Supporting Information for

Asymmetric Reduction of Oxime Ethers Promoted by Chiral Spiroborate Esters with an O_3BN Framework

Yunbo Chu, Zixing Shan*, Dejun Liu and Nannan Sun

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General.

Melting points were uncorrected. Column chromatography was performed on silica gel (200-300 mesh). 300 MHz ^{1}H NMR and 75 MHz ^{13}C NMR spectra were recorded in $CDCl_3$ in ppm, *J* in Hz. Optical rotations were recorded on digital polarimeter, Enantiomeric excess of the (*S*)-1-aralkylamines was determined for their acetylated derivative by HPLC analysis with a chiral column, flow rate 1.0 mL/min; hexane/2-propanol = 90/10; UV, 254 nm; the racemic aralkylamines were prepared according to a similar procedure to the literature.[1] All the reductions were carried out under an atmosphere of dry Ar. Tetrahydrofuran (THF) was freshly distilled from sodium benzophenone under Ar prior to use. 1-(4-methoxyphenyl)ethanone, 1-(4-methoxy phenyl)-propan-1-one and 1-(4-methoxy phenyl)butan-1-one were prepared according to the standard method.

Preparation of the oximes:

A representative procedure is as follows

1-(4-methoxyphenyl)ethanone oxime: To a suspension of hydroxylamine hydrochloride (4.17 g) and anhydrous K_2CO_3 (8.28 g) in 150 mL absolute ethanol was added 1-(4-methoxyphenyl)-ethanone (7.50 g). The reaction mixture was stirred for 24 h under reflux, filtered, and then evaporated to remove ethanol. The residue was dissolved in water and extracted with diethyl ether (3 × 40 mL). The combined ether layers were washed with saturated brine and dried over anhydrous Na_2SO_4, 7.01 g of 1-(4-methoxyphenyl)ethanone oxime was afforded as white solid after evaporation of the solvent and column chromatography on silica gel (petroleum ether/ethyl acetate, 10 : 2, v/v), 85% yield, mp. 76-78 °C. IR (KBr, cm^{-1}): ν 3244, 3007, 2965, 2936, 2839, 1610, 1574, 1516, 1461, 1368, 1258, 1175, 1025, 922, 831. ^1H NMR ($CDCl_3$): δ 2.27 (s, 3H), 3.84 (s, 3H), 6.90 (d, *J*=9.0 Hz, 2H), 7.58 (d, *J*=9.0 Hz, 2H), 7.80 (s, 1H). Anal. Calcd. for $C_9H_{11}NO_2$: C, 65.44; H, 6.71; N, 8.48. Found: C, 65.21; H, 6.69; N, 8.65.

Other oximes were prepared according to similar procedure.

1-(4-Methoxyphenyl)propan-1-one oxime: white solid, 87% yield, mp 65-67 °C. IR (KBr, cm^{-1}): ν 3287, 3065, 2971, 2938, 2838, 1607, 1575, 1515, 1465, 1299, 1254, 1180, 1037, 970, 914, 835. ^1H NMR ($CDCl_3$): δ 1.06 (t, *J*=6.9 Hz, 3H), 2.69(q, *J*=6.9 Hz, 2H), 3.73 (s, 3H), 6.80 (d, *J*=7.5 Hz, 2H), 7.46 (d, *J*=7.5 Hz, 2H), 8.52 (s, 1H). Anal. Calcd. for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.78; H, 7.26; N, 7.78.

1-(4-Methoxyphenyl)butan-1-one oxime: white solid, 82% yield, mp 54-56 °C. IR (KBr, cm^{-1}): ν 3286, 3251, 3069, 2927, 2871, 2838, 1601, 1517, 1464, 1327, 1260, 1177, 1029, 935, 827. ^1H NMR ($CDCl_3$): δ 0.98 (t, *J* = 7.2 Hz, 3H), 1.60 (m, *J*=7.2 Hz, 2H), 2.77 (t, *J*=7.2 Hz, 2H), 3.83 (s, 3H), 6.90 (d, *J*=8.1 Hz, 2H), 7.56 (d, *J*=8.1 Hz, 2H). Anal. Calcd. for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25. Found: C, 68.28; H, 7.75; N, 7.21.

Acetophenone oxime: white solid, 92% yield, mp 54-55 °C (lit.[2] mp 59 °C). IR (KBr, cm^{-1}): ν 3250, 3112, 3067, 3035, 2932, 1648, 1495, 1450, 1371, 1302, 1080, 1007, 927, 762, 694. ^{1}H NMR ($CDCl_3$): δ 2.29 (s, 3H), 7.37-7.64 (m, 5H), 7.44 (s, 1 H). Anal. Calcd. for C_8H_9NO: C, 71.09; H, 6.71; N, 10.36. Found: C, 70.82; H, 6.56; N, 10.45.

Propiophenone oxime: white solid, 90% yield, mp 51-52 °C. IR (KBr, cm^{-1}): ν 3296, 3250, 3059, 2975, 2938, 2878, 1629, 1576, 1498, 1464, 1297, 1036, 971, 915, 767, 694. ^{1}H NMR ($CDCl_3$): δ 1.18 (t, *J*=7.2 Hz, 3H), 2.84 (d, *J*=7.2 Hz, 2H), 7.38-7.61 (m, 5H). Anal. Calcd. for $C_9H_{11}NO$: C, 72.46; H, 7.43; N, 9.39, Found: C, 72.34; H, 7.38; N, 9.42.

1-(4-Nitrophenyl)ethanone oxime: light yellow solid, 88% yield, mp 170-171 °C (lit.[3] mp 172-174 °C). IR (KBr, cm^{-1}): ν 3253, 3112, 3078, 2926, 1601 1513, 1340, 1112, 1010, 930, 854, 774, 753, 691. ^{1}H NMR ($CDCl_3$): δ 2.33 (s, 3H), 7.81 (d, *J*=7.5 Hz, 2H), 8.24 (d, *J*=7.5 Hz, 2H). Anal. Calcd. for $C_8H_8N_2O_3$: C, 53.33; H, 4.48; N, 15.55. Found: C, 53.12; H, 4.42; N, 15.45.

Preparation of the oxime ethers:

A representative procedure is as follows:

1-(4-Methoxyphenyl)ethanone O-methyl oxime. To a suspension of NaH (1.15 g) in 100 mL dry N,N-dimethylformamide (DMF) was added 1-(4-methoxyphenyl)ethanone oxime (6.60 g) in dry DMF (50 mL) at 0 °C (ice-bath). The reaction mixture was stirred for 2 h at 0 °C and MeI (6.82 g) was added. The ice-bath was removed and the reaction mixture stirred for an additional 4 h. After removal of DMF under reduced pressure, the residue was dissolved in water and extracted with diethyl ether (3 × 40mL). The combined ether layers were washed with saturated brine and dried anhydrous Na_2SO_4. After evaporation of the solvent and column chromatography on silica gel (petroleum ether/ethyl acetate, 16 : 2, v/v), 5.73 g 1-(4-methoxyphenyl)ethanone O-methyl oxime was afforded, 80% yield, mp 52-53 °C (lit.[4] mp 50-52 °C). IR (KBr, cm^{-1}): ν 3003, 2962, 2941, 2903, 2839, 2822, 1613, 1599, 1516, 1458, 1444, 1257, 1175, 1049, 1026, 892, 828. ^{1}H NMR ($CDCl_3$): δ 2.21(s, 3H), 3.83(s, 3H), 3.98(s, 3H), 6.88 (d, *J*=8.7 Hz, 2H), 7.59 (d, *J*=8.7 Hz, 2H). Anal. Calcd. for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.88; H, 7.28; N, 7.74.

Other oxime ethers were prepared similarly.

(4-Methoxyphenyl)-propan-1-one O-methyl oxime, colourless oil, 84% yield. IR (neat, cm^{-1}): ν 3045, 2968, 2937, 2896, 2837, 2816, 1610, 1568, 1513, 1464, 1338, 1306, 1253, 1179, 1051, 958, 878, 835. ^1H NMR ($CDCl_3$): δ 1.13 (t, *J*=7.2 Hz, 3H), 2.72 (q, *J*=7.2 Hz, 2H), 3.83 (s, 3H), 3.96(s, 3H), 6.89 (d, *J*=9.0 Hz, 2H), 7.57(d, *J*=9.0 Hz, 2H). Anal. Calcd. for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25. Found: C, 68.18; H, 7.78; N, 7.20.

