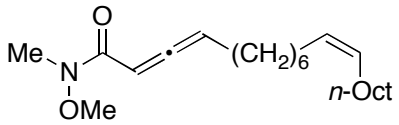
(1) Schinzer, D.; Limberg, A.; Böhm, O. M. *Chem. Eur. J.* **1996**, *2*, 1477–1482.



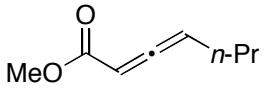
(±)-(*Z*)-*N*-Methoxy-*N*-methylnonadeca-2,3,10-trienamide. Prepared from *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide and oleoyl chloride via the representative procedure (purification by flash chromatography: 25% EtOAc in hexanes; 55% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.15 (quintet, J = 2.9 Hz, 1H), 5.64 (q, J = 6.9 Hz, 1H), 5.37-5.30 (m, 2H), 3.71 (s, 3H), 3.24 (s, 3H), 2.16-2.11 (m, 2H), 2.02-1.99 (m, 4H), 1.50 (m, 2H), 1.37-1.22 (m, 18H), 0.88 (t, J = 6.9 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.3, 166.2, 130.1, 129.9, 95.6, 86.4, 61.8, 32.7, 32.1, 29.9, 29.8, 29.7, 29.53, 29.52, 29.2, 29.1, 29.0, 27.8, 27.4, 27.3, 22.9, 14.3.

IR (film) 3300, 3003, 2923, 2853, 2361, 2338, 1959, 1653, 1457, 1420, 1362 cm^{-1}.

LRMS (ES+) calcd for $C_{22}H_{40}NO_2$ ($M+H^+$) 350, found 350.



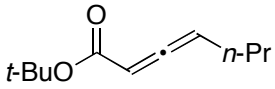
(±)-Methyl hepta-2,3-dienoate [111425-91-5]. Prepared from methyl (triphenylphosphoranylidene)acetate and pentanoyl chloride via the representative procedure (purification by distillation; 71% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.59-5.52 (m, 2H), 3.68 (s, 3H), 2.10-2.05 (m, 2H), 1.48-1.41 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.6, 166.9, 95.4, 88.0, 52.1, 29.7, 22.1, 13.6.

IR (film) 2960, 2935, 2875, 2361, 2337, 1961, 1723, 1437, 1262 cm^{-1}.

LRMS (ES+) calcd for $C_8H_{13}O_2$ ($M+H^+$) 141, found 141.



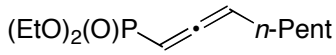
(±)-*tert*-Butyl hepta-2,3-dienoate [151860-31-0]. Prepared from (*tert*-butoxycarbonyl-methylene)triphenylphosphorane and pentanoyl chloride via the representative procedure (purification by flash chromatography: 5% EtOAc in hexanes; 38% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.55 (q, J = 6.9 Hz, 1H), 5.47 (q, J = 2.6 Hz, 1H), 2.09-2.02 (m, 2H), 1.52-1.42 (m, 11H), 0.95 (t, J = 7.3 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 212.1, 165.8, 95.0, 89.9, 80.8, 29.8, 28.3, 22.2, 13.7.

IR (film) 3004, 2967, 2934, 2875, 2361, 2338, 1960, 1717, 1368, 1147 cm^{-1}.

LRMS (ES+) calcd for $C_{11}H_{19}O_2$ ($M+H^+$) 183, found 183.



(±)-Diethyl octa-1,2-dienylphosphonate [344554-28-5]. Prepared according to a literature procedure (purification by flash chromatography: 20 → 100% EtOAc in hexanes; 75% yield).[2]

^1H NMR ($CDCl_3$, 500 MHz) δ 5.43 (sextet, J = 7.0 Hz, 1H), 5.29 (sextet, J = 3.4 Hz, 1H), 4.14-4.07 (m, 4H), 2.09 (quintet of doublets, J = 7.2, 3.4 Hz, 2H), 1.46-1.41 (m, 2H), 1.35-1.29 (m, 10H), 1.33 (t, J = 7.0 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 211.9, 92.4, 80.8, 79.2, 62.2, 31.2, 28.67, 28.64, 27.4, 22.5, 16.4, 14.1.

IR (film) 3482, 2980, 2958, 2931, 2872, 2859, 2360, 2338, 1955, 1258 cm^{-1}.

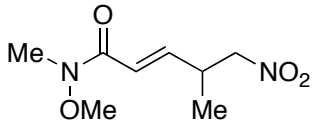
LRMS (ES+) calcd for $C_{12}H_{24}O_3P$ ($M+H^+$) 247, found 247.

(2) Altenbach, H.-J.; Korff, R. *Tetrahedron Lett.* **1981**, *22*, 5175–5178.

II. Phosphine-Catalyzed Asymmetric γ Additions

General Procedure. In a glovebox, catalyst (*S*)-**1** (29 mg, 0.075 mmol; 0.10 equiv) and phenol (7.0 mg, 0.075 mmol; 0.10 equiv) were added to an oven-dried 20-mL vial. These solids were dissolved in anhydrous dioxane (15 mL), and then nitromethane (225 μL, 4.15 mmol; 5.5 equiv) and the allene (0.75 mmol; 1.0 equiv) were added via syringe. The vial was capped and removed from the glovebox, and the reaction mixture was stirred at room temperature for 15 h. The solvent was then evaporated, and the product was purified by flash chromatography.

Glovebox-free Procedure (Table 2, entry 2). On a benchtop, catalyst (*S*)-**1** (43.5 mg, 0.113 mmol; 0.15 equiv; with 10% (*S*)-**1**, a small amount of unreacted allene was observed after 15 h) and phenol (10.5 mg, 0.113 mmol; 0.15 equiv) were added to an oven-dried 20-mL vial. The vial was capped with a septum, and then it was evacuated and refilled with argon (three cycles). Next, anhydrous dioxane (15 mL), nitromethane (225 μL, 4.15 mmol; 5.5 equiv), and (±)-*N*-methoxy-*N*-methylhepta-2,3-dienamide (127 mg, 0.75 mmol; 1.0 equiv) were added in order via syringe through the septum. The reaction mixture was stirred at room temperature for 15 h. It was then concentrated and purified by flash chromatography (25%EtOAc in pentane), which afforded the desired product as a colorless oil (140 mg, 81% yield) with 93% ee.



(*E*)-*N*-Methoxy-*N*-4-dimethyl-5-nitropent-2-enamide (Table 2, entry 1). The compound was prepared according to the general procedure with (±)-*N*-methoxy-*N*-methylpenta-2,3-dienamide (106 mg, 0.75 mmol). After purification by flash chromatography (30% EtOAc in hexanes), the title compound was isolated as a colorless oil (144 mg, 95% yield) with 97% ee.

$[\alpha]_D^{22} = -45$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 38.7 min (minor), 44.5 min (major).

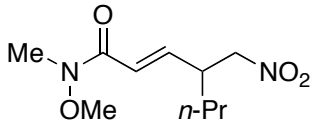
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (140 mg, 93% yield) with 97% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.73 (dd, *J* = 15.4, 7.8 Hz, 1H), 6.45 (d, *J* = 15.4 Hz, 1H), 4.37 (dd, *J* = 12.2, 7.7 Hz, 1H), 4.31 (dd, *J* = 12.2, 7.0 Hz, 1H), 3.63 (s, 3H), 3.18 (s, 3H), 3.24-3.15 (m, 1H), 1.15 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.9, 145.2, 120.6, 79.9, 62.0, 35.8, 32.4, 17.0.

IR (film) 3287, 2972, 2361, 2339, 1669, 1558 cm^{-1}.

LRMS (ES+) calcd for $C_8H_{15}N_2O_4$ ($M+H^+$) 203, found 203.



(*E*)-*N*-Methoxy-*N*-methyl-4-(nitromethyl)hept-2-enamide (Table 2, entry 2). The compound was prepared according to the general procedure with (±)-*N*-methoxy-*N*-methylhepta-2,3-dienamide (127 mg, 0.75 mmol). After purification by flash chromatography (30% EtOAc in hexanes), the title compound was isolated as a colorless oil (137 mg, 80% yield) with 93% ee.

$[\alpha]_D^{22} = -30$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 24.5 min (major), 28.7 min (minor).

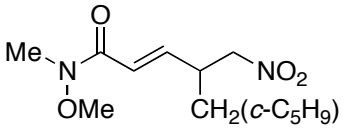
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (141 mg, 82% yield) with 93% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.63 (dd, *J* = 15.4, 9.1 Hz, 1H), 6.44 (d, *J* = 15.4 Hz, 1H), 4.38 (dd, *J* = 12.3, 5.9 Hz, 1H), 4.30 (dd, *J* = 12.2, 8.9 Hz, 1H), 3.61 (s, 3H), 3.17 (s, 3H), 3.09-3.01 (m, 1H), 1.46-1.17 (m, 4H), 0.85 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.7, 144.2, 122.0, 79.1, 62.0, 41.3, 33.6, 32.4, 20.0, 13.9.

IR (film) 2961, 2935, 2874, 2361, 2338, 1668, 1635, 1558, 1379 cm^{-1}.

LRMS (ES+) calcd for $C_{10}H_{19}N_2O_4$ ($M+H^+$) 231, found 231.



(*E*)-5-Cyclopentyl-*N*-methoxy-*N*-methyl-4-(nitromethyl)pent-2-enamide (Table 2, entry 3). The compound was prepared according to the general procedure with (±)-5-cyclopentyl-*N*-methoxy-*N*-methylpenta-2,3-dienamide (157 mg, 0.75 mmol). After purification by flash chromatography (30% EtOAc in hexanes), the title compound was isolated as a colorless oil (146 mg, 72% yield) with 87% ee.

$[\alpha]_D^{22} = -3.5$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 3.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 33.7 min (major), 46.2 min (minor).

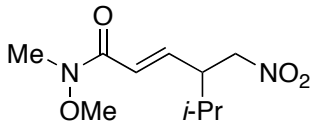
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (152 mg, 75% yield) with 86% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.65 (dd, *J* = 15.4, 9.4 Hz, 1H), 6.47 (d, *J* = 15.4 Hz, 1H), 4.38 (dd, *J* = 12.3, 5.8 Hz, 1H), 4.30 (dd, *J* = 12.2, 9.0 Hz, 1H), 3.63 (s, 3H), 3.18 (s, 3H), 3.13-3.04 (m, 1H), 1.75-1.70 (m, 3H), 1.58-1.36 (m, 6H), 1.05-1.00 (m, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.8, 144.4, 122.0, 79.4, 62.0, 41.0, 38.0, 37.4, 33.3, 32.1, 25.2.

IR (film) 2941, 2867, 2361, 2339, 1669, 1653, 1635, 1558 cm^{-1}.

LRMS (ES+) calcd for $C_{13}H_{23}N_2O_4$ ($M+H^+$) 271, found 271.



(*E*)-*N*-Methoxy-*N*,5-dimethyl-4-(nitromethyl)hex-2-enamide (Table 2, entry 4). The compound was prepared according to the general procedure (except 15% catalyst was used) with (±)-*N*-methoxy-*N*,5-dimethylhexa-2,3-dienamide (127 mg, 0.75 mmol). After purification by flash chromatography (30% EtOAc in hexanes), the title compound was isolated as a colorless oil (102 mg, 60% yield) with 81% ee.

$[\alpha]_D^{22} = -30$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 19.7 min (major), 24.0 min (minor).

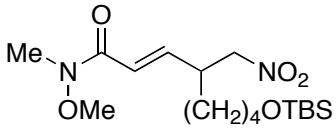
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (108 mg, 63% yield) with 81% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.72 (dd, *J* = 15.4, 9.5 Hz, 1H), 6.44 (d, *J* = 15.4 Hz, 1H), 4.49 (dd, *J* = 12.2, 5.1 Hz, 1H), 4.35 (dd, *J* = 12.1, 9.8 Hz, 1H), 3.63 (s, 3H), 3.19 (s, 3H), 2.91 (septet, *J* = 5.4 Hz, 1H), 1.80 (sextet, *J* = 6.7 Hz, 1H), 0.92 (dd, *J* = 13.0, 6.7 Hz, 6H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.6, 142.5, 122.8, 77.9, 62.0, 47.6, 32.4, 30.0, 20.5, 19.2.

IR (film) 2965, 2876, 2361, 2338, 1668, 1653, 1635, 1558, 1472, 1457 cm^{-1}.

LRMS (ES+) calcd for $C_{10}H_{19}N_2O_4$ ($M+H^+$) 231, found 231.



(*E*)-8-(*tert*-Butyldimethylsilyloxy)-*N*-methoxy-*N*-methyl-4-(nitromethyl)oct-2-enamide (Table 2, entry 5). The compound was prepared according to the general procedure with (±)-8-(*tert*-butyldimethylsilyloxy)-*N*-methoxy-*N*-methylocta-2,3-dienamide (235 mg, 0.75 mmol). After purification by flash chromatography (25% EtOAc in hexanes), the title compound was isolated as a colorless oil (156 mg, 56% yield) with 92% ee.

$[\alpha]_D^{22} = -19$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 16.6 min (major), 18.4 min (minor).

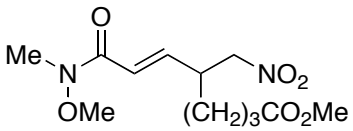
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (163 mg, 58% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.65 (dd, *J* = 15.4, 9.1 Hz, 1H), 6.53 (d, *J* = 15.4 Hz, 1H), 4.39 (dd, *J* = 12.3, 6.0 Hz, 1H), 4.32 (dd, *J* = 12.3, 9.0 Hz, 1H), 3.63 (s, 3H), 3.57-3.50 (m, 2H), 3.18 (s, 3H), 3.09-3.02 (m, 1H), 1.54-1.24 (m, 6H), 0.83 (s, 9H), –0.01 (s, 6H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.6, 144.1, 122.1, 79.0, 62.9, 62.0, 41.6, 32.6, 31.4, 26.1, 23.3, 18.5, –5.1.

IR (film) 2933, 2858, 2361, 2339, 1668, 1653, 1635, 1557, 1380 cm^{-1}.

LRMS (ES+) calcd for $C_{17}H_{35}N_2O_5Si$ ($M+H^+$) 375, found 375.



(*E*)-Methyl 8-(methoxy(methyl)amino)-5-(nitromethyl)-8-oxooct-6-enoate (Table 2, entry 6). The compound was prepared according to the general procedure with (±)-methyl 8-(methoxy(methyl)amino)-8-oxoocta-5,6-dienoate (170 mg, 0.75 mmol). After purification by flash chromatography (15 → 50% EtOAc in hexanes), the title compound was isolated as a colorless oil (165 mg, 76% yield) with 94% ee.

$[\alpha]_D^{22}$ = –30 (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 59.9 min (major), 74.3 min (minor).

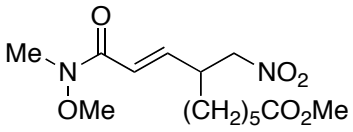
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (158 mg, 73% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.61 (dd, *J* = 15.4, 9.1 Hz, 1H), 6.47 (d, *J* = 15.5 Hz, 1H), 4.39 (dd, *J* = 12.4, 6.0 Hz, 1H), 4.31 (dd, *J* = 12.3, 8.8 Hz, 1H), 3.61 (s, 3H), 3.58 (s, 3H), 3.16 (s, 3H), 3.08-3.00 (m, 1H), 2.25 (t, *J* = 6.9 Hz, 2H), 1.64-1.41 (m, 4H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 173.5, 165.6, 143.5, 122.5, 78.8, 62.0, 51.8, 41.3, 33.6, 32.4, 30.8, 22.2.

IR (film) 2952, 2871, 2361, 2338, 1734, 1664, 1635, 1557 cm^{-1}.

LRMS (ES+) calcd for $C_{12}H_{21}N_2O_6$ ($M+H^+$) 289, found 289.



(*E*)-Methyl 10-(methoxy(methyl)amino)-7-(nitromethyl)-10-oxodec-8-enoate (Table 2, entry 7). The compound was prepared according to the general procedure with (±)-methyl 10-(methoxy(methyl)amino)-10-oxodeca-7,8-dienoate (191 mg, 0.75 mmol). After purification by flash chromatography (20 → 50% EtOAc in hexanes), the title compound was isolated as a colorless oil (195 mg, 82% yield) with 92% ee.

$[\alpha]_D^{22} = -26$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 56.7 min (major), 63.5 min (minor).

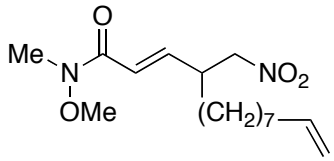
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (193 mg, 82% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.58 (dd, *J* = 15.4, 9.1 Hz, 1H), 6.40 (d, *J* = 15.4 Hz, 1H), 4.35 (dd, *J* = 12.3, 5.9 Hz, 1H), 4.28 (dd, *J* = 12.3, 8.7 Hz, 1H), 3.58 (s, 3H), 3.55 (s, 3H), 3.13 (s, 3H), 3.02-2.94 (m, 1H), 2.19 (t, *J* = 7.4 Hz, 3H), 1.52-1.16 (m, 7H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 174.0, 165.7, 144.0, 122.1, 79.0, 61.9, 51.6, 41.4, 33.9, 32.3, 31.3, 28.9, 26.5, 24.7.

IR (film) 2938, 2861, 2361, 2339, 1734, 1558 cm^{-1}.

LRMS (ES+) calcd for $C_{14}H_{25}N_2O_6$ ($M+H^+$) 317, found 317.



(*E*)-*N*-Methoxy-*N*-methyl-4-(nitromethyl)trideca-2,12-dienamide (Table 2, entry 8). The compound was prepared according to the general procedure with (±)-*N*-methoxy-*N*-methyltrideca-2,3,12-trienamide (189 mg, 0.75 mmol). After purification by flash chromatography (5 → 40% EtOAc in hexanes), the title compound was isolated as a colorless oil (199 mg, 85% yield) with 92% ee.

$[\alpha]_D^{22} = -26$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK AD-H column; 5.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 14.7 min (major), 18.0 min (minor).

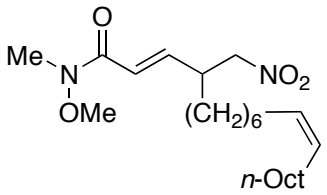
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (190 mg, 81% yield) with 92% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.62 (dd, *J* = 15.4, 9.1 Hz, 1H), 6.43 (d, *J* = 15.4 Hz, 1H), 5.71 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 4.93-4.83 (m, 2H), 4.37 (dd, *J* = 12.3, 6.0 Hz, 1H), 4.29 (dd, *J* = 12.1, 8.8 Hz, 1H), 3.60 (s, 3H), 3.16 (s, 3H), 3.06-2.98 (m, 1H), 1.97-1.93 (m, 2H), 1.48-1.14 (m, 12H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.7, 144.3, 139.2, 122.0, 114.4, 79.1, 61.9, 60.5, 41.5, 33.9, 31.6, 29.4, 29.1, 29.0, 26.8, 14.3.

IR (film) 3289, 3075, 2925, 2855, 2361, 2339, 1653 cm^{-1}.

LRMS (ES+) calcd for $C_{16}H_{29}N_2O_4$ ($M+H^+$) 313, found 313.



(2*E*,11*Z*)-*N*-Methoxy-*N*-methyl-4-(nitromethyl)icosa-2,11-dienamide (Table 2, entry 9). The compound was prepared according to the general procedure with (±)-(*Z*)-*N*-methoxy-*N*-methylnonadeca-2,3,10-trienamide (262 mg, 0.75 mmol). After purification by flash chromatography (7 → 14% EtOAc in hexanes), the title compound was isolated as a colorless oil (257 mg, 83% yield) with 93% ee.

$[\alpha]_D^{22} = -24$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 1.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 57.4 min (major), 64.0 min (minor).

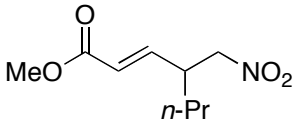
The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (260 mg, 84% yield) with 93% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.64 (dd, *J* = 15.4, 9.4 Hz, 1H), 6.47 (dd, *J* = 15.4 Hz, 1H), 5.31-5.20 (m, 2H), 4.38 (dd, *J* = 12.3, 6.0 Hz, 1H), 4.30 (dd, *J* = 12.1, 8.9 Hz, 1H), 3.61 (s, 3H), 3.17 (s, 3H), 3.08-2.98 (m, 1H), 2.00-1.88 (m, 4H), 1.48-1.10 (m, 22H), 0.81 (t, *J* = 6.5 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.8, 144.3, 130.2, 129.7, 122.0, 79.1, 61.9, 41.5, 36.8, 32.4, 32.0, 31.6, 29.9, 29.8, 29.7, 29.5, 29.4, 29.2, 27.4, 27.3, 26.9, 22.9, 14.3.

IR (film) 3003, 2926, 2855, 2361, 2339, 1667, 1635, 1557, 1464 cm^{-1}.

LRMS (ES+) calcd for $C_{23}H_{43}N_2O_4$ ($M+H^+$) 411, found 411.



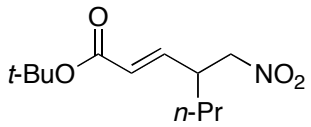
(*E*)-Methyl 4-(nitromethyl)hept-2-enoate (Table 3, entry 1). The compound was prepared according to the general procedure with (±)-methyl hepta-2,3-dienoate (105 mg, 0.75 mmol). After purification by flash chromatography (30% hexanes in CH_2Cl_2), the title compound was isolated as a colorless oil (109 mg, 72% yield) with 92% ee.

$[\alpha]_D^{22} = -32$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 1.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 24.8 min (minor), 32.7 min (major).

The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (113 mg, 74% yield) with 93% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.72 (dd, *J* = 15.6, 9.1 Hz, 1H), 5.90 (d, *J* = 15.7 Hz, 1H), 4.42 (dd, *J* = 12.3, 5.9 Hz, 1H), 3.85 (dd, *J* = 12.3, 8.7 Hz, 1H), 3.73 (s, 3H), 3.10-3.03 (m, 1H), 1.50-1.25 (m, 4H), 0.91 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 166.3, 146.1, 124.2, 78.9, 52.0, 41.0, 33.5, 20.0, 13.9.
IR (film) 2960, 2935, 2875, 2361, 2339, 1717, 1661, 1558, 1436 cm^{-1}.
LRMS (ES+) calcd for $C_9H_{16}NO_4$ ($M+H^+$) 202, found 202.



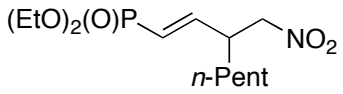
(*E*)-*tert*-Butyl 4-(nitromethyl)hept-2-enoate (Table 3, entry 2). The compound was prepared according to the general procedure with (±)-*tert*-butyl hepta-2,3-dienoate (137 mg, 0.75 mmol). After purification by flash chromatography (10% EtOAc in hexanes), the title compound was isolated as a colorless oil (173 mg, 95% yield) with 90% ee.

$[\alpha]_D^{22} = -29$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 1.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 13.6 min (minor), 17.4 min (major).

The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (168 mg, 93% yield) with 90% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.56 (dd, *J* = 15.6, 9.0 Hz, 1H), 5.77 (d, *J* = 15.6 Hz, 1H), 4.37 (dd, *J* = 12.3, 6.1 Hz, 1H), 4.31 (dd, *J* = 12.3, 8.5 Hz, 1H), 3.04-2.96 (m, 1H), 1.42-1.20 (m, 13H), 0.86 (t, *J* = 6.6 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 165.1, 144.6, 126.1, 80.9, 78.9, 40.8, 33.5, 28.2, 20.0, 13.9.
IR (film) 2964, 2934, 2875, 2361, 2339, 1713, 1654, 1554, 1368, 1159 cm^{-1}.
LRMS (ES+) calcd for $C_8H_{12}NO_4$ (M-*t*-Bu^+) 186, found 186.



(*E*)-Diethyl 3-(nitromethyl)oct-1-enylphosphonate (Table 3, entry 3). The compound was prepared according to the general procedure (except 3.0 equiv of phenol was used and the reaction mixture was heated at 60 °C) with (±)-diethyl octa-1,2-dienylphosphonate (185 mg, 0.75 mmol). After purification by flash chromatography (70% EtOAc in hexanes), the title compound was isolated as a colorless oil (203 mg, 89% yield) with 75% ee.

$[\alpha]_D^{22} = -18$ (c = 1.0, $CHCl_3$). HPLC analysis of the product: Daicel CHIRALPAK OD-H column; 2.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 41.4 min (minor), 46.7 min (major).

The second run was performed with (*R*)-**1**. The product was isolated as a colorless oil (193 mg, 84% yield) with 72% ee.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.45 (ddd, *J* = 21.6, 17.1, 8.7 Hz, 1H), 5.64 (dd, *J* = 18.9, 17.1 Hz, 1H), 4.35 (dd, *J* = 12.2, 5.6 Hz, 1H), 4.26 (dd, *J* = 12.2, 9.0 Hz, 1H), 4.01-3.87 (m, 4H), 2.96-2.89 (m, 1H), 1.42-1.30 (m, 2H), 1.28-1.11 (m, 12H), 0.77 (t, *J* = 6.6 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125 MHz) δ 150.6, 121.7, 78.8, 62.0, 43.2, 43.0, 31.5, 31.1, 26.4, 22.5, 16.5, 16.4, 14.0.

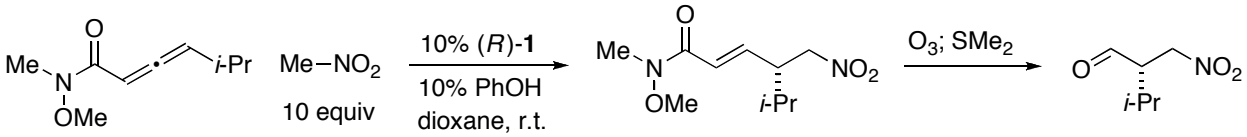
IR (film) 2958, 2932, 2860, 2361, 2339, 1639, 1553, 1380, 1246 cm^{-1}.

LRMS (ES+) calcd for $C_{13}H_{27}NO_5P$ ($M+H^+$) 308, found 308.

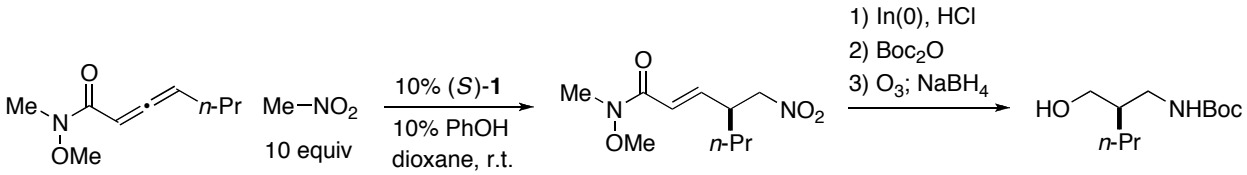
III. Determination of Absolute Stereochemistry

The stereochemistry of two of the γ-addition products was assigned by correlation with known compounds. The stereochemistry of the other products was assigned by analogy.

(*S*)-3-methyl-2-(nitromethyl)butanal:[3]



(*R*)-*tert*-butyl 2-(hydroxymethyl)pentylcarbamate:[4]



(3) Enders, D.; Syrig, R.; Raabe, G.; Fernández, R.; Gasch, C.; Lassaletta, J.-M.; Llera, J.-M. *Synthesis* **1996**, 48–52.

(4) Chi, Y.; Gellman, S. H. *J. Am. Chem. Soc.* **2006**, *128*, 6804–6805.

STANDARD PROTON PARAMETERS

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ACQUISITION		dmf	10000
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tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
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tof	1519.5	wexp	
nt	16	wbs	
ct	8	wnt	wft
alock	n		
gain	not used		
FLAGS			
fl	n		
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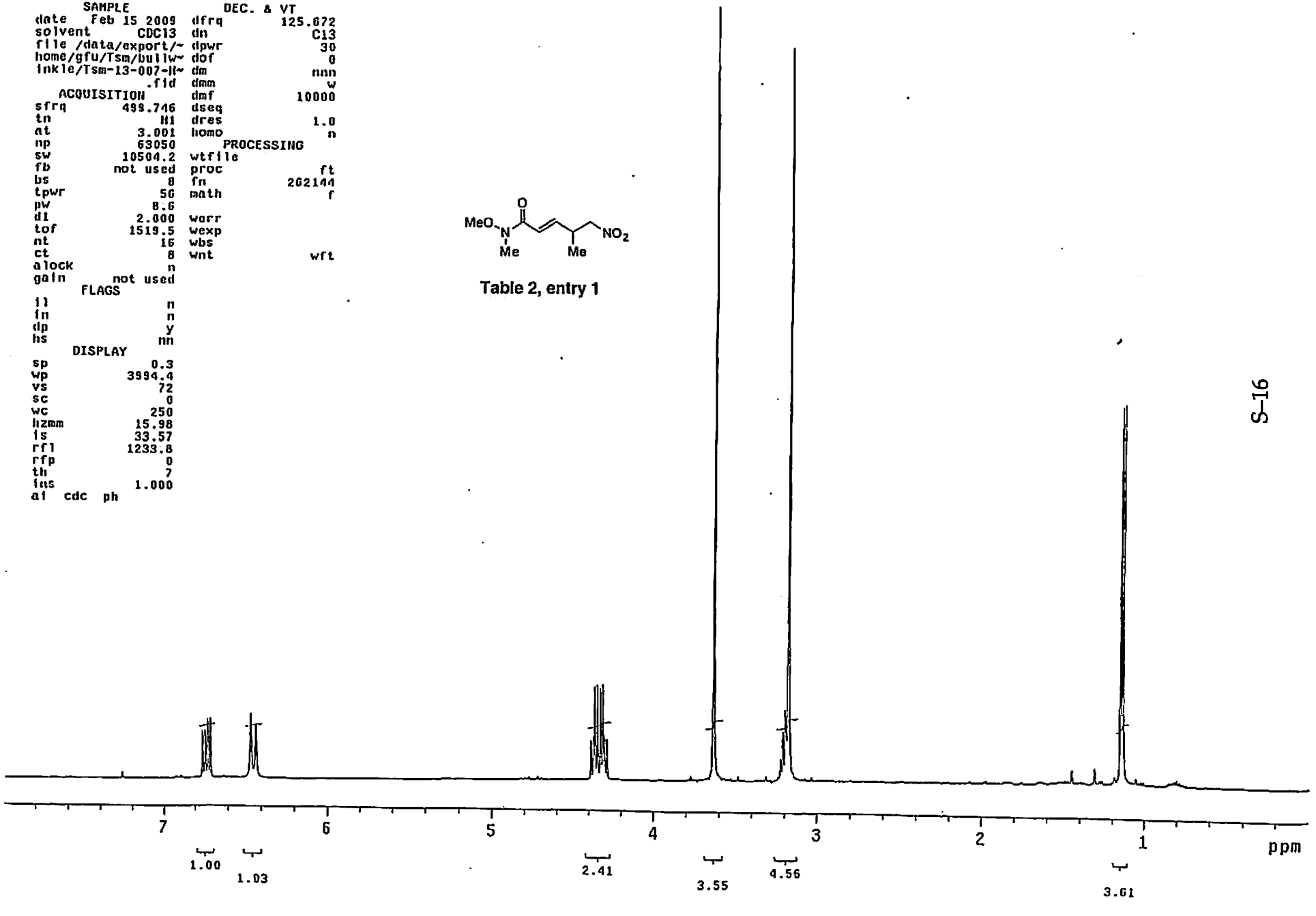
al cdc ph