1-(4-Methoxyphenyl)butan-1-one O-methyl oxime, colourless oil, 80% yield. IR (neat, cm^{-1}): ν 3045, 2961, 2935, 2899, 2873, 2837, 2816, 1611, 1567, 1514, 1464, 1303, 1251, 1178, 1052, 907, 888, 834. ^1H NMR ($CDCl_3$): δ 0.97 (t, *J*=7.2 Hz, 3H), 1.56 (m, *J*=7.2 Hz, 2H), 2.70 (m, *J*=7.2 Hz, 2H), 3.82 (s, 3H), 3.96 (s, 3H), 6.89 (d, *J*=9.0 Hz, 2H), 7.59 (d, *J*=9.0 Hz, 2H). Anal. Calcd. for $C_{12}H_{17}NO_2$: C, 69.54; H, 8.27; N, 6.76. Found: C, 69.32; H, 8.24; N, 6.62.

Acetophenone O-methyl oxime, colourless oil, 90% yield. IR (neat, cm^{-1}): ν 3057, 2933, 2854, 2817, 1611, 1496, 1463, 1444, 1369, 1052, 893, 760, 693. ^1H NMR ($CDCl_3$): δ 2.24 (s, 3H), 4.02 (s, 3H), 7.36-7.64 (m, 5H). Anal. Calcd. for $C_9H_{11}NO$: C, 72.46; H, 7.43; N, 9.39. Found: C, 72.30; H, 7.38; N, 9.29.

Acetophenone O-benzyl oxime, colourless oil, 88% yield. IR (neat, cm^{-1}): ν 3061, 3031, 2926, 2872, 1608, 1496, 1449, 1367, 1311, 1021, 929, 880, 759, 695. ^1H NMR ($CDCl_3$): δ 2.27 (s, 3H), 5.24 (s, 2H), 7.30-7.65 (m, 10H). Anal. Calcd. for $C_{15}H_{15}NO$: C, 79.97; H, 6.71; N, 6.22. Found: C, 79.78; H, 6.65; N, 6.12.

Propiophenone O-methyl oxime, colourless oil, 89%. IR (neat, cm^{-1}): ν 3059, 2973, 2938, 2899, 2879, 2817, 1690, 1597, 1496, 1463, 1342, 1220, 1051, 957, 879, 768, 694. ^1H NMR ($CDCl_3$): δ 1.13 (t, *J*=7.2 Hz, 3H), 2.75 (q, *J*=7.2 Hz, 2H), 3.98 (s, 3H), 7.34-7.64 (m, 5H). Anal. Calcd. for $C_{10}H_{13}NO$: C, 73.59; H, 8.03; N, 8.58. Found: C, 73.32; H, 7.93; N, 8.48.

Propiophenone O-benzyl oxime, colourless oil, 84% yield. IR (neat, cm^{-1}): ν 3060, 3031, 2974, 2936, 2876, 1606, 1571, 1496, 1453, 1366, 1306, 1022, 959, 873, 768, 740, 695. ^1H NMR ($CDCl_3$): δ 1.14 (t, *J*=7.5 Hz, 3H), 2.79 (q, *J*=7.5 Hz, 2H), 5.23 (s, 2H), 7.28-7.64 (m, 10H). Anal. Calcd. for $C_{16}H_{17}NO$: C, 80.30; H, 7.16; N, 5.85. Found: C, 80.08; H, 7.12, N, 5.68.

Propiophenone O-n-butyl oxime, colourless oil, 82% yield. IR (neat, cm^{-1}): ν 3059, 2960, 2935, 2874, 1497, 1464, 1376, 1071, 1031, 965, 916, 886, 768, 694. ^1H NMR ($CDCl_3$): δ 0.96 (t, *J*=7.2 Hz, 3H), 1.14 (t, *J*=7.5 Hz, 3H), 1.44 (m, *J*=7.5 Hz, 2H), 1.71 (m, *J*=7.5 Hz, 2H), 2.75(m, *J*=7.5 Hz, 2H), 4.18 (t, *J*=6.6 Hz, 2H). Anal. Calcd. for $C_{13}H_{19}NO$: C, 76.06, H, 9.33, N, 6.82. Found: C, 75.84; H, 9.18; N, 6.75.

1-(4-Nitrophenyl)ethanone O-methyl oxime, light brown solid, 81% yield, mp 108-109 °C (lit.[4] mp 110-112 °C). IR (KBr, cm^{-1}): ν 3109, 3055, 3007, 2975, 2941, 2823, 1616, 1599, 1585, 1513, 1346, 1052, 904, 854, 752, 690. ^1H NMR ($CDCl_3$): δ 2.26 (s, 3H), 4.05 (s, 3H), 7.82 (d, *J*=8.7 Hz, 2H), 8.22 (d, *J*=8.7 Hz, 2H). Anal. Calcd. for $C_9H_{10}N_2O_3$: C, 55.67; H, 5.19; N, 14.43. Found: C, 55.45; H, 5.16; N, 14.28.

1-(4-Nitrophenyl)ethanone O-benzyl oxime, light yellow solid, 78% yield, mp 79-80 °C. IR (KBr, cm^{-1}): ν 3086, 3053, 3031, 2948, 2883, 1596, 1581, 1515, 1344, 1020, 942, 854, 753, 699, 687. ^1H NMR ($CDCl_3$): δ 2.30 (s, 3H), 5.28 (s, 2H), 7.33-7.41 (m, 5H), 7.82 (d, *J*=9.0 Hz, 2H), 8.21 (d, *J*=9.0 Hz, 2H). Anal. Calcd. for $C_{15}H_{14}N_2O_3$: C, 66.66; H, 5.22; N, 10.36. Found: C, 66.42; H, 5.15; N, 10.22.

1-(4-Nitrophenyl)ethanone O-*n*-butyl oxime, light brown oil, 75% yield. IR (neat, cm^{-1}): ν 3081, 2960, 2934, 2873, 1958, 1584, 1519, 1461, 1346, 1112, 1066, 1031, 938, 906, 855, 775, 753, 693. ^1H NMR ($CDCl_3$): δ 0.97 (t, *J*=7.2 Hz, 3H), 1.42 (m, *J*=7.2 Hz, 2H), 1.73 (m, *J*=7.2 Hz, 2H), 2.26 (s, 3H), 4.25 (t, J=7.2 Hz, 2H), 7.82 (d, *J*=9.0 Hz, 2H), 8.21 (d, J= 9.0 Hz, 2H). Anal. Calcd. for $C_{12}H_{16}N_2O_3$: C, 61.00; H, 6.83; N, 11.86. Found: C, 60.82; H, 6.58; N, 11.72.

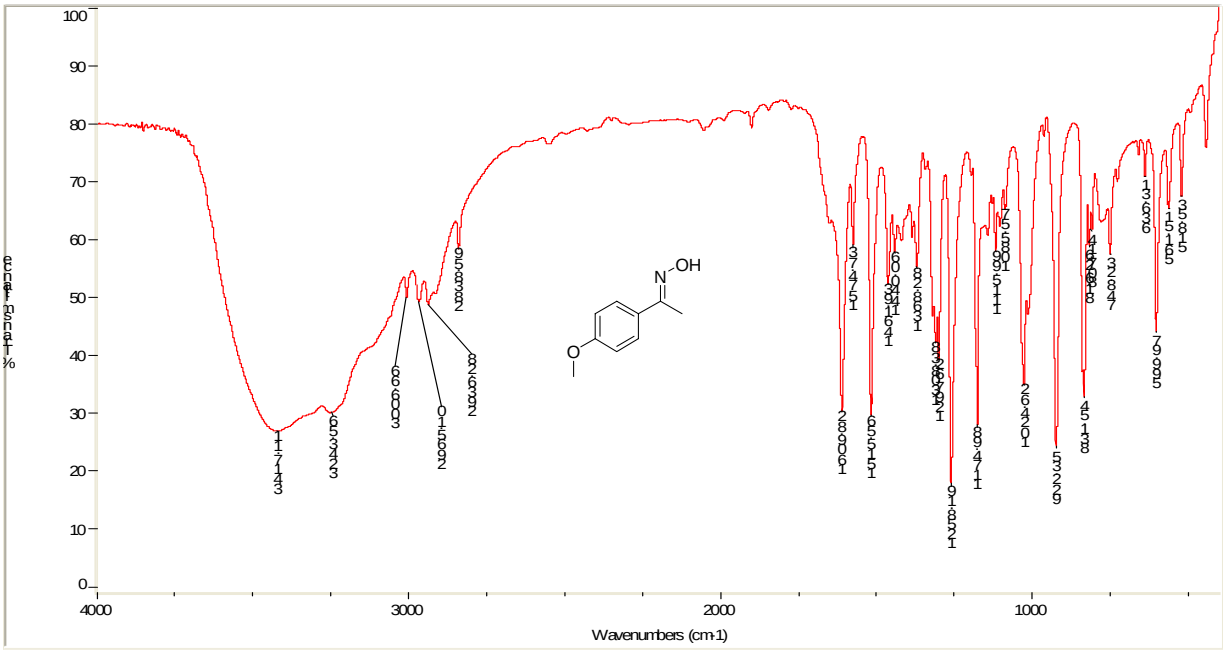
References:

1 Ingersoll, A. W. *Organic Syntheses*, **1943**, Coll. Vol. *2*, 503.

2 (a) Coustard, J. M.; Jacquesy, J. C.; Violeau, B. *Tetrahedron Lett.* **1992**, *33*, 8085. (b) Smith, J. H.; Kaiser, E. T. *J. Org. Chem.***1974**, *39*, 729.