Table 2, entry 1



STANDARD PROTON PARAMETERS

expl s2pul

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inkle/Tsm-13-005-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
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tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
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dl	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	8	wnt	wft
alock	n		
gain	not used		
FLAGS			
ll	n		
in	n		
dp	y		
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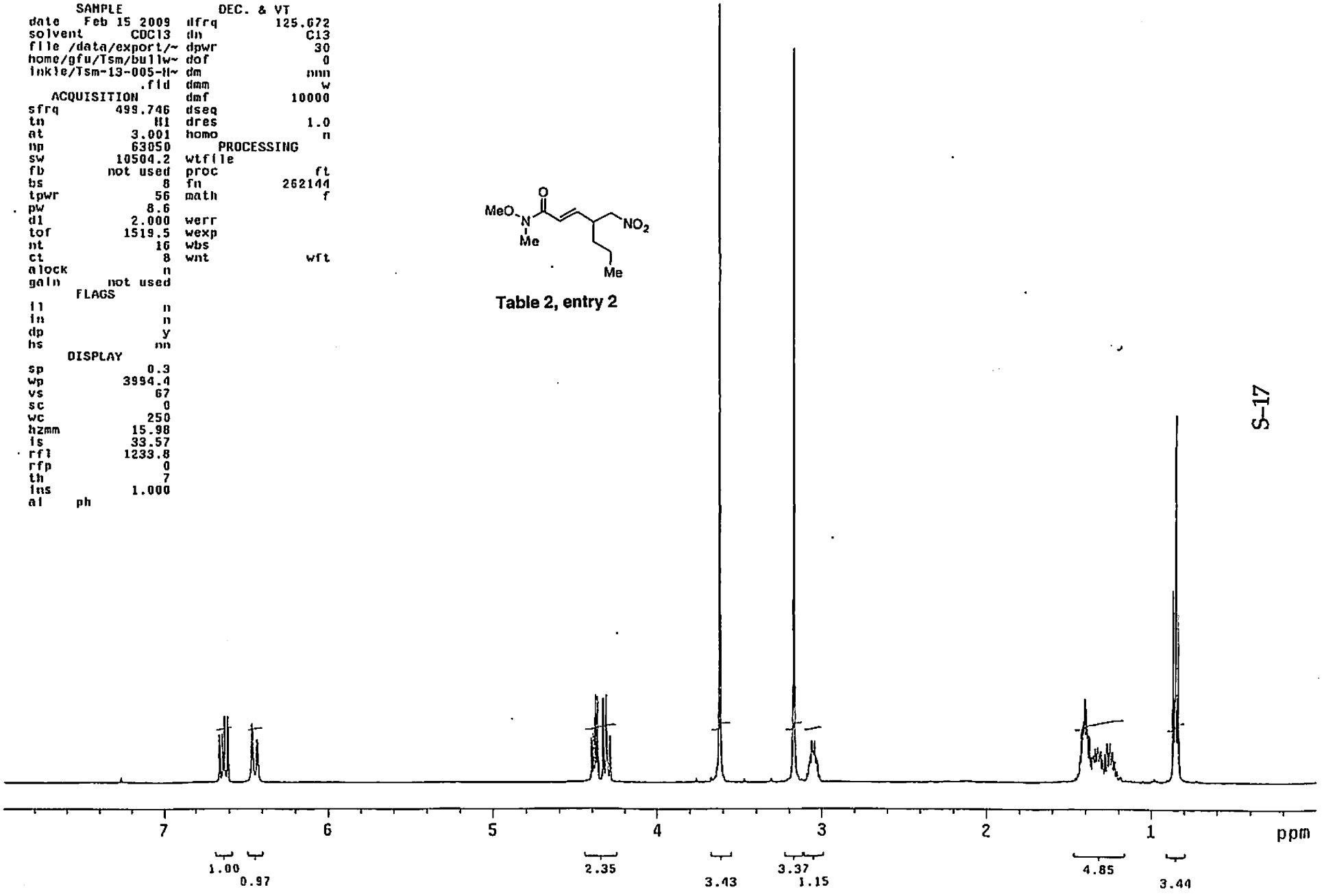
rfp	0		
th	7		
ins	1.000		
al	ph		

Table 2, entry 2



STANDARD PROTON PARAMETERS

expl s2pul

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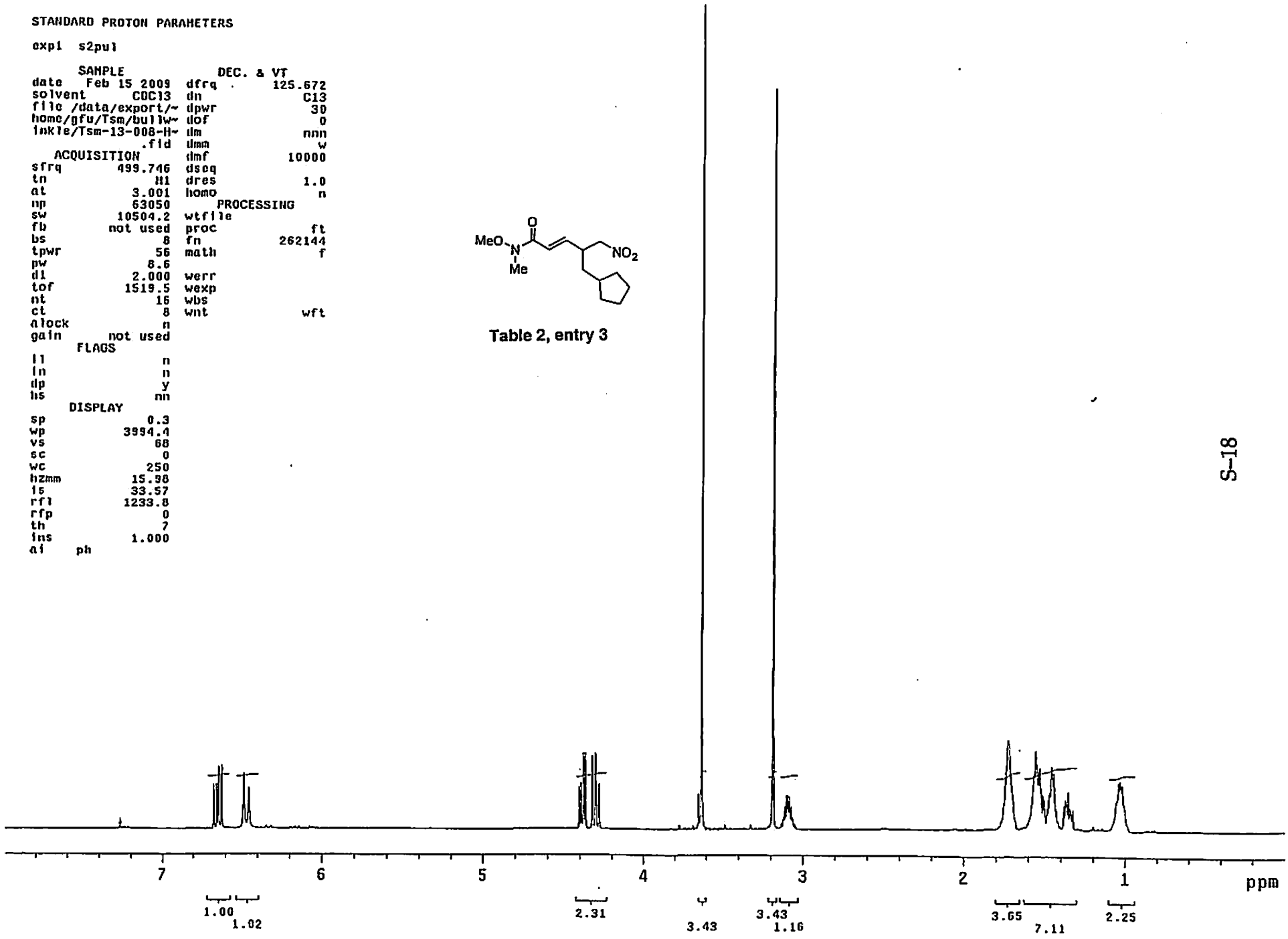
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-008-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
dl	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	8	wnt	wft
alock	n		
gain	not used		
FLAGS			
ll	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	0.3		
wp	3994.4		
vs	68		
sc	0		
wc	250		
hzmm	15.98		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
ai	ph		

Table 2, entry 3

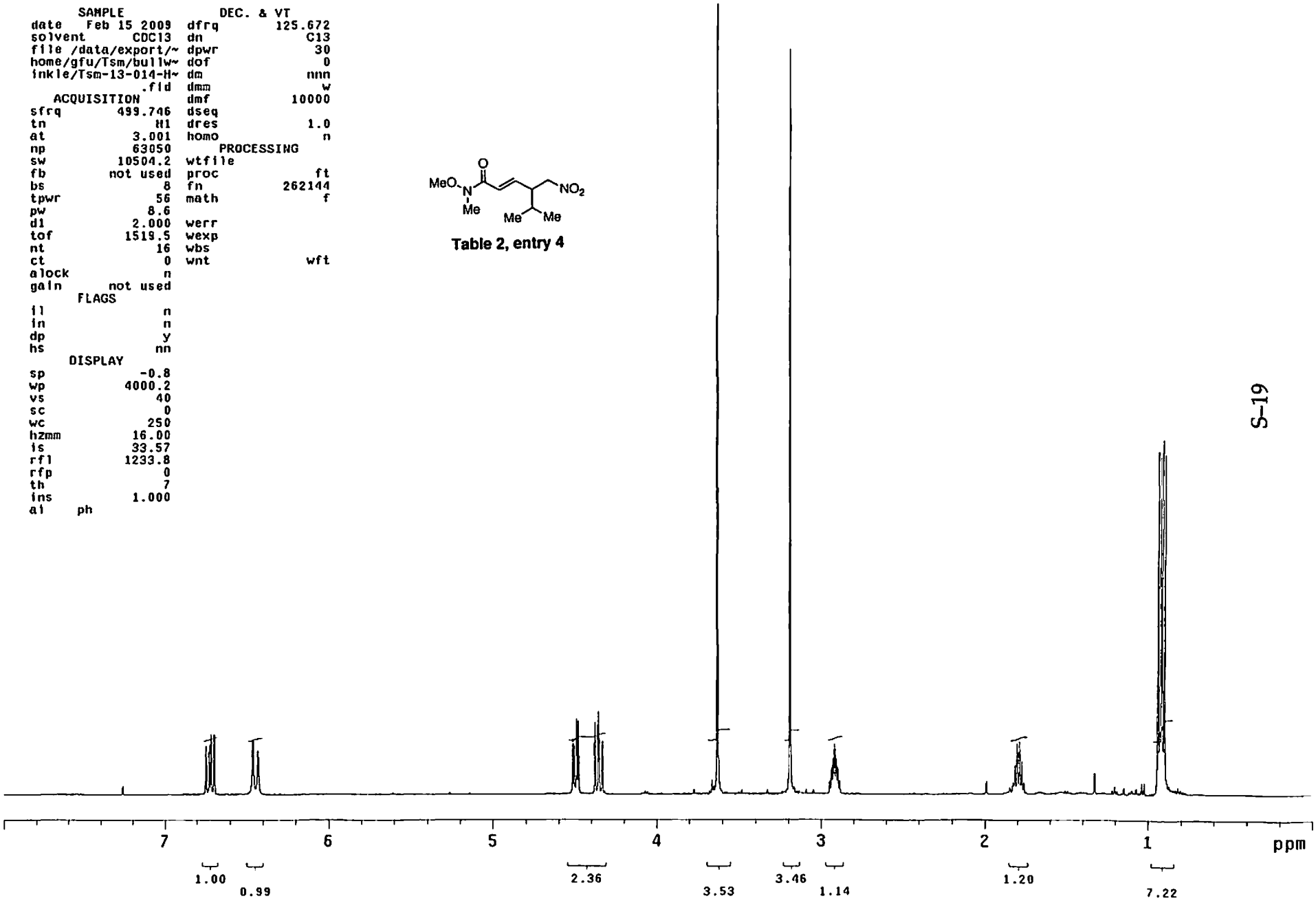


STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-014-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	PROCESSING	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.8		
wp	4000.2		
vs	40		
sc	0		
wc	250		
hzmm	16.00		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
ai	ph		

Table 2, entry 4

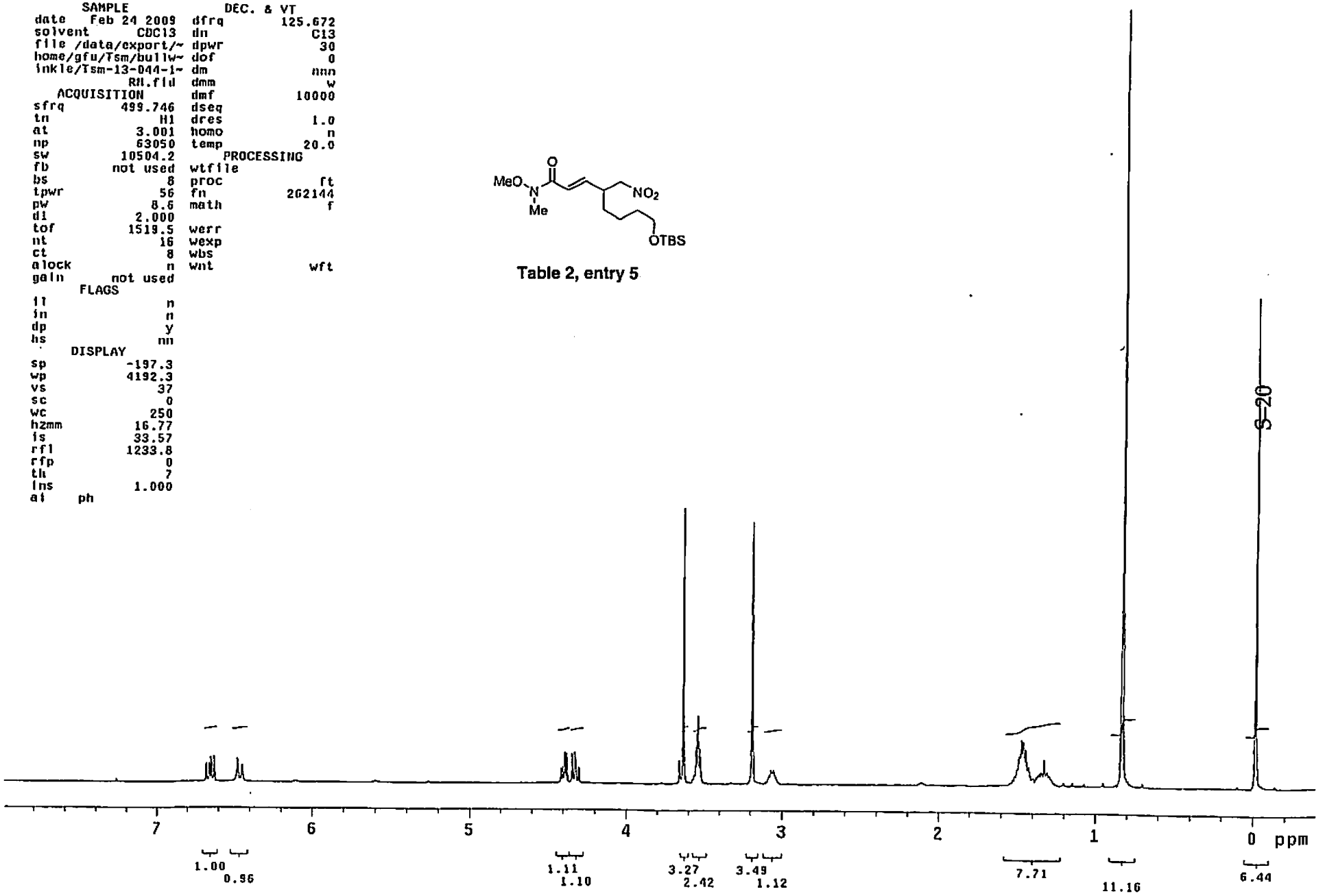


STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 24 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-044-1~		dm	nnn
	RH.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	temp	20.0
sw	10504.2	PROCESSING	
fb	not used	wtfile	
bs	8	proc	ft
tpwr	56	fn	262144
pw	8.6	math	f
d1	2.000		
tof	1519.5	werr	
nt	16	wexp	
ct	8	wbs	
alock	n	wnt	wft
gain	not used		
FLAGS			
ll	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-197.3		
wp	4192.3		
vs	37		
sc	0		
wc	250		
hzmm	16.77		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
at	ph		