3 Uno, T.; Gong, B.; Schultz, P. G. *J. Am. Chem. Soc.***1994**, *116*, 1145.

4 Johnson, J. E.; Silk, N. M.; Arfan, M. *J. Org. Chem.***1982**, *47*, 1958.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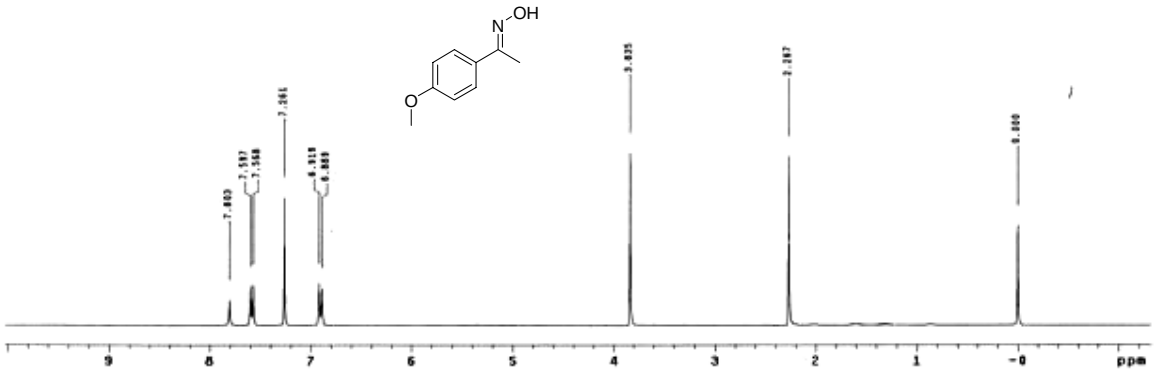
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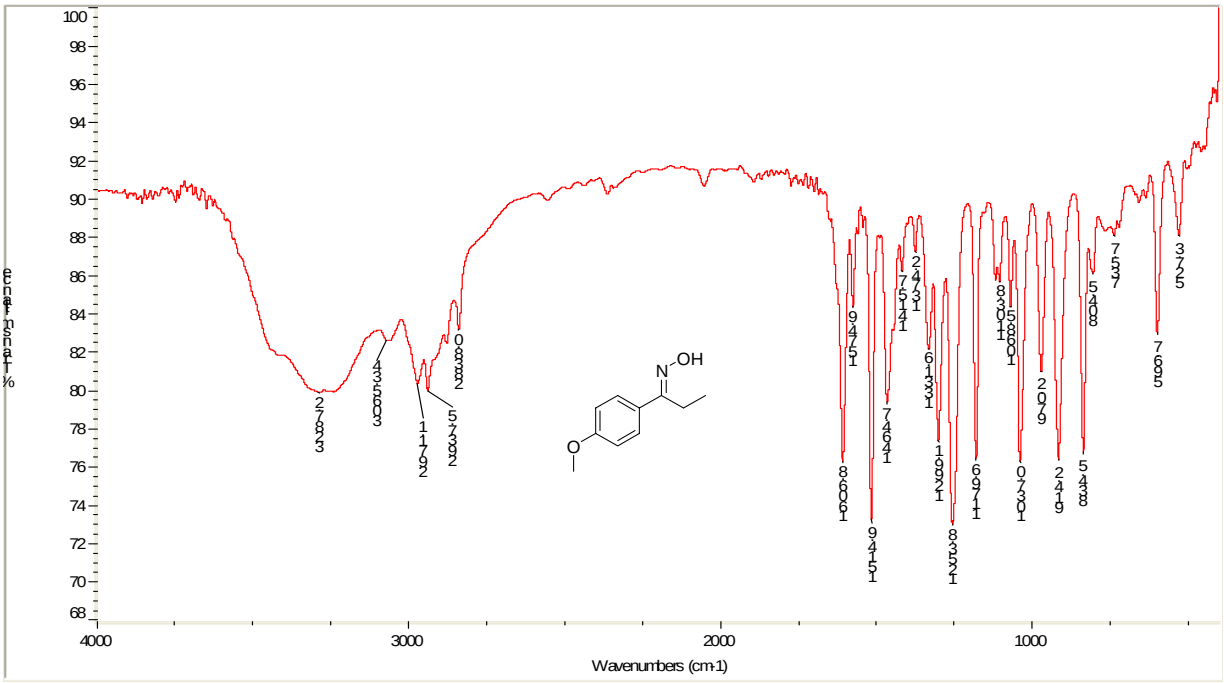