Table 2, entry 5

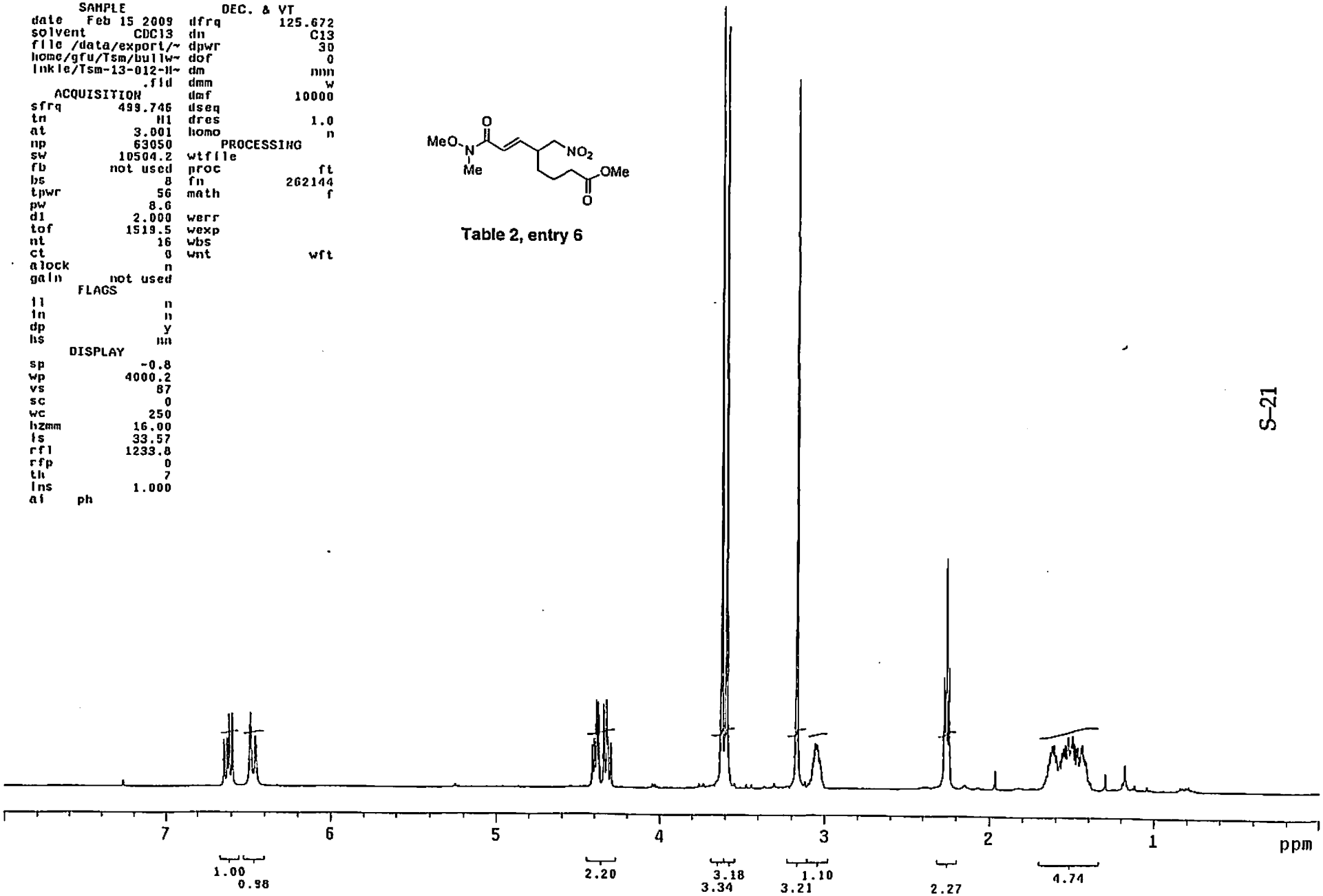


STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
linkle/Tsm-13-012-N~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
ll	n		
ln	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.8		
wp	4000.2		
vs	87		
sc	0		
wc	250		
hzmm	16.00		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
ai	ph		

Table 2, entry 6

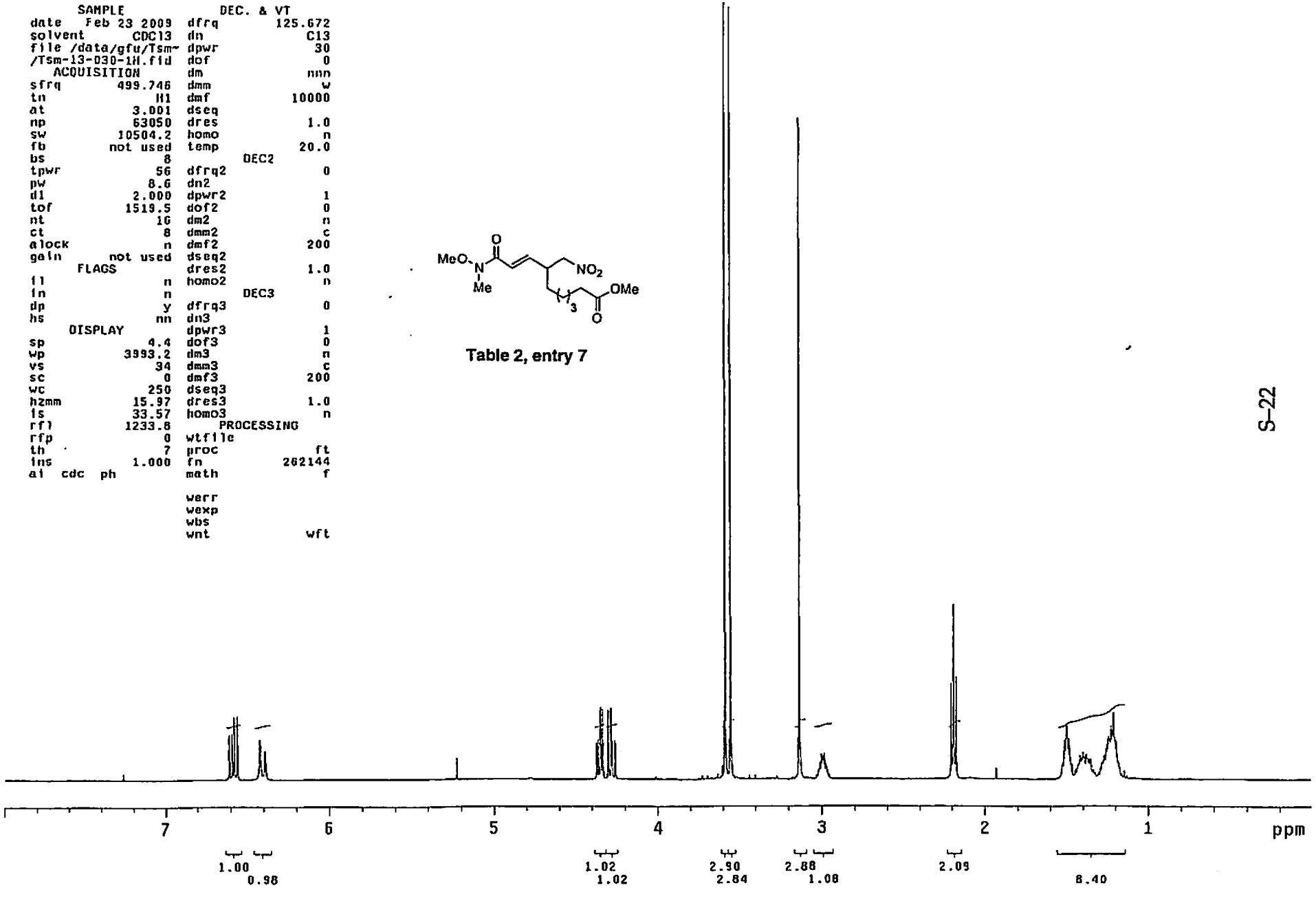


STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/gfu/Tsm~	dpwr	30
	/Tsm-13-030-1H.fid	dof	0
ACQUISITION		dm	nnn
sfrq	499.746	dmm	w
tn	H1	dmf	10000
at	3.001	dseq	
np	63050	dres	1.0
sw	10504.2	homo	n
fb	not used	temp	20.0
bs	8	DEC2	
tpwr	56	dfrq2	0
pw	8.6	dn2	
d1	2.000	dpwr2	1
tof	1519.5	dof2	0
nt	16	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
fl	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	4.4	dof3	0
wp	3993.2	dm3	n
vs	34	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.97	dres3	1.0
is	33.57	homo3	n
rfl	1233.8	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	262144
ai cdc ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

Table 2, entry 7



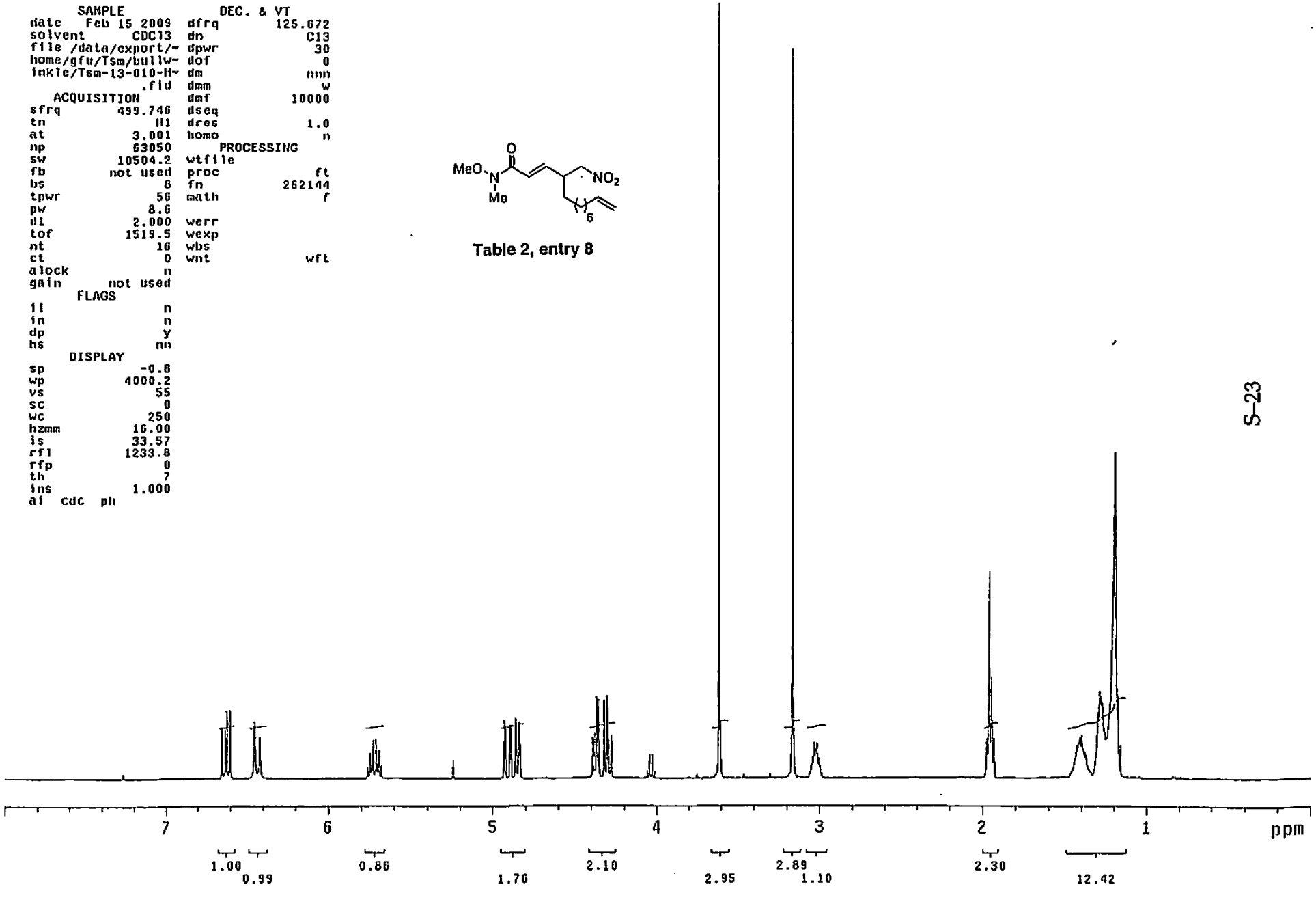
STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-010-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
dl	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.8		
wp	4000.2		
vs	55		
sc	0		
wc	250		
hzmm	16.00		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		

ai cdc ph

Table 2, entry 8

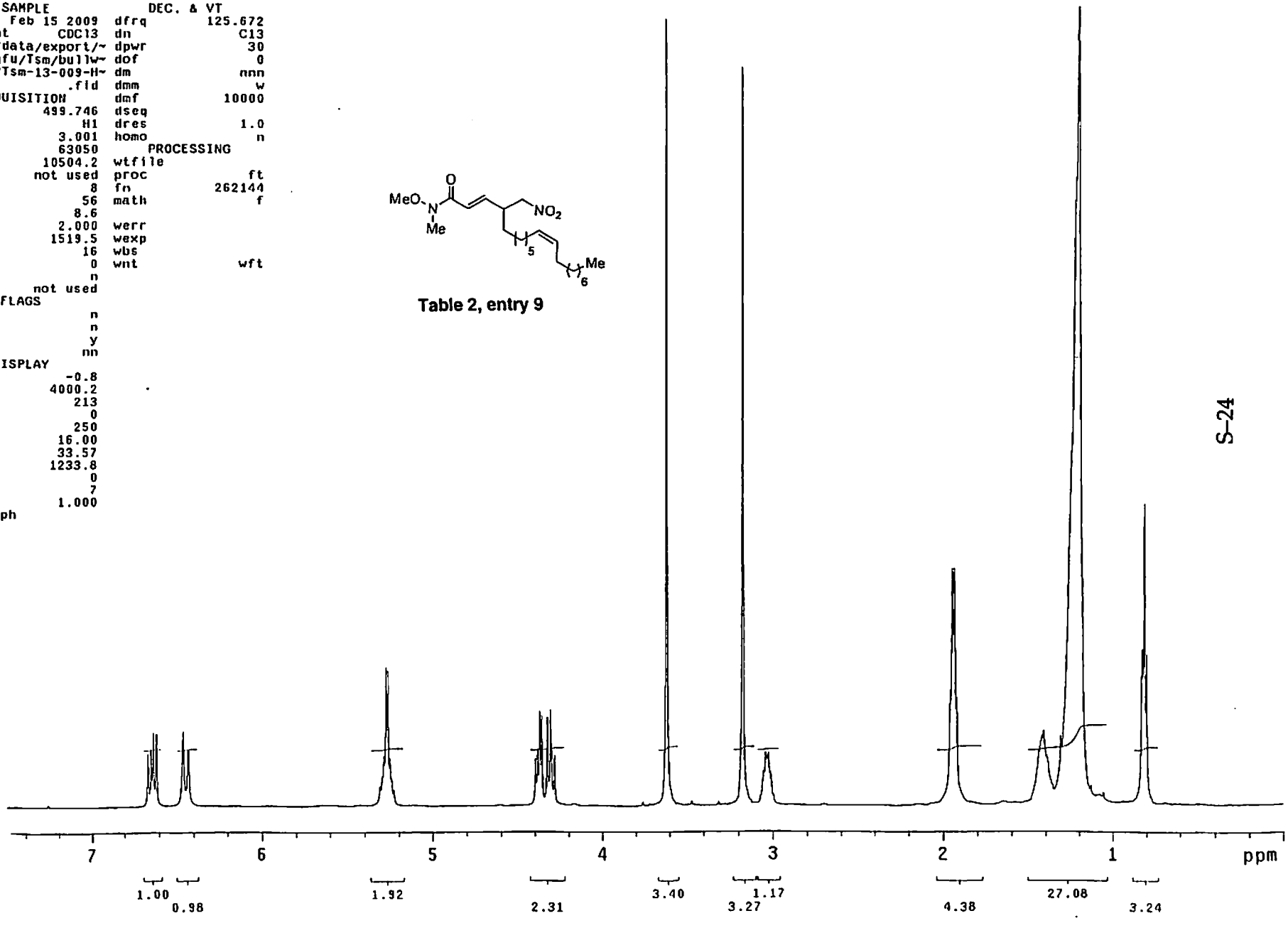


STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-009-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.8		
wp	4000.2		
vs	213		
sc	0		
wc	250		
hzmm	16.00		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
ai	ph		