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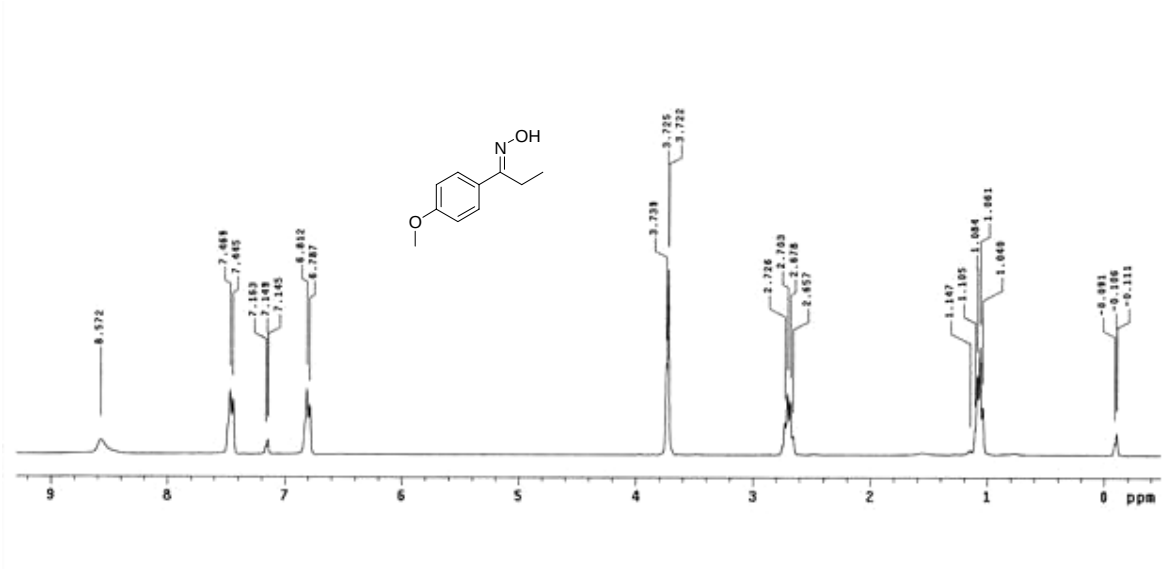
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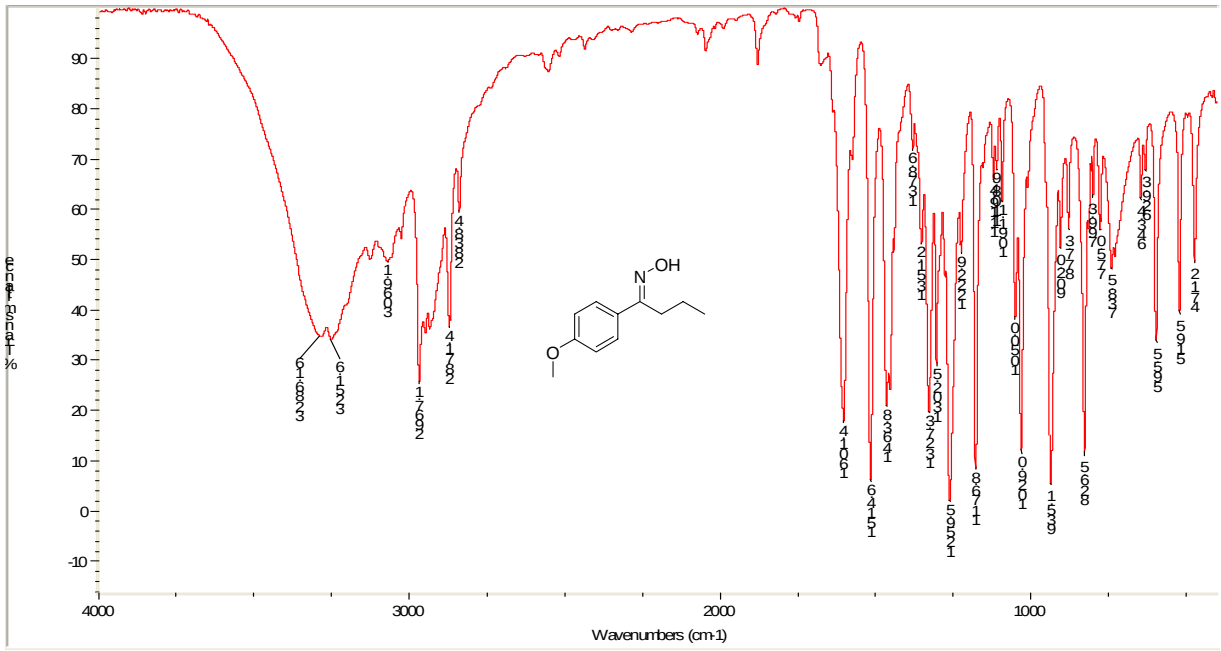
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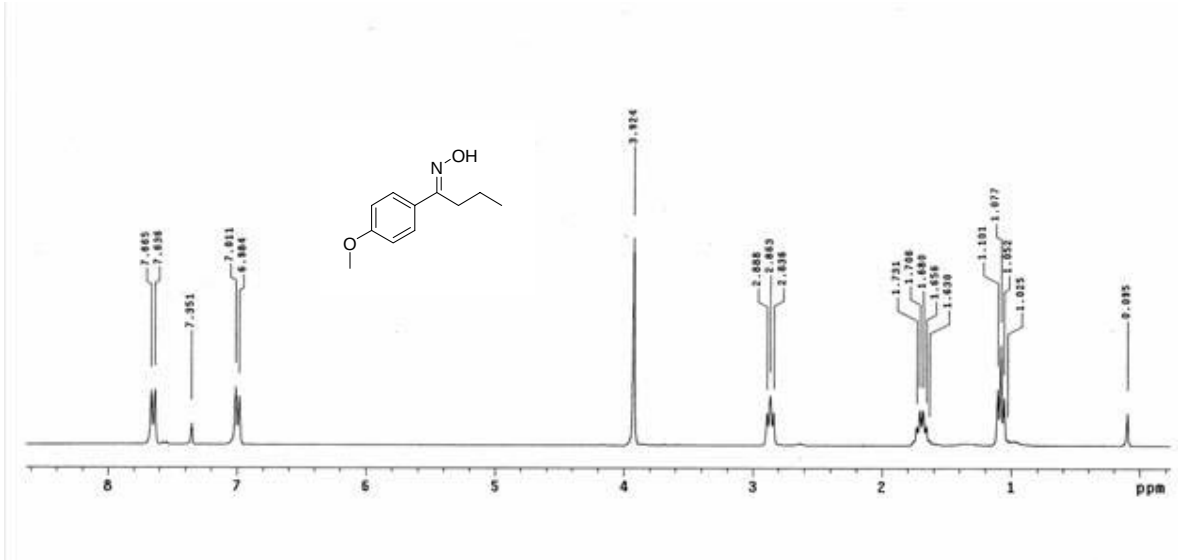
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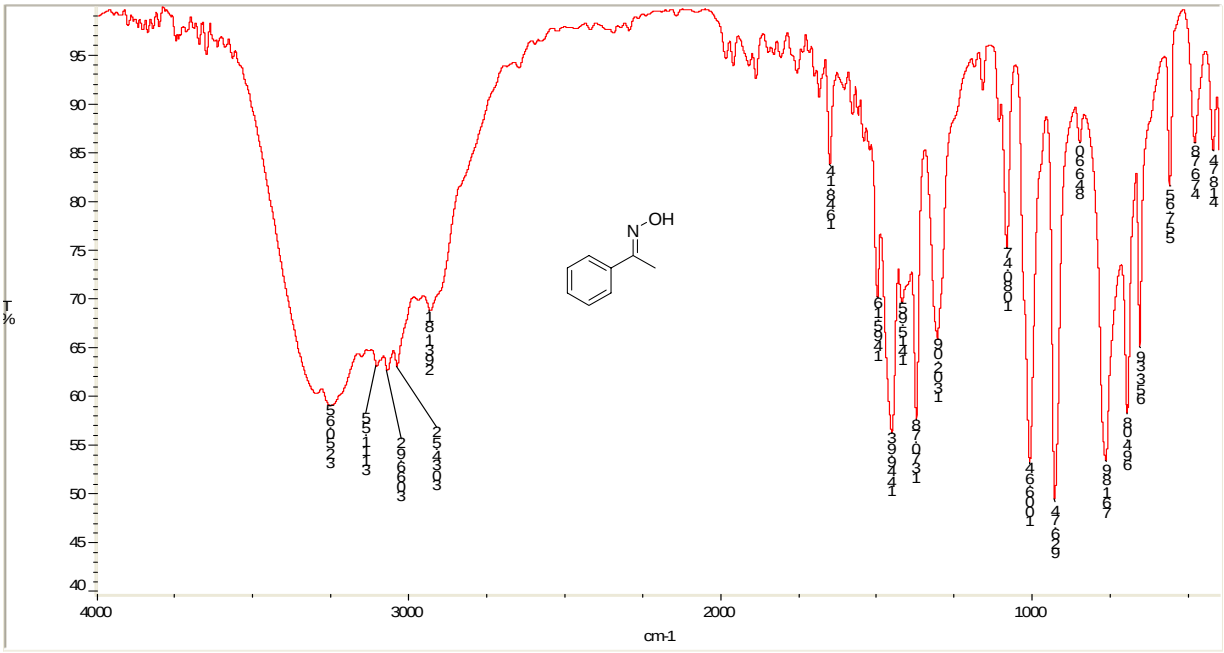