Table 2, entry 9

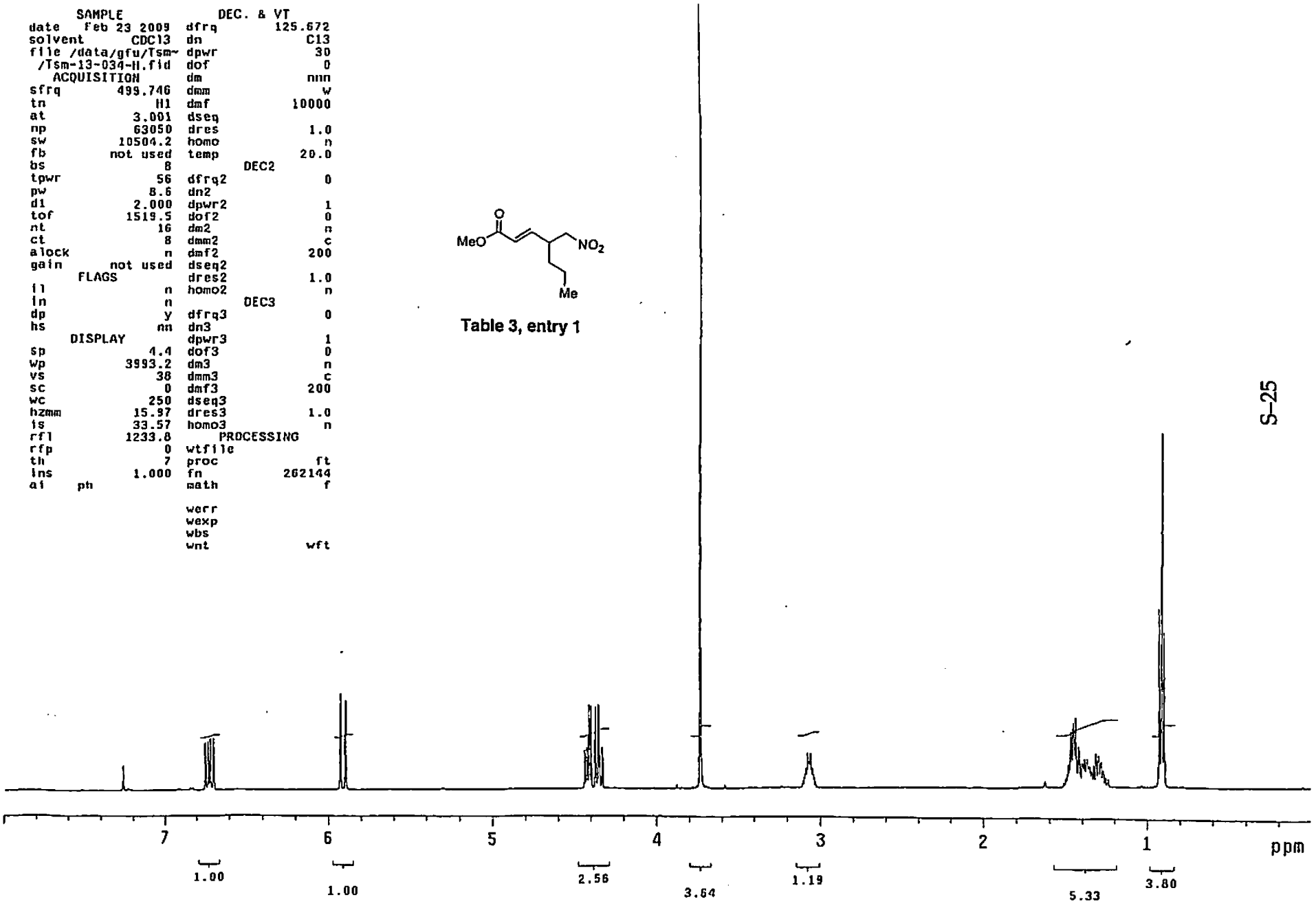


STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/gfu/Tsm~	dpwr	30
	/Tsm-13-034-H.fid	dof	0
ACQUISITION		dm	nnn
sfrq	499.746	dmm	w
tn	H1	dmf	10000
at	3.001	dseq	
np	63050	dres	1.0
sw	10504.2	homo	n
fb	not used	temp	20.0
bs	8	DEC2	
tpwr	56	dfrq2	0
pw	8.6	dn2	
d1	2.000	dpwr2	1
tof	1519.5	dof2	0
nt	16	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	4.4	dof3	0
wp	3993.2	dm3	n
vs	38	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.97	dres3	1.0
is	33.57	homo3	n
rfl	1233.8	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
ins	1.000	fn	262144
ai ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

Table 3, entry 1

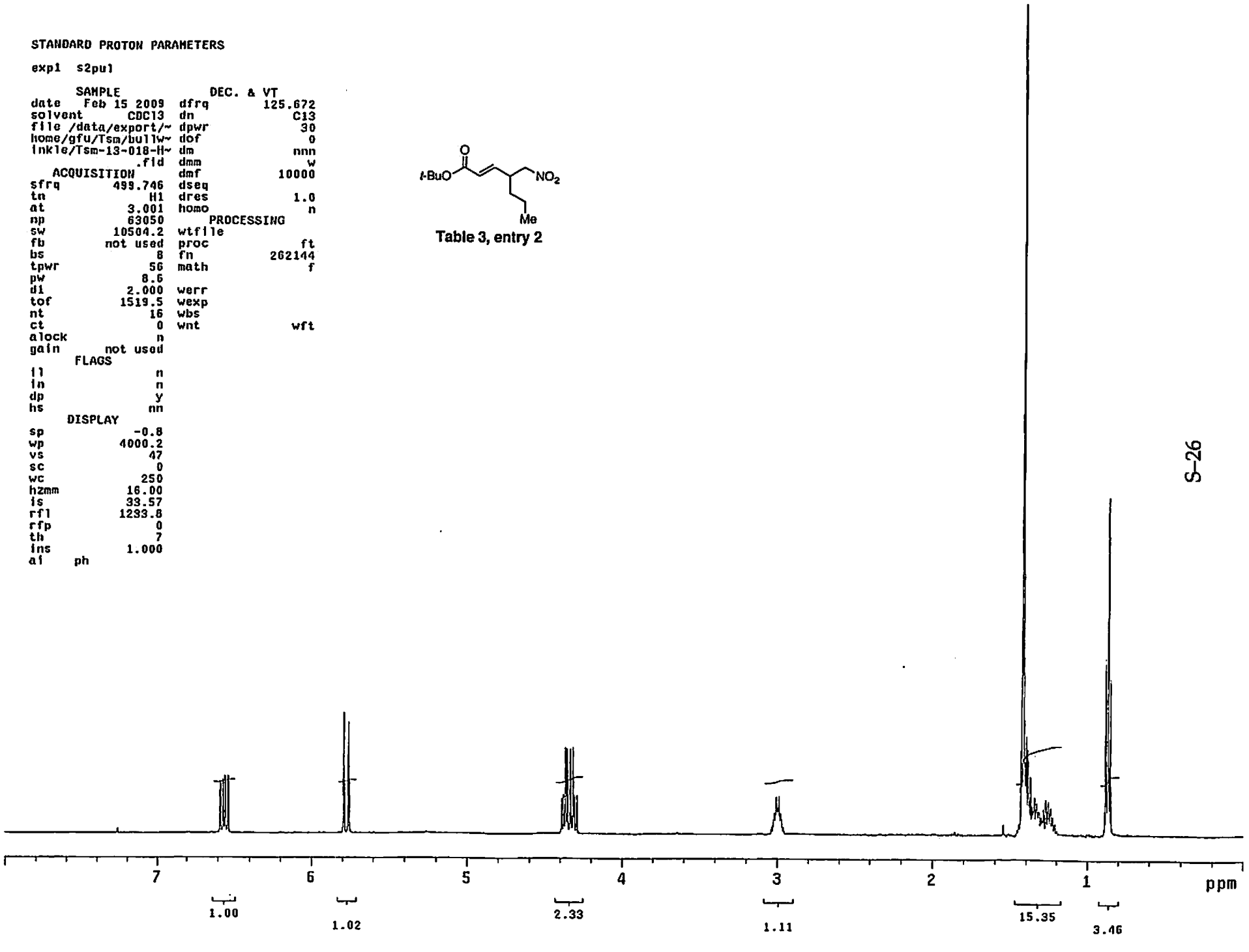


STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 15 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/Tsm/bullw~		dof	0
inkle/Tsm-13-018-H~		dm	nnn
	.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	**PROCESSING**	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	8	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-0.8		
wp	4000.2		
vs	47		
sc	0		
wc	250		
hzmm	16.00		
is	33.57		
rfl	1233.8		
rfp	0		
th	7		
ins	1.000		
ai	ph		

Table 3, entry 2



STANDARD PROTON PARAMETERS

expl s2pul

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	125.672
solvent	CDCl3	dn	C13
file	/data/gfu/Tsm~ /Tsm-13-035-H.fid	dpwr	30
		dof	0
ACQUISITION		dm	nnn
sfrq	499.746	dmm	w
tn	H1	dmf	10000
at	3.001	dseq	
np	63050	dres	1.0
sw	10504.2	homo	n
fb	not used	temp	20.0
bs	8	DEC2	
tpwr	56	dfrq2	0
pw	8.6	dn2	
d1	2.000	dpwr2	1
tof	1519.5	dof2	0
nt	16	dm2	n
ct	8	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
li	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	4.4	dof3	0
wp	3993.2	dm3	n
vs	71	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	15.97	dres3	1.0
is	33.57	homo3	n
rfl	1233.8	PROCESSING	
rfp	0	wtfile	
th	7	proc	ft
lns	100.000	fn	262144
al ph		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

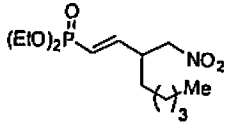
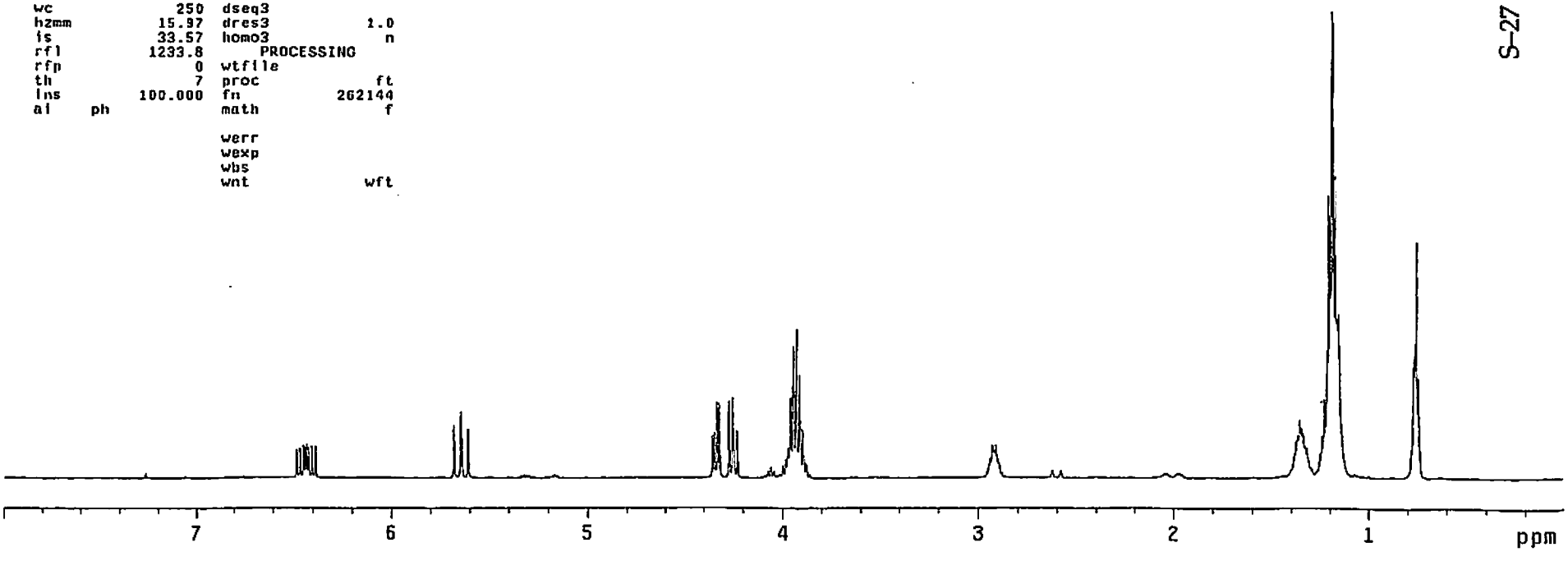


Table 3, entry 3



Data File C:\HPCHEM\1\DATA\SM\13007-1A.D

OD-H

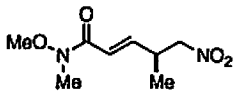
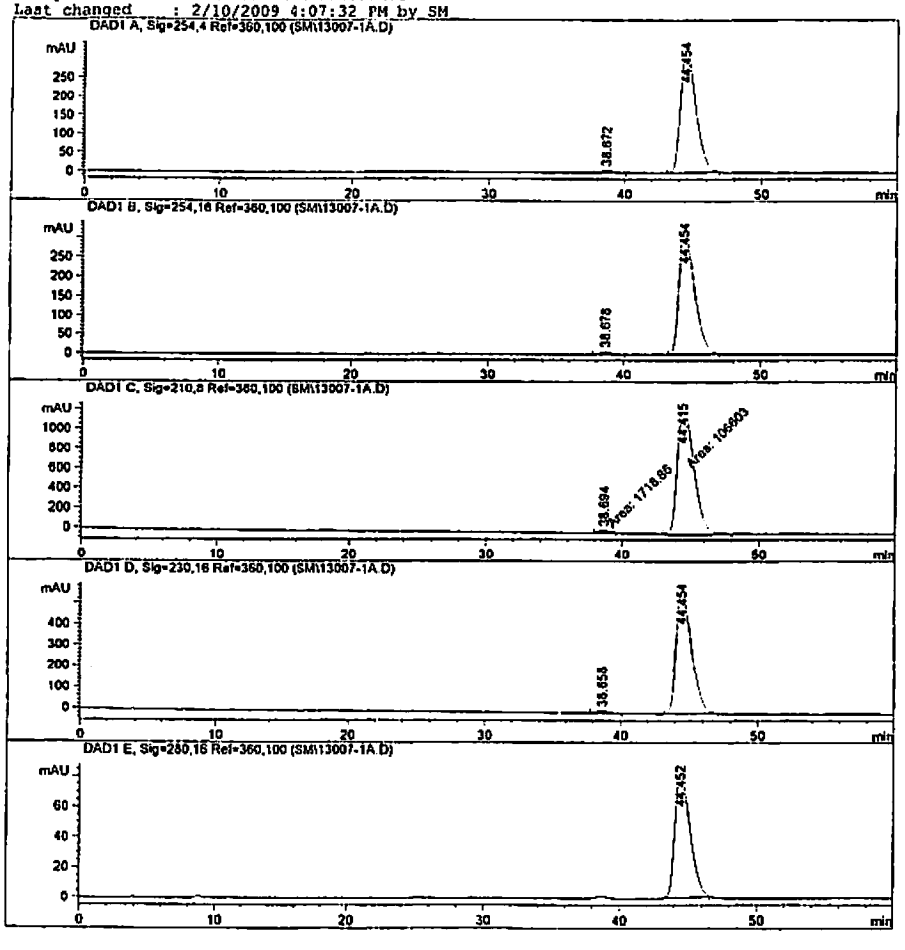


Table 2, entry 1

Injection Date : 2/10/2009 10:29:36 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual
Acq. Method : C:\HPCHEM\1\METHODS\05-60-1.M
Last changed : 3/9/2007 8:41:24 AM by EL
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 9
Location : Vial 1
Inj : 1
Inj Volume : 5 µl
Inj Volume : 8 µl



Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.672	BP	0.6864	363.56030	6.28910	1.4251
2	44.454	BB	1.0796	2.51485e4	329.08517	98.5749
Totals :				2.55120e4	335.37427	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.678	BP	0.6885	361.97119	6.20584	1.4313
2	44.454	BB	1.1042	2.49286e4	325.96777	98.5687
Totals :				2.52905e4	332.17361	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.694	MM	0.9852	1718.86450	29.07885	1.5868
2	44.415	MM	1.3986	1.06603e5	1270.31580	98.4132
Totals :				1.08322e5	1299.39465	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.658	BP	0.7399	707.64868	11.46887	1.5374
2	44.454	PB	1.0926	4.53211e4	588.40594	98.4626
Totals :				4.60287e4	599.87481	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	44.452	BB	1.0564	6322.28271	83.78082	100.0000
Totals :				6322.28271	83.78082	

Results obtained with enhanced integrator!