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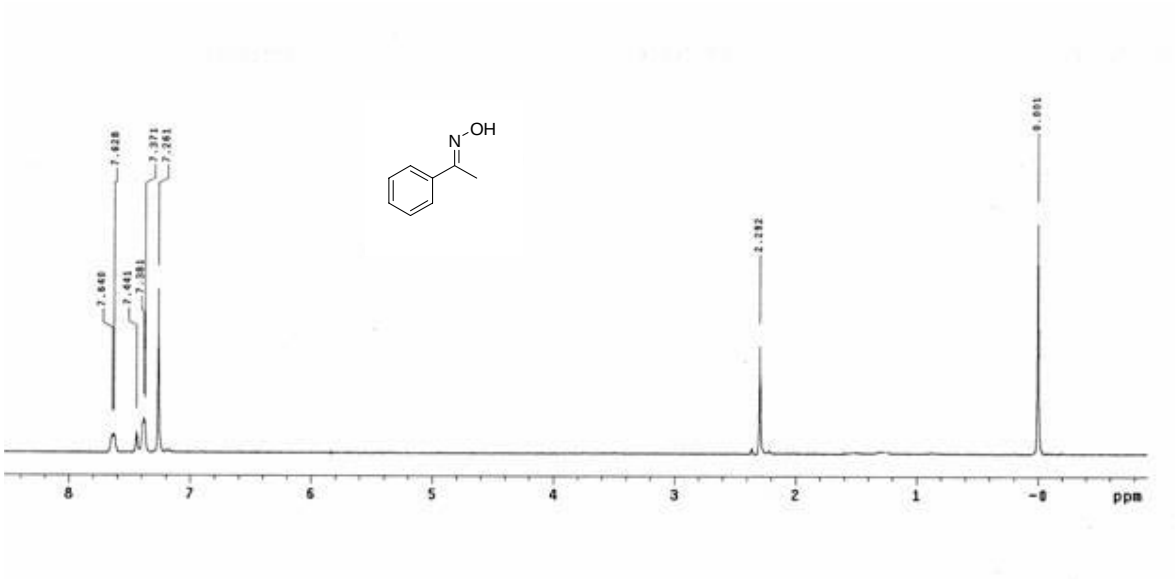
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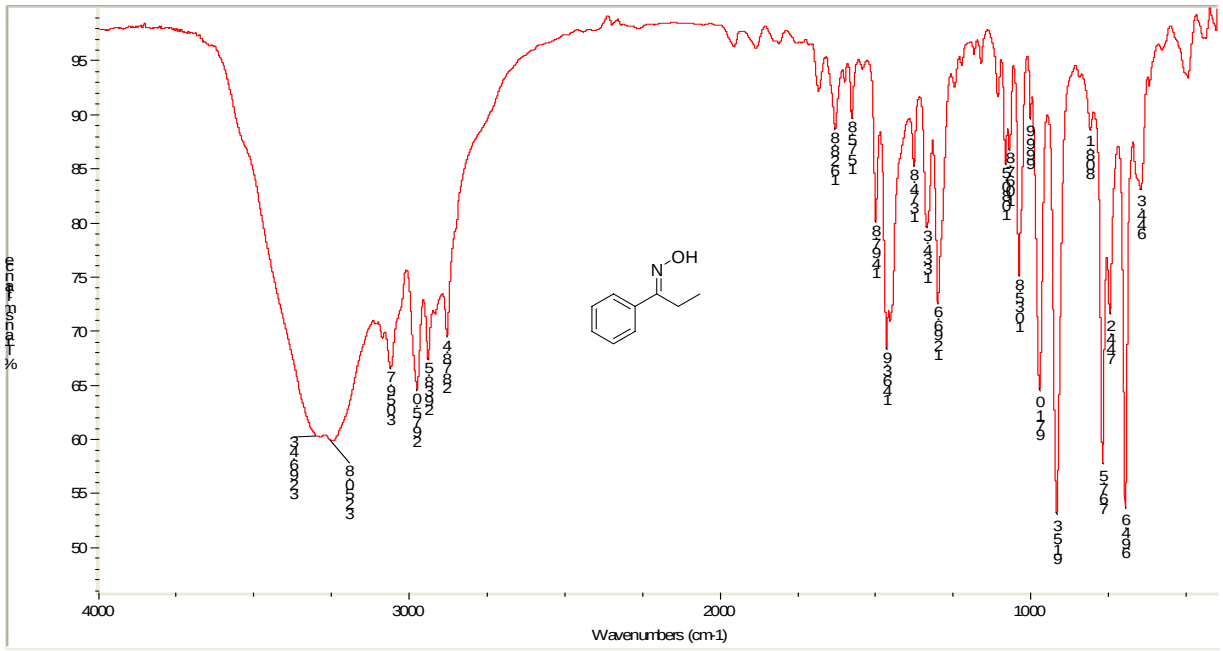
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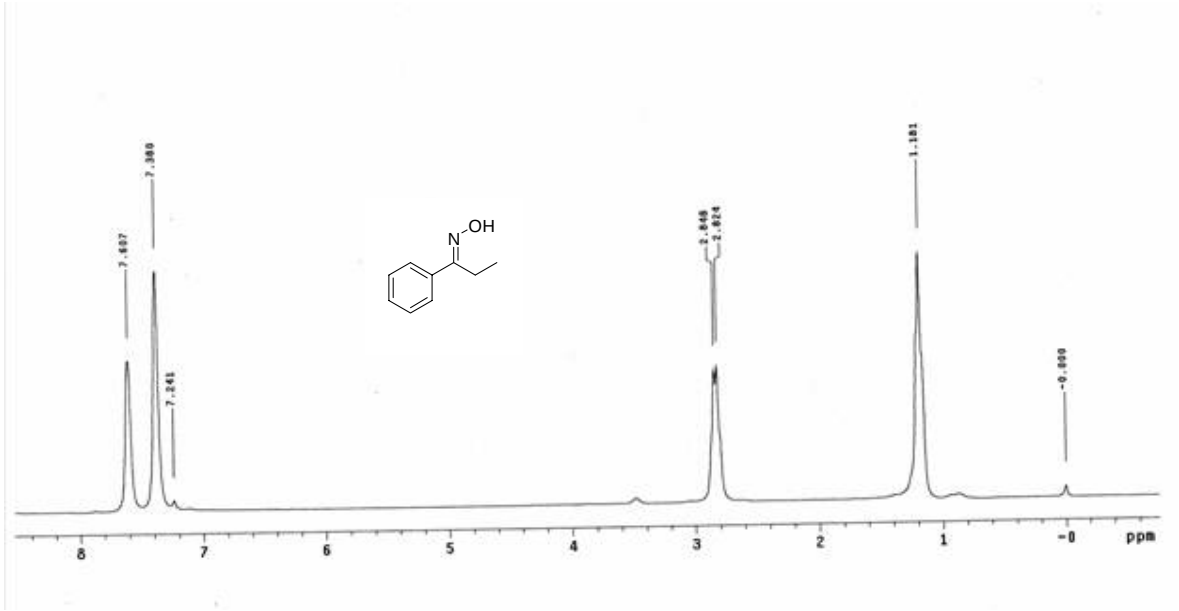
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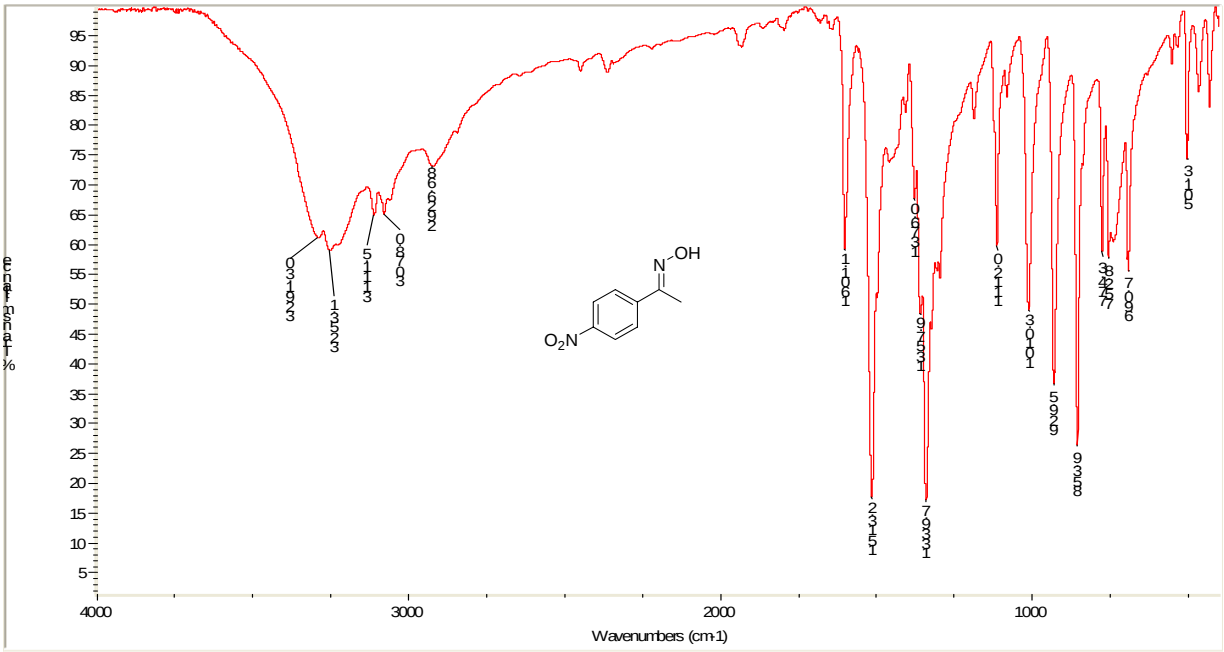
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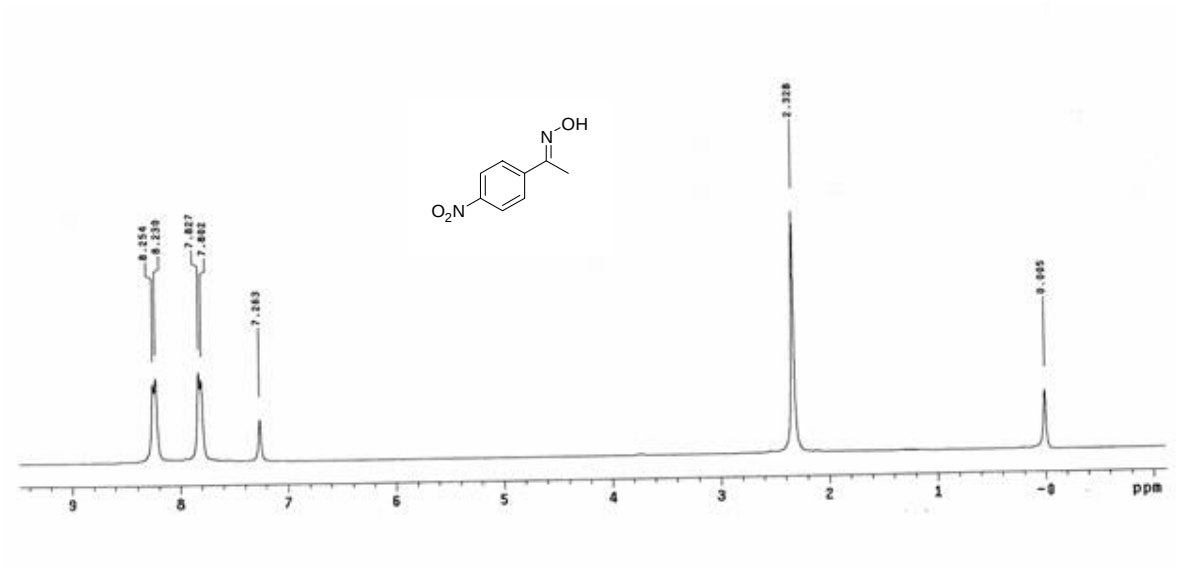
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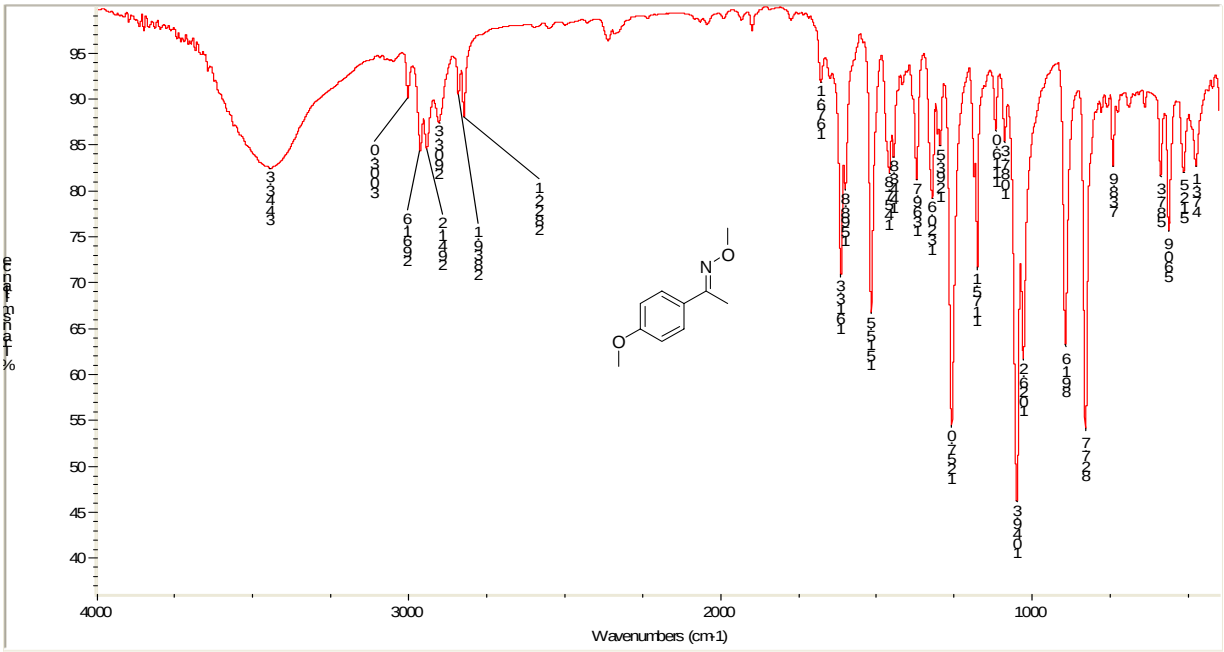
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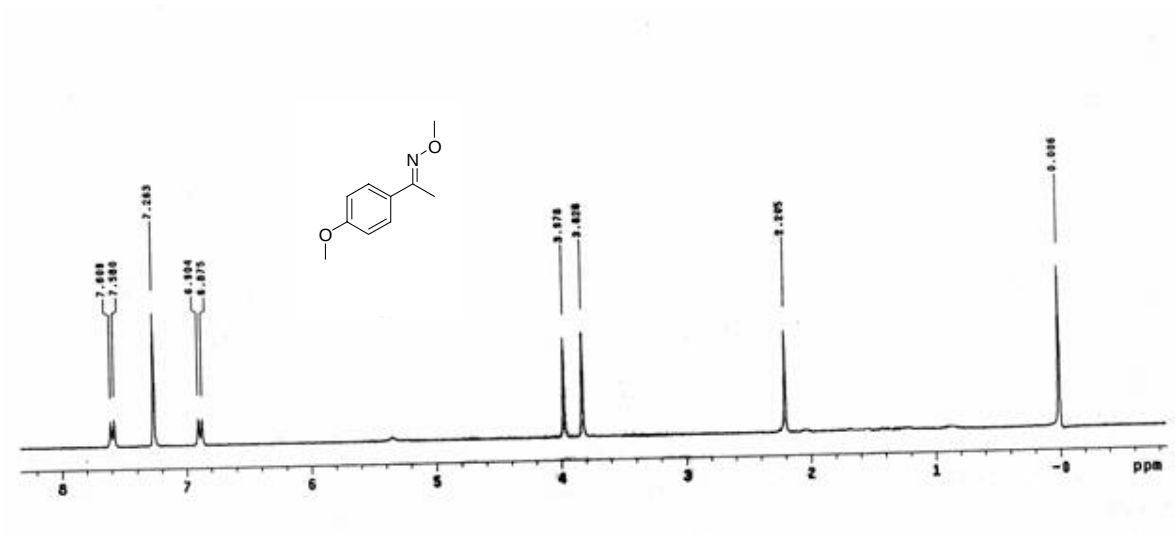
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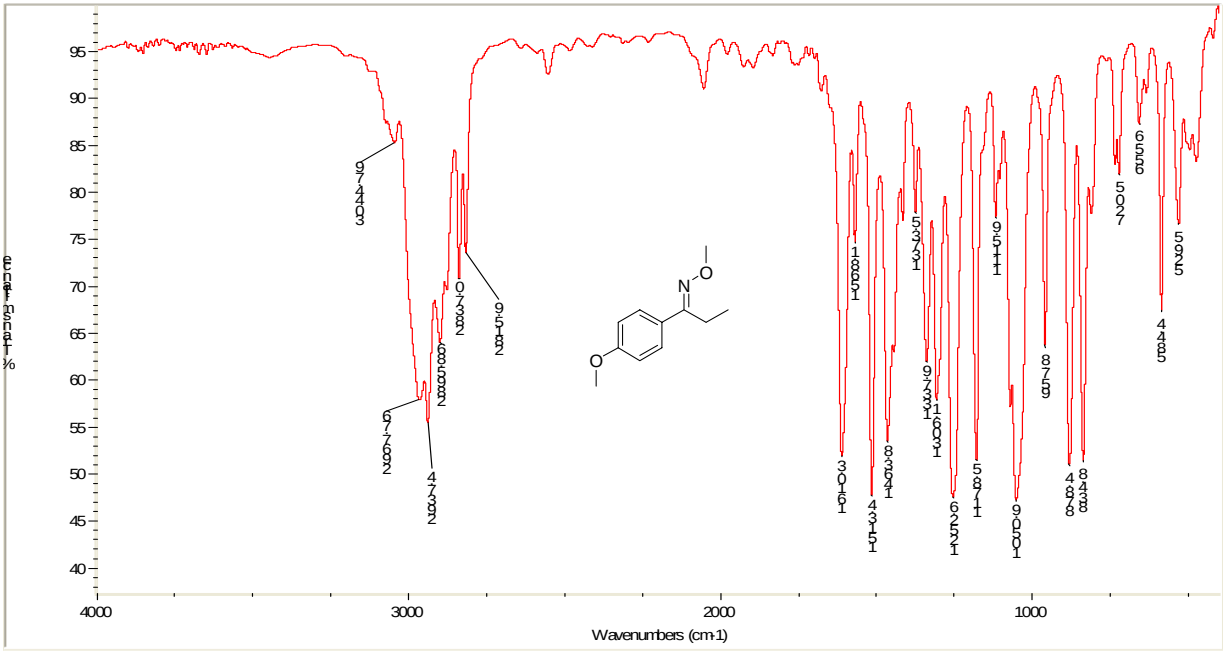