Data File C:\HPCHEM\1\DATA\SM\13005-1.D

OD-H

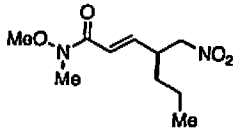
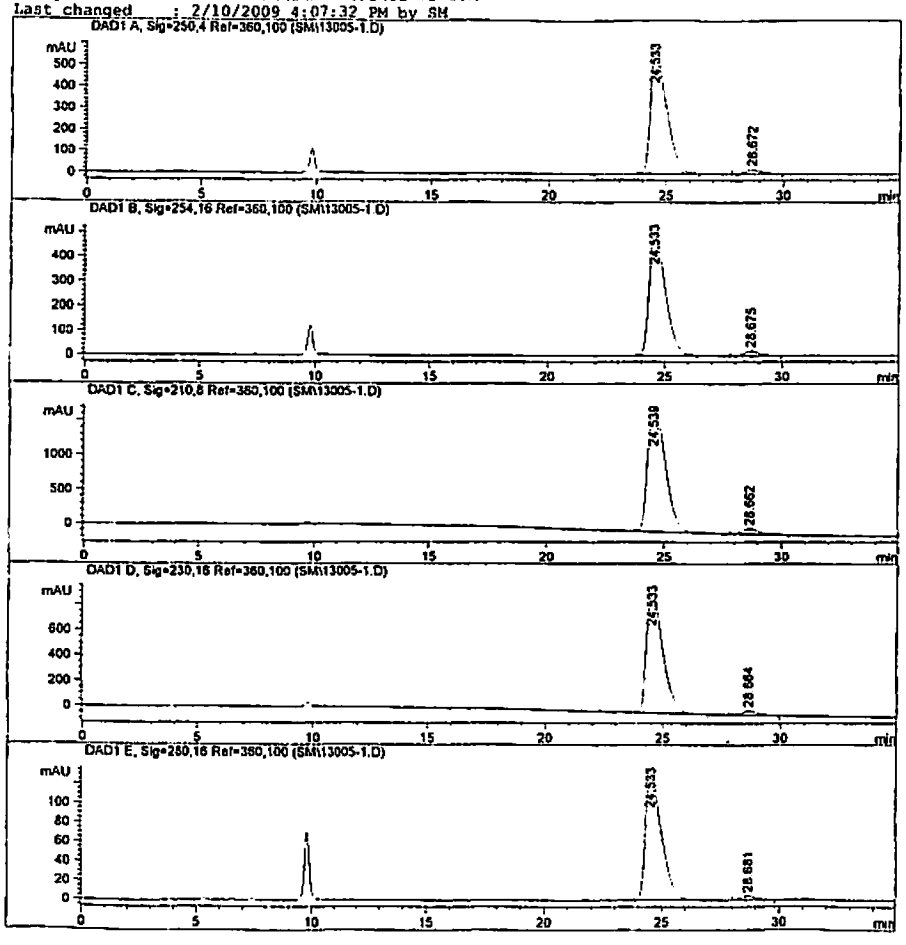


Table 2, entry 2

```
Injection Date  : 2/8/2009 5:02:23 PM          Seq. Line :   2
Sample Name     :                             Location : Vial 2
Acq. Operator   : SM                             Inj :   1
Acq. Instrument : Instrument 1                Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\05-30-1.M
Last changed    : 2/8/2009 5:19:13 PM by SM
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed    : 2/10/2009 4:07:32 PM by SM
```



Area Percent Report

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

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.533	BB	0.7097	2.74622e4	580.76764	96.6940
2	28.672	BB	0.5863	938.95251	19.07285	3.3060
Totals :				2.84011e4	599.84048	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.533	BB	0.7155	2.42312e4	512.62604	96.6803
2	28.675	BB	0.6383	832.01758	16.90186	3.3197
Totals :				2.50632e4	529.52789	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.539	VB	0.6432	9.48996e4	1754.01440	96.2429
2	28.662	BP	0.6886	3704.61230	75.75037	3.7571
Totals :				9.86042e4	1829.76478	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.533	BP	0.6845	4.58879e4	958.59790	96.6958
2	28.664	BB	0.6780	1568.02551	32.09200	3.3042
Totals :				4.74559e4	990.68990	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.533	BB	0.7058	6240.06641	132.40425	96.8554
2	28.681	BB	0.5771	202.59367	4.24286	3.1446
Totals :				6442.66008	136.64711	

Data File C:\HPCHEM\1\DATA\SM\13008-1.D

OD-H

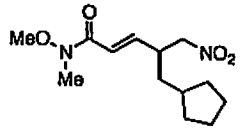
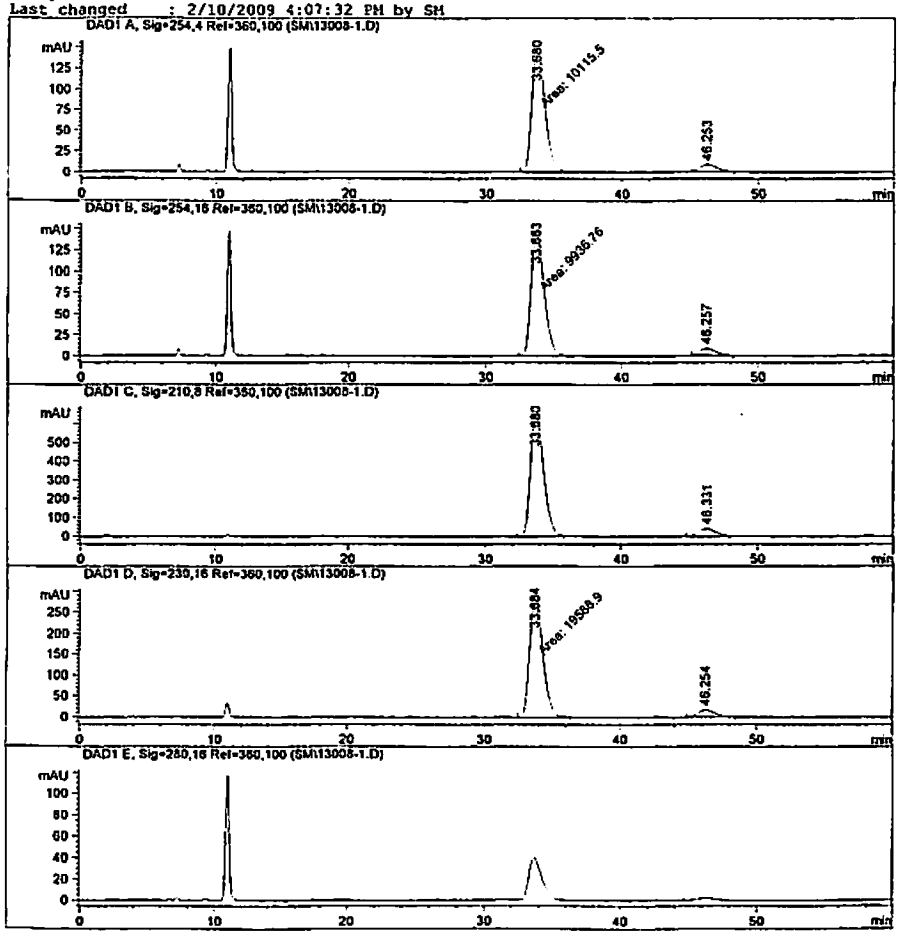


Table 2, entry 3

Injection Date : 2/10/2009 6:02:29 PM Seq. Line : 4
Sample Name : Location : Vial 2
Acq. Operator : SM Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 4 µl
Acq. Method : C:\HPCHEM\1\METHODS\03-60-1.M
Last changed : 7/17/2008 12:45:47 PM by SL
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM



Data File C:\HPCHEM\1\DATA\SM\13008-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.680	MM	1.1113	1.01155e4	151.70935	93.3345
2	46.253	BB	0.9851	722.39441	8.66056	6.6655
Totals :				1.08379e4	160.36991	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.683	MM	1.1039	9936.75586	150.03645	93.3405
2	46.257	BB	0.9934	708.95245	8.47941	6.6595
Totals :				1.06457e4	158.51586	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.680	VV	0.8758	4.39591e4	662.75397	91.9122
2	46.331	VV	1.0809	3868.17773	42.06319	8.0878
Totals :				4.78272e4	704.81716	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.684	MM	1.1046	1.95889e4	295.57217	92.8863
2	46.254	BP	1.0308	1500.21838	17.21408	7.1137
Totals :				2.10891e4	312.78625	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

Data File C:\HPCHEM\1\DATA\SM\13014-1.D

Injection Date : 2/12/2009 7:08:06 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual
Acq. Method : C:\HPCHEM\1\METHODS\05-30-1.M
Last changed : 7/1/2008 11:11:04 AM by sl
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 2
Location : Vial 1
Inj : 1
Inj Volume : 5 µl
Inj Volume : 6 µl

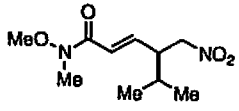
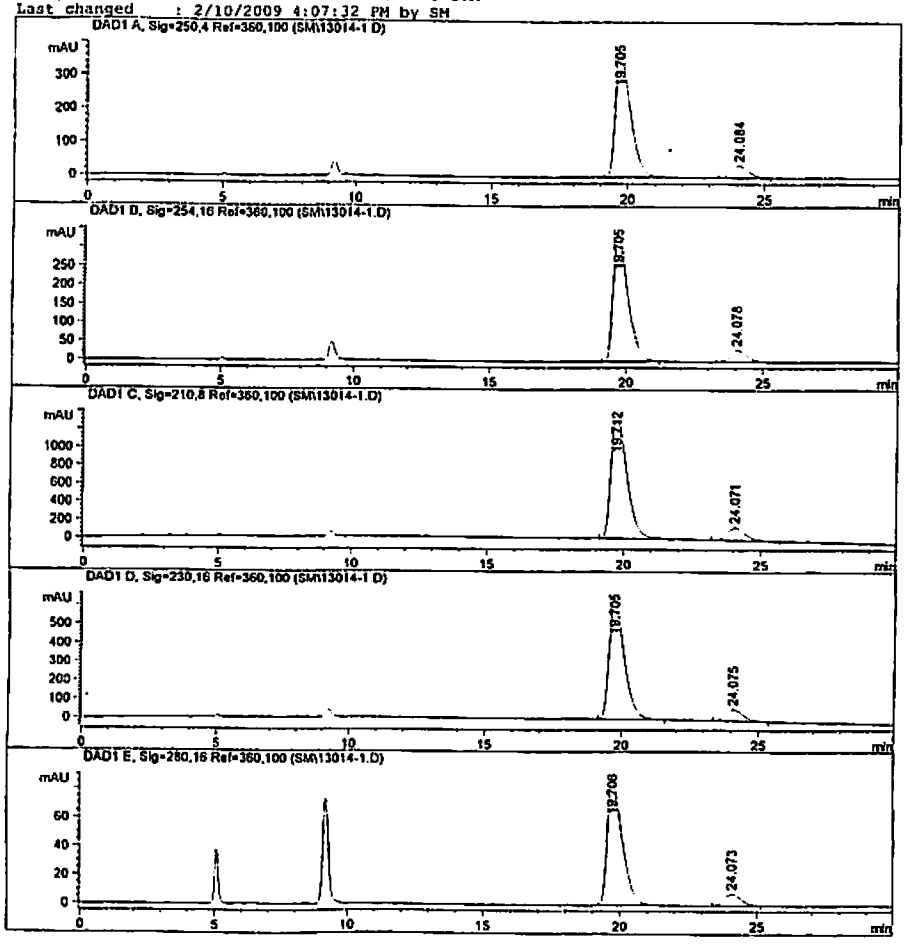


Table 2, entry 4



Data File C:\HPCHEM\1\DATA\SM\13014-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.705	BB	0.5641	1.44734e4	381.95975	90.3656
2	24.084	BB	0.5953	1543.08093	35.91256	9.6344
Totals :				1.60164e4	417.87231	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.705	BB	0.5661	1.27890e4	337.50305	90.3324
2	24.078	BB	0.6309	1368.70984	31.75201	9.6676
Totals :				1.41577e4	369.25506	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.712	BB	0.5545	5.43865e4	1331.56702	89.2348
2	24.071	VB	0.6111	6561.10986	145.23511	10.7652
Totals :				6.09476e4	1476.80212	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.705	BB	0.5770	2.42892e4	633.89062	90.1904
2	24.075	BB	0.6633	2641.82080	60.75649	9.8096
Totals :				2.69310e4	694.64712	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.706	BB	0.5568	3291.18262	87.50635	90.4473
2	24.073	BB	0.5178	347.60388	8.14615	9.5527
Totals :				3638.78650	95.65250	

Data File C:\HPCHEM\1\DATA\SM\13044-S.D

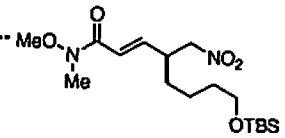
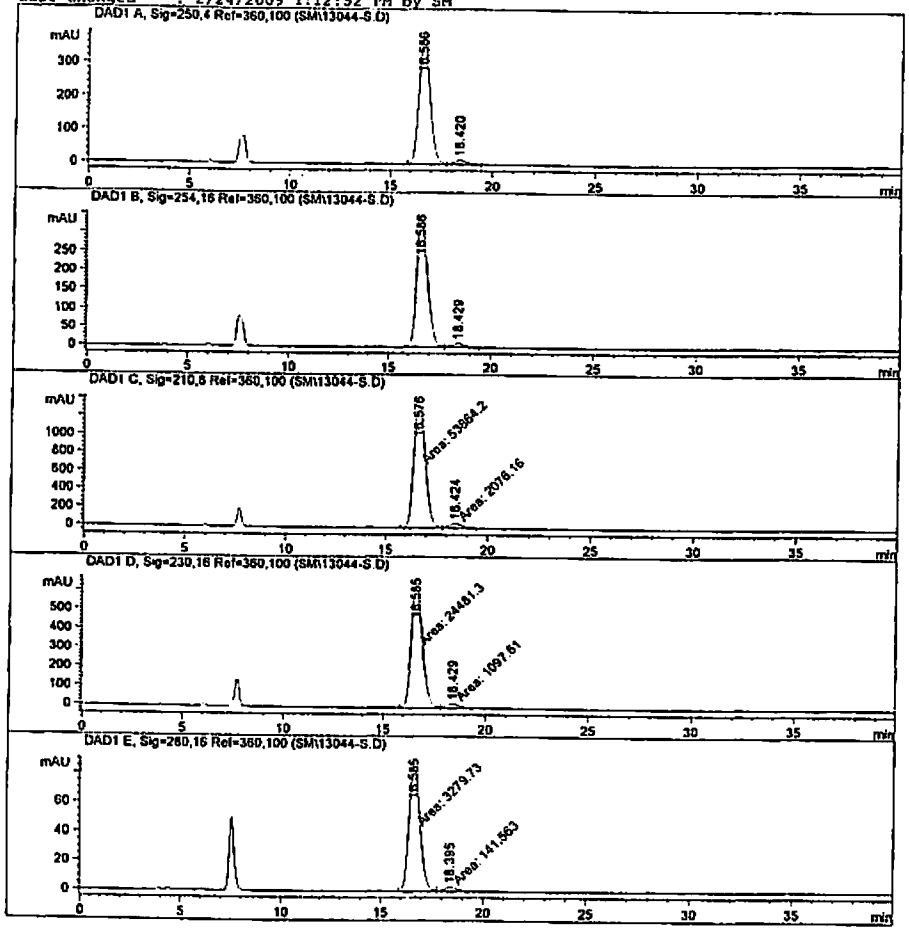


Table 2, entry 5

Injection Date : 2/24/2009 4:50:41 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual Inj Volume : 6 µl
Acq. Method : C:\HPCHEM\1\METHODS\05-40-1.M
Last changed : 10/30/2006 10:49:30 AM by EL
Analysis Method : C:\HPCHEM\1\METHODS\005-60-1.M
Last changed : 2/24/2009 1:12:52 PM by SM

Seq. Line : 2
Location : Vial 3
Inj : 1
Inj Volume : 5 µl



Data File C:\HPCHEM\1\DATA\SM\13044-S.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.586	BB	0.5796	1.42014e4	380.39679	96.5013
2	18.420	BB	0.5168	514.88049	11.94796	3.4987
Totals :				1.47162e4	392.34475	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.586	BB	0.5776	1.25333e4	335.69812	96.5001
2	18.429	BB	0.5341	454.56274	10.56262	3.4999
Totals :				1.29879e4	346.26074	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.576	MF	0.6490	5.38642e4	1383.25171	96.2886
2	18.424	FM	0.7183	2076.15698	48.17566	3.7114
Totals :				5.59403e4	1431.42736	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.585	MF	0.6316	2.44813e4	646.05109	95.7089
2	18.429	FM	0.7944	1097.61475	23.02757	4.2911
Totals :				2.55790e4	669.07866	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.585	MF	0.6260	3279.72900	87.32043	95.8623
2	18.395	FM	0.7892	141.56334	2.98963	4.1377
Totals :				3421.29234	90.31006	

Data File C:\HPCHEM\1\DATA\SM\13012-1.D

AD-H

Injection Date : 2/12/2009 9:52:18 AM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual Inj Volume : 6 µl
Acq. Method : C:\HPCHEM\1\METHODS\05-90-1.M
Last changed : 2/12/2009 9:54:19 AM by SM
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 3
Location : Vial 2
Inj : 1
Inj Volume : 5 µl

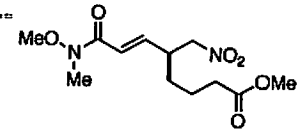
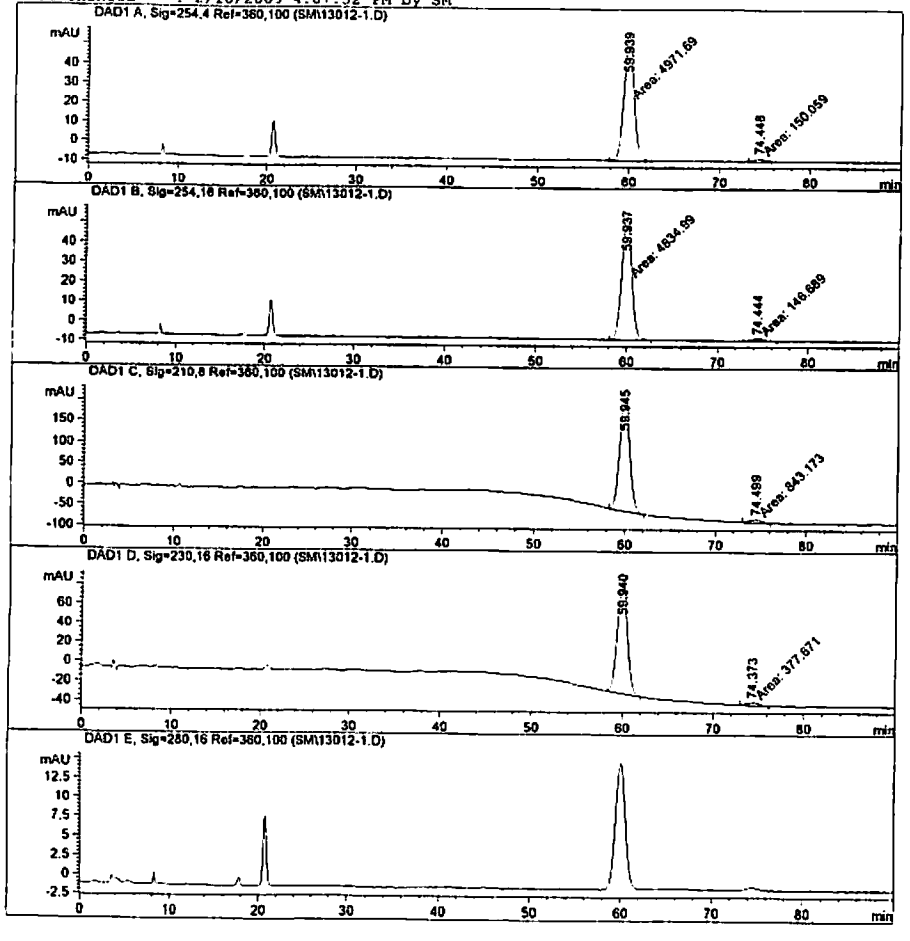


Table 2, entry 6



Data File C:\HPCHEM\1\DATA\SM\13012-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	59.939	MM	1.2985	4971.69385	63.81089	97.0702
2	74.448	MM	1.4971	150.05936	1.67054	2.9298
Totals :				5121.75320	65.48144	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	59.937	MM	1.2812	4834.98926	62.89619	97.0554
2	74.444	MM	1.6270	146.68878	1.50263	2.9446
Totals :				4981.67804	64.39882	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	59.945	VV	0.9096	2.13515e4	276.27301	96.2010
2	74.499	MM	1.7707	843.17291	7.93630	3.7990
Totals :				2.21947e4	284.20931	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	59.940	BP	1.0500	8946.74707	116.75940	95.9497
2	74.373	MM	1.9100	377.67056	3.29563	4.0503
Totals :				9324.41763	120.05503	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

S-33

Data File C:\HPCHEM\1\DATA\SM\13030-S.D

Injection Date	: 2/17/2009 5:24:47 PM	Seq. Line	: 2
Sample Name	:	Location	: Vial 1
Acq. Operator	: SM	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 5 µl
Different Inj Volume from Sequence !	Actual	Inj Volume	: 6 µl
Acq. Method	: C:\HPCHEM\1\METHODS\05-90-1.M		
Last changed	: 2/17/2009 6:37:47 PM by SM (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\05-20-1.M		
Last changed	: 12/14/2008 2:53:35 PM by sl		

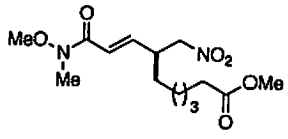
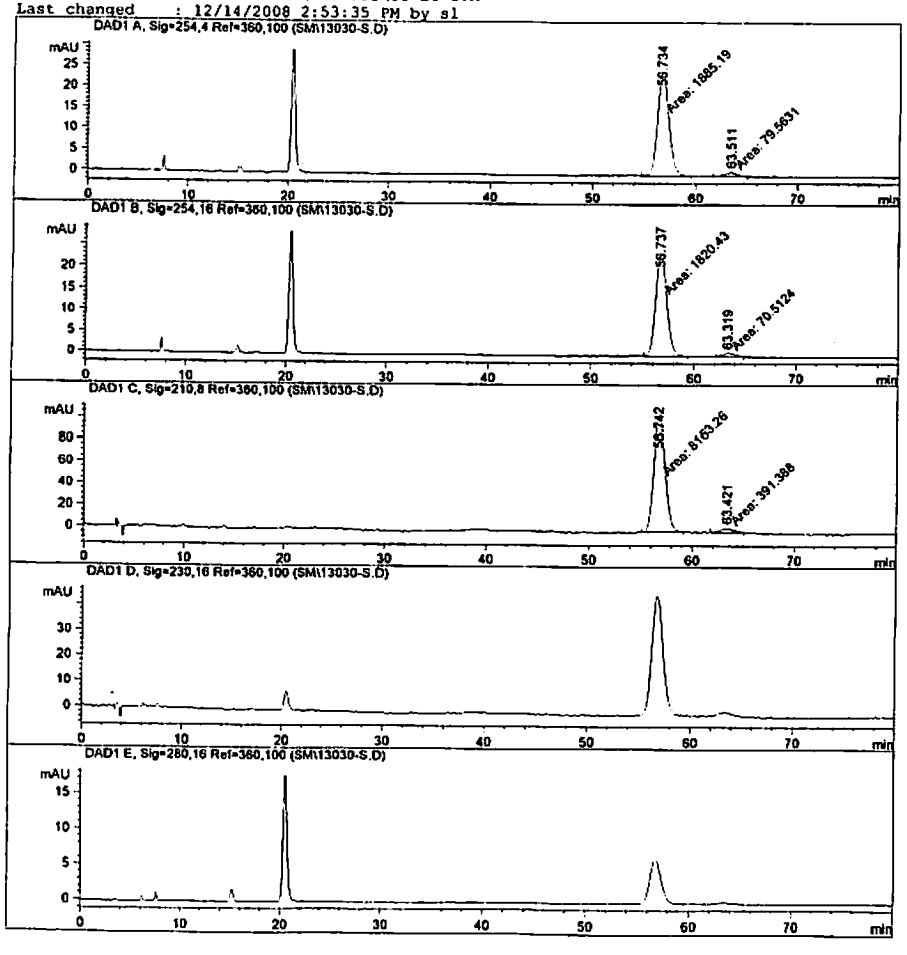


Table 2, entry 7



Data File C:\HPCHEM\1\DATA\SM\13030-S.D

Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.734	MM	1.2661	1885.18872	24.81620	95.9505
2	63.511	MM	1.3438	79.56310	9.86770e-1	4.0495
Totals :				1964.75182	25.80297	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.737	MM	1.2441	1820.43127	24.38770	96.2710
2	63.319	MM	1.3321	70.51237	8.82234e-1	3.7290
Totals :				1890.94364	25.26993	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.742	MM	1.2512	8163.25781	108.73572	95.4248
2	63.421	MM	1.5687	391.38815	4.15827	4.5752
Totals :				8554.64597	112.89398	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

Data File C:\HPCHEM\1\DATA\SM\13010-1.D

AD-H

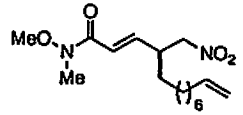
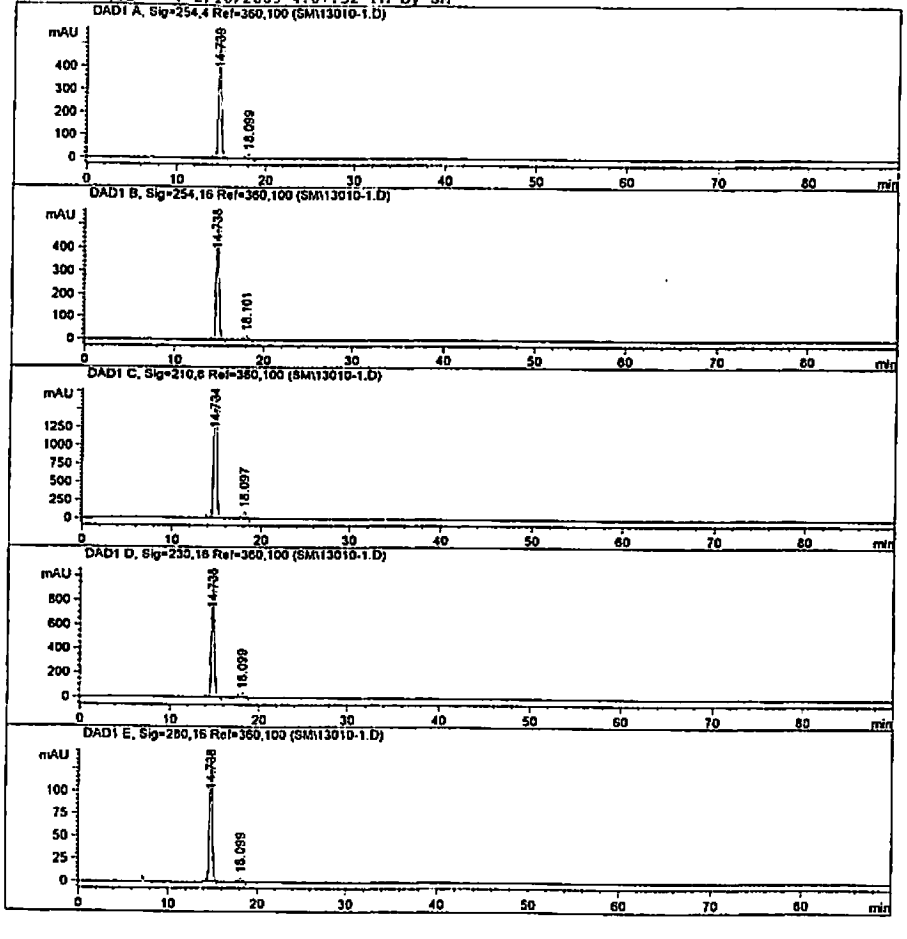


Table 2, entry 8

Injection Date : 2/11/2009 6:34:57 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual
Acq. Method : C:\HPCHEM\1\METHODS\05-90-1.M
Last changed : 2/11/2009 6:08:05 PM by SM
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 2
Location : Vial 1
Inj : 1
Inj Volume : 5 µl
Inj Volume : 6 µl



Data File C:\HPCHEM\1\DATA\SM\13010-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.739	BB	0.3246	1.15140e4	549.25677	95.9999
2	18.099	BB	0.3787	479.75781	19.66072	4.0001
Totals :				1.19937e4	568.91749	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.738	BB	0.3244	1.14076e4	544.44757	96.0089
2	18.101	BB	0.3789	474.21533	19.41656	3.9911
Totals :				1.18818e4	563.86413	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.734	BB	0.3944	4.22315e4	1696.68774	95.2896
2	18.097	BV	0.3708	2087.61768	86.13805	4.7104
Totals :				4.43191e4	1782.82580	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

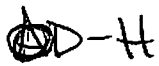
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.738	BB	0.3303	2.16140e4	1015.39569	96.0174
2	18.099	PP	0.3714	896.49451	37.17045	3.9826
Totals :				2.25104e4	1052.56614	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.738	BB	0.3204	2920.27173	140.53752	96.0468
2	18.099	PP	0.3360	120.19625	4.97303	3.9532
Totals :				3040.46798	145.51055	