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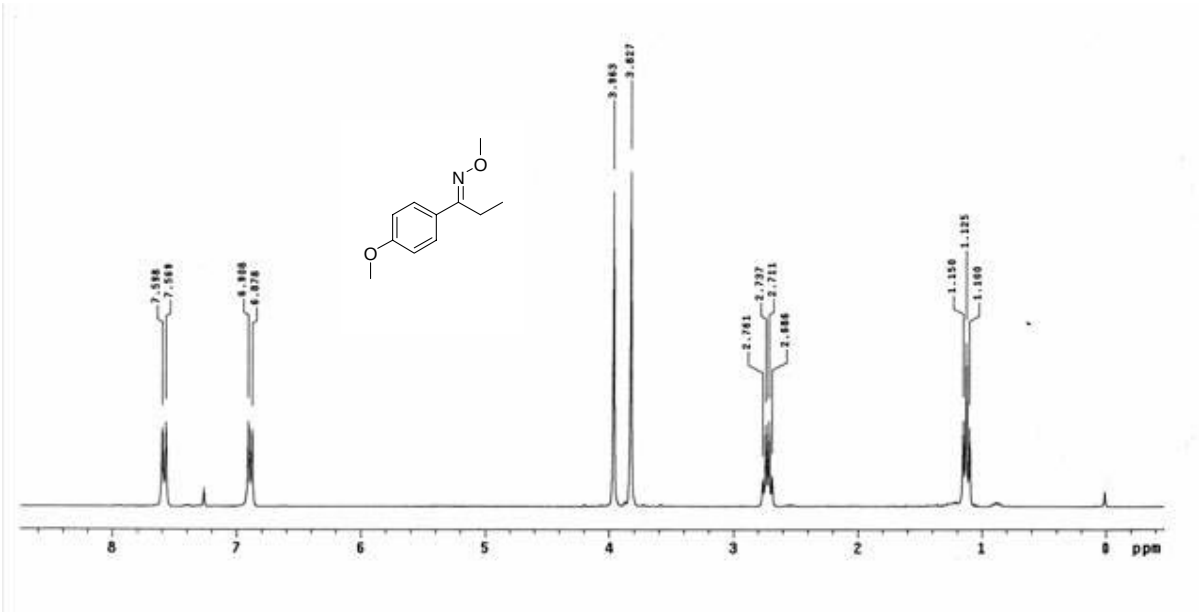
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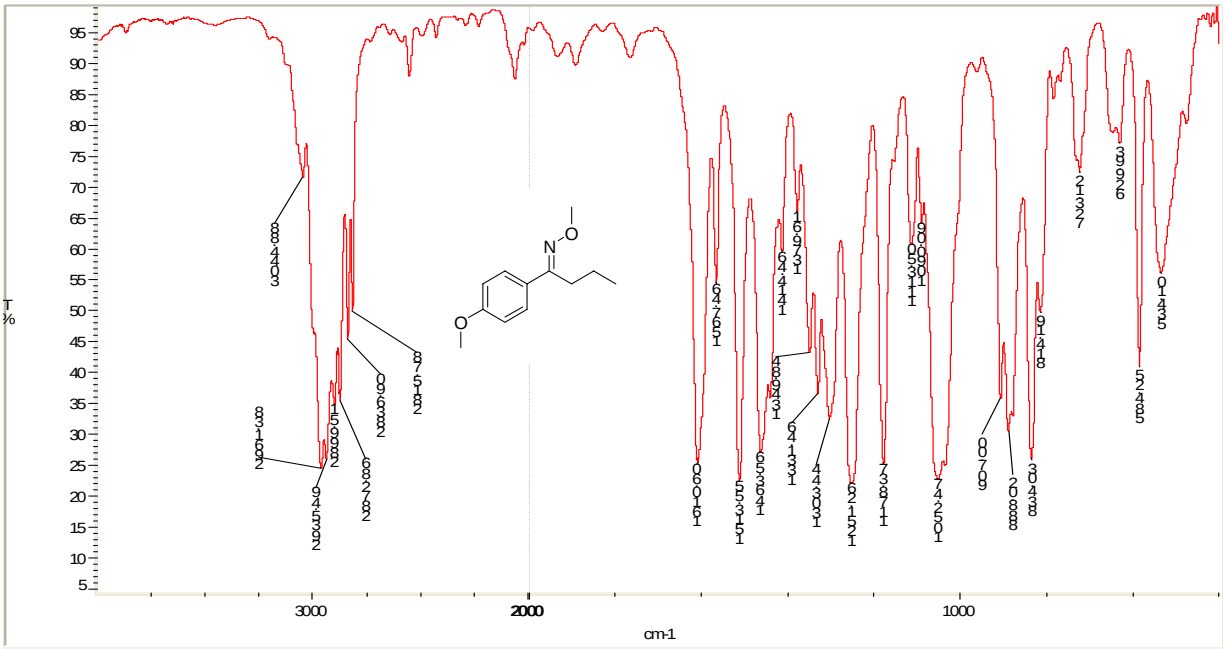
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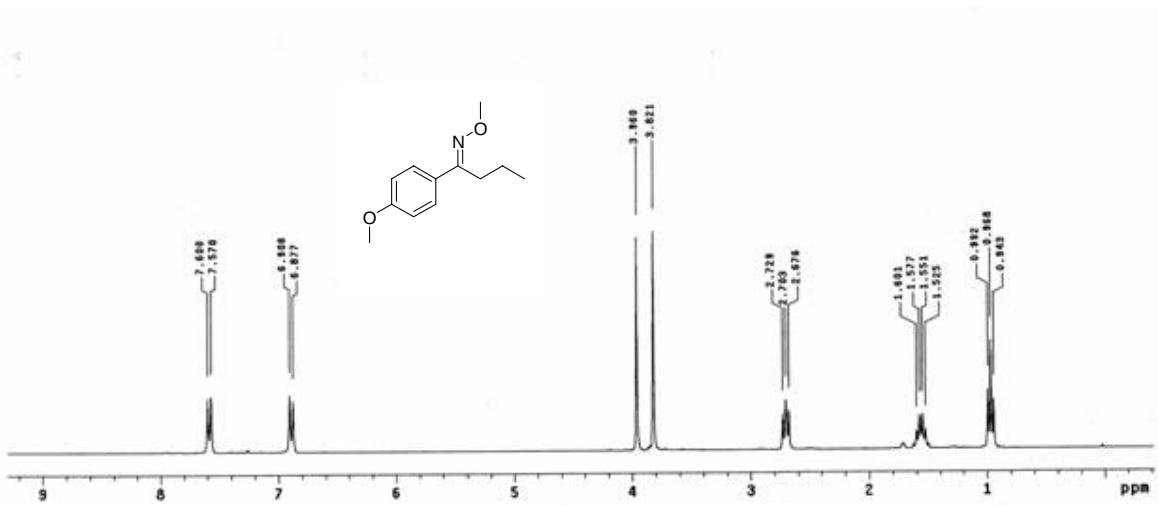
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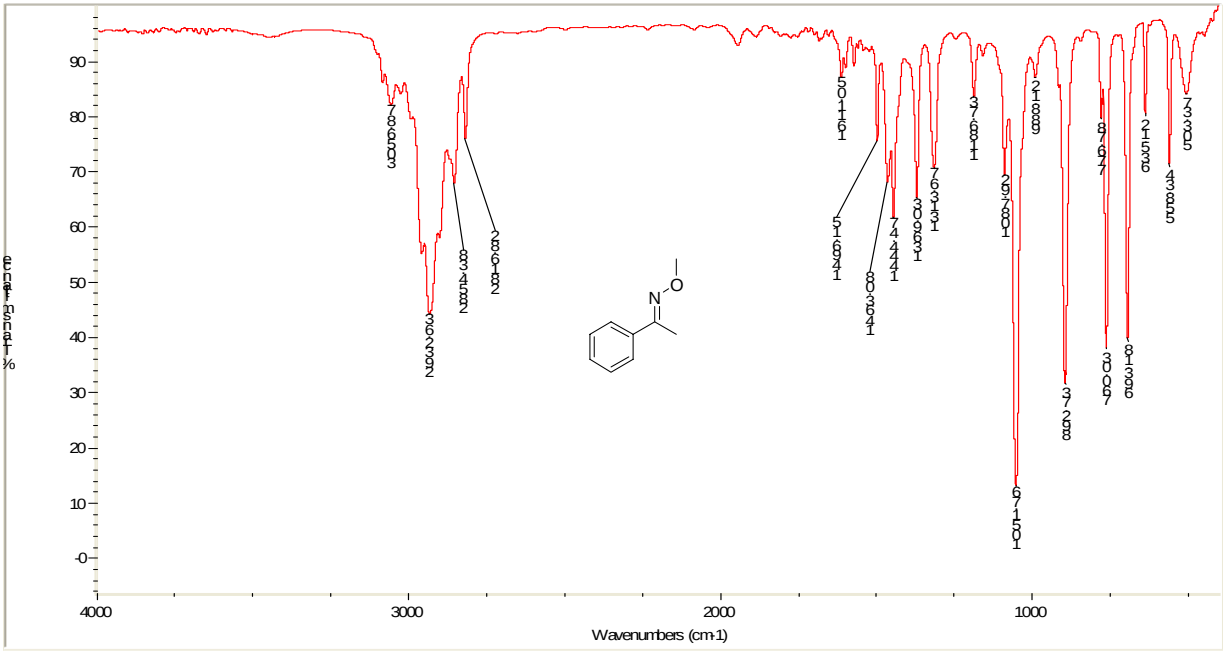