Data File C:\HPCHEM\1\DATA\SM\13009-1A.D



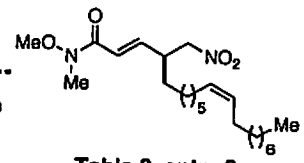
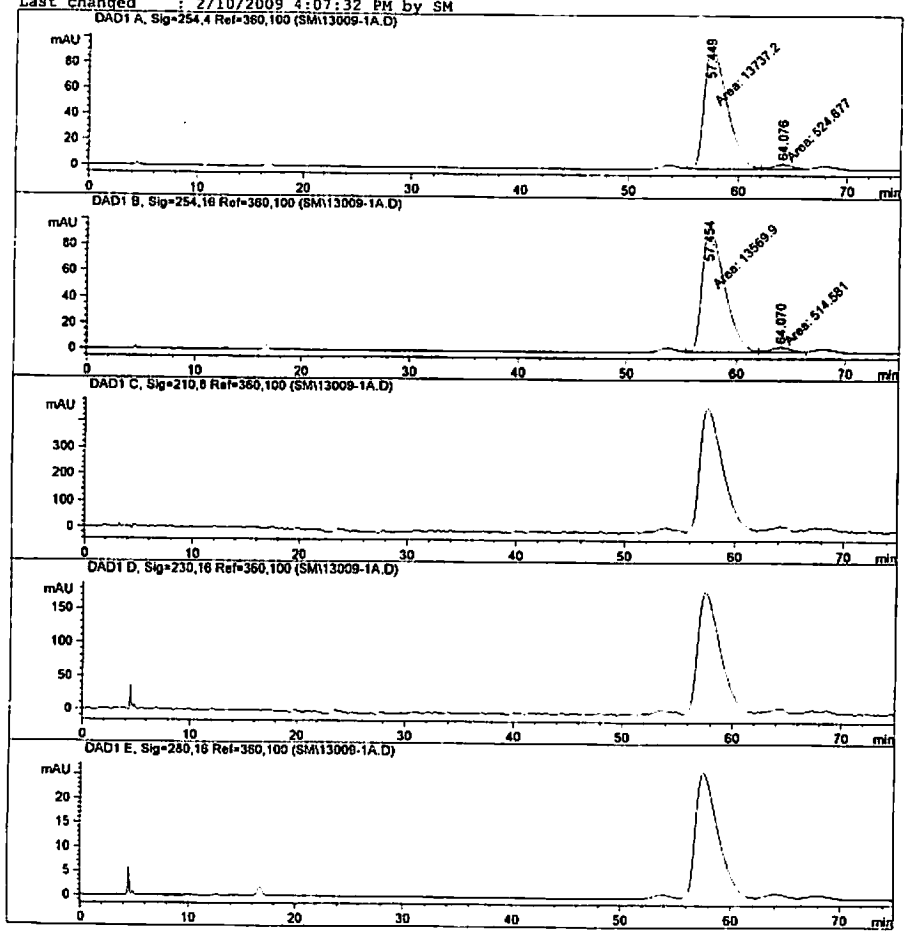


Table 2, entry 9

Injection Date : 2/10/2009 8:51:42 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual Inj Volume : 8 µl
Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 7
Location : Vial 3
Inj : 1
Inj Volume : 5 µl



Data File C:\HPCHEM\1\DATA\SM\13009-1A.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	57.449	MF	2.3473	1.37372e4	97.54067	96.3211
2	64.076	FM	2.2343	524.67651	3.91379	3.6789
Totals :				1.42619e4	101.45446	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	57.454	MF	2.3463	1.35699e4	96.39020	96.3465
2	64.070	FM	2.2276	514.58142	3.85001	3.6535
Totals :				1.40844e4	100.24020	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***

S–36

Data File C:\HPCHEM\1\DATA\SM\13020-1.D

Injection Date : 2/20/2009 10:29:16 PM
Sample Name :
Acq. Operator : sm
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual
Acq. Method : C:\HPCHEM\1\METHODS\02-40-1.M
Last changed : 3/3/2007 9:41:51 AM by XD
Analysis Method : C:\HPCHEM\1\METHODS\05-20-1.M
Last changed : 12/14/2008 2:53:35 PM by sl

Seq. Line : 5
Location : Vial 3
Inj : 1
Inj Volume : 5 µl
Inj Volume : 6 µl

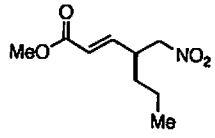
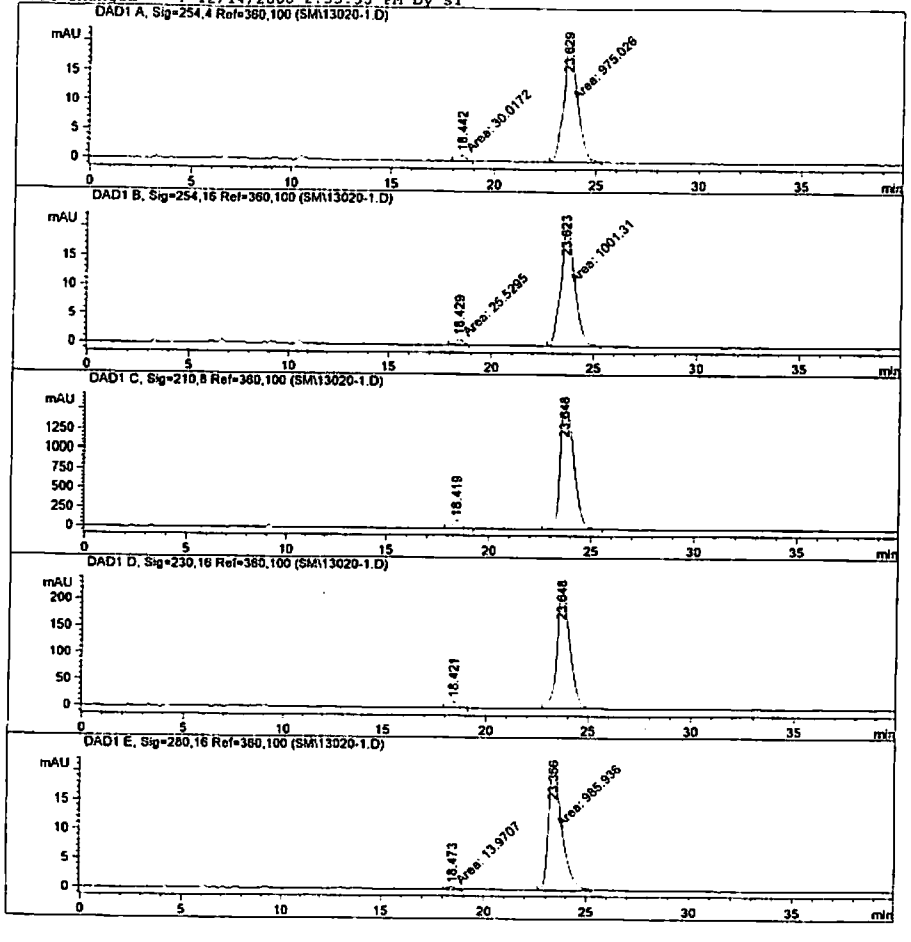


Table 3, entry 1



Data File C:\HPCHEM\1\DATA\SM\13020-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.442	MM	0.4747	30.01718	1.05398	2.9867
2	23.629	MM	0.7619	975.02582	21.32962	97.0133
Totals :				1005.04300	22.38360	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.429	MM	0.4337	25.52950	9.81158e-1	2.4862
2	23.623	MM	0.7694	1001.31482	21.69151	97.5138
Totals :				1026.84432	22.67267	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.419	BB	0.4093	2961.02026	108.87658	3.8205
2	23.648	VB	0.5992	7.45419e4	1624.98608	96.1795
Totals :				7.75029e4	1733.86266	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.421	BB	0.3775	318.10266	11.83834	3.1625
2	23.648	BB	0.6192	9740.58594	229.49974	96.8375
Totals :				1.00587e4	241.33808	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.473	MM	0.5180	13.97071	4.49494e-1	1.3972
2	23.366	MM	0.7677	985.93646	21.40515	98.6028
Totals :				999.90717	21.85465	

Data File C:\HPCHEM\1\DATA\SM\13018-1.D

Injection Date : 2/12/2009 9:13:50 PM
Sample Name :
Acq. Operator : SM
Acq. Instrument : Instrument 1
Different Inj Volume from Sequence ! Actual
Acq. Method : C:\HPCHEM\1\METHODS\01-20-1.M
Last changed : 6/6/2008 2:46:18 PM by SM
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

Seq. Line : 6
Location : Vial 3
Inj : 1
Inj Volume : 5 µl
Inj Volume : 6 µl

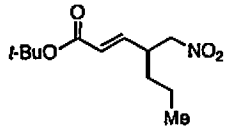
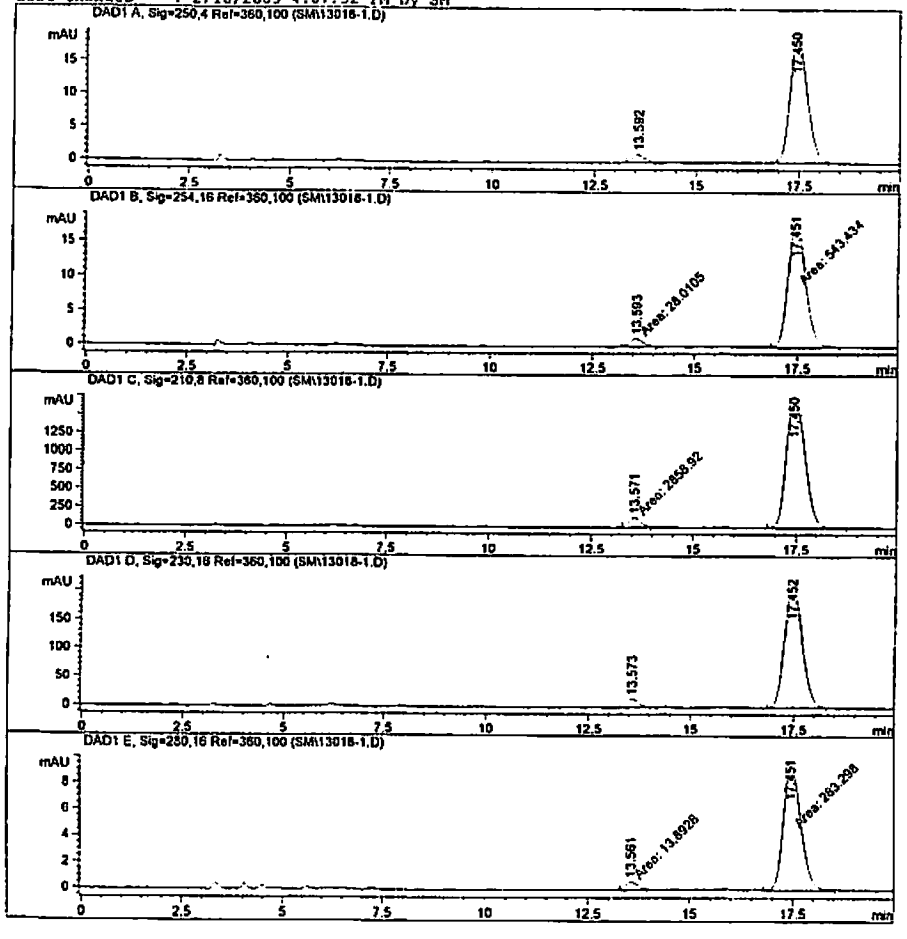


Table 3, entry 2



Data File C:\HPCHEM\1\DATA\SM\13018-1.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.592	PB	0.2515	24.54738	1.17634	4.2871
2	17.450	BB	0.4138	548.03314	18.92690	95.7129
Totals :				572.58052	20.10324	

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.593	MM	0.3823	28.01051	1.22115	4.9017
2	17.451	MM	0.4931	543.43402	18.36876	95.0983
Totals :				571.44453	19.58991	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.571	MM	0.3551	2858.91895	134.20009	4.9956
2	17.450	BB	0.5061	5.43695e4	1686.35242	95.0044
Totals :				5.72284e4	1820.55251	

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.573	BP	0.3365	313.62592	14.37480	4.7328
2	17.452	PB	0.4556	6312.99707	214.35719	95.2672
Totals :				6626.62299	228.73199	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.561	MM	0.3605	13.89283	6.42298e-1	4.6747
2	17.451	MM	0.4942	283.29761	9.55461	95.3253
Totals :				297.19044	10.19690	

Data File C:\HPCHEM\1\DATA\SM\13019-01.D

Injection Date : 2/13/2009 7:58:08 PM Seq. Line : 3
Sample Name : Location : Vial 2
Acq. Operator : SM Inj : 1
Acq. Instrument : Instrument 1 Inj Volume : 5 µl
Different Inj Volume from Sequence ! Actual Inj Volume : 6 µl
Acq. Method : C:\HPCHEM\1\METHODS\02-60-1.M
Last changed : 8/31/2008 3:07:52 PM by GR
Analysis Method : C:\HPCHEM\1\METHODS\01-75-1.M
Last changed : 2/10/2009 4:07:32 PM by SM

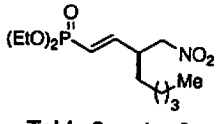
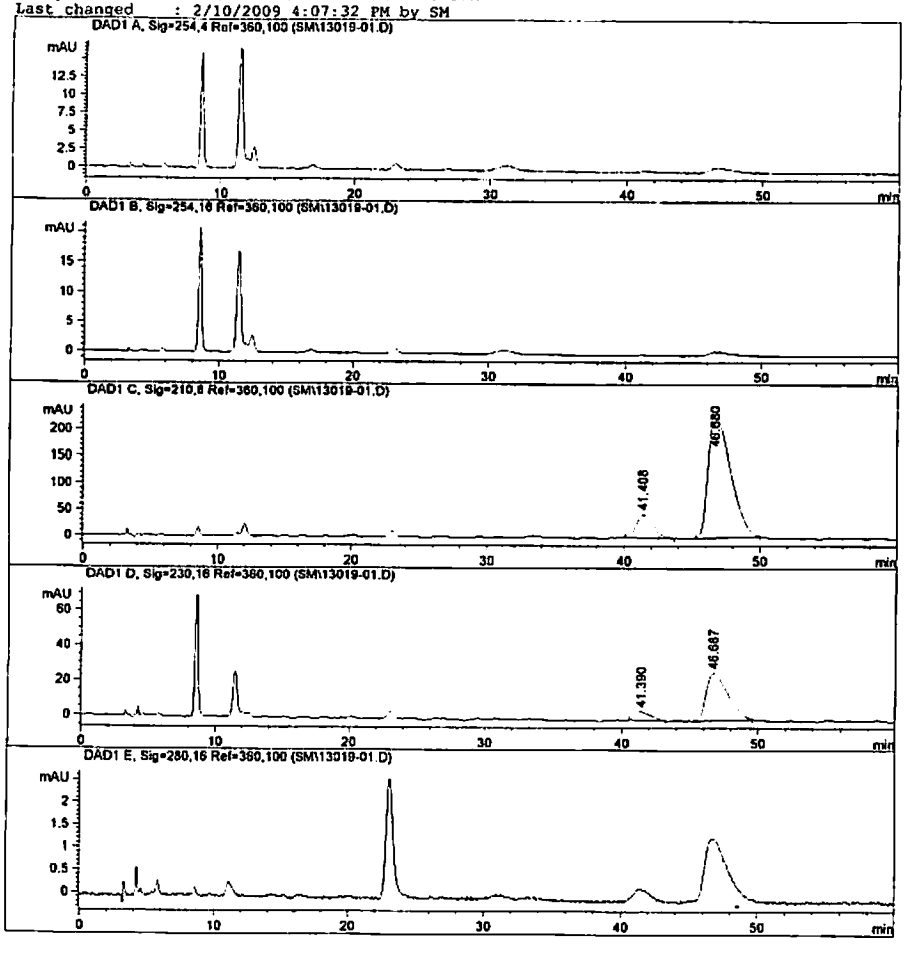


Table 3, entry 3



Data File C:\HPCHEM\1\DATA\SM\13019-01.D

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	41.408	VP	1.0040	3788.84009	44.83234	12.4095
2	46.680	VB	1.3280	2.67429e4	237.45172	87.5905

Totals : 3.05318e4 282.28407

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	41.390	BB	0.9385	395.13983	4.98633	11.6755
2	46.687	PB	1.3101	2989.19727	26.90652	88.3245

Totals : 3384.33710 31.89284

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***