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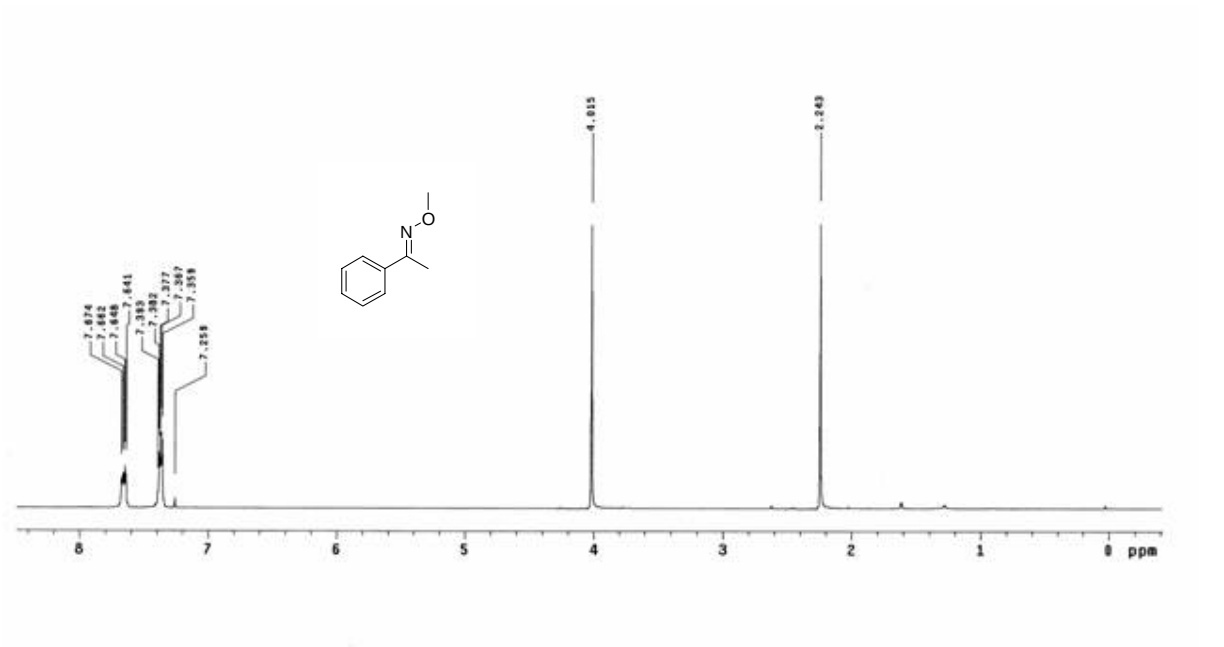
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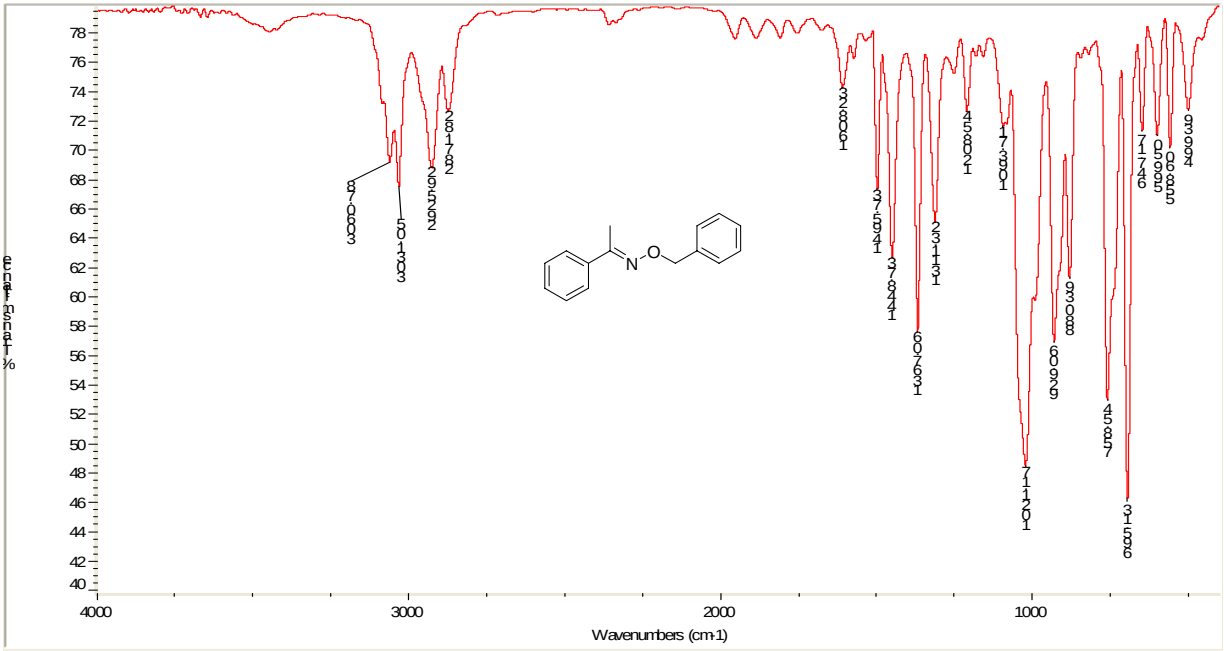
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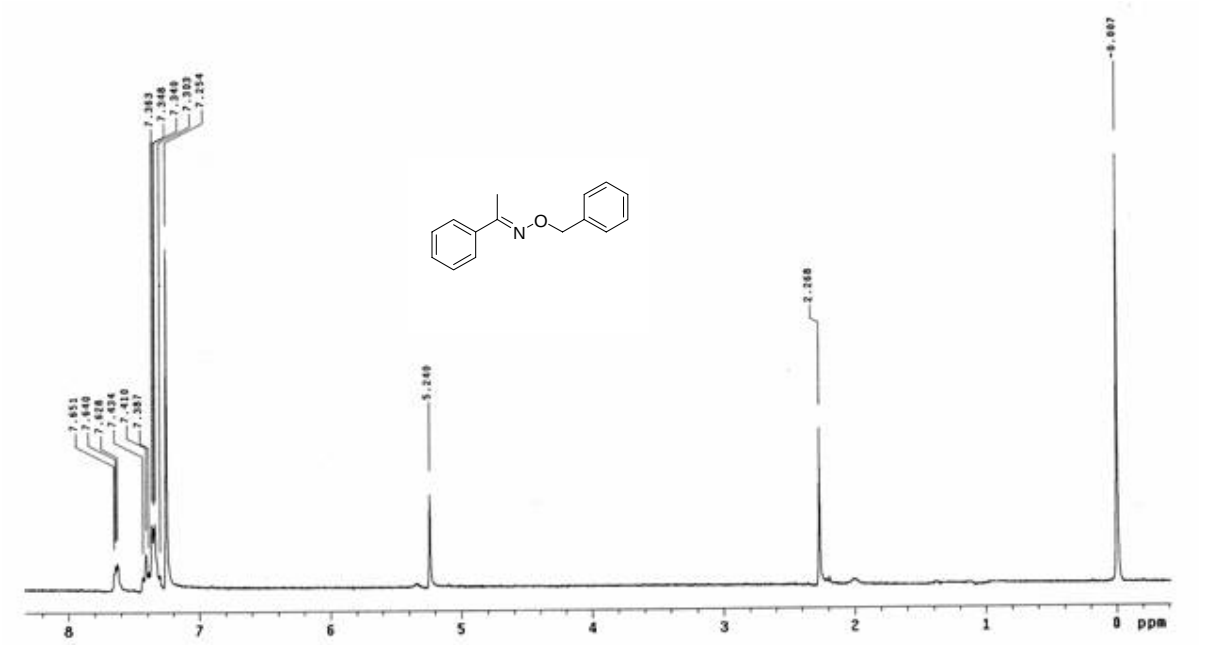
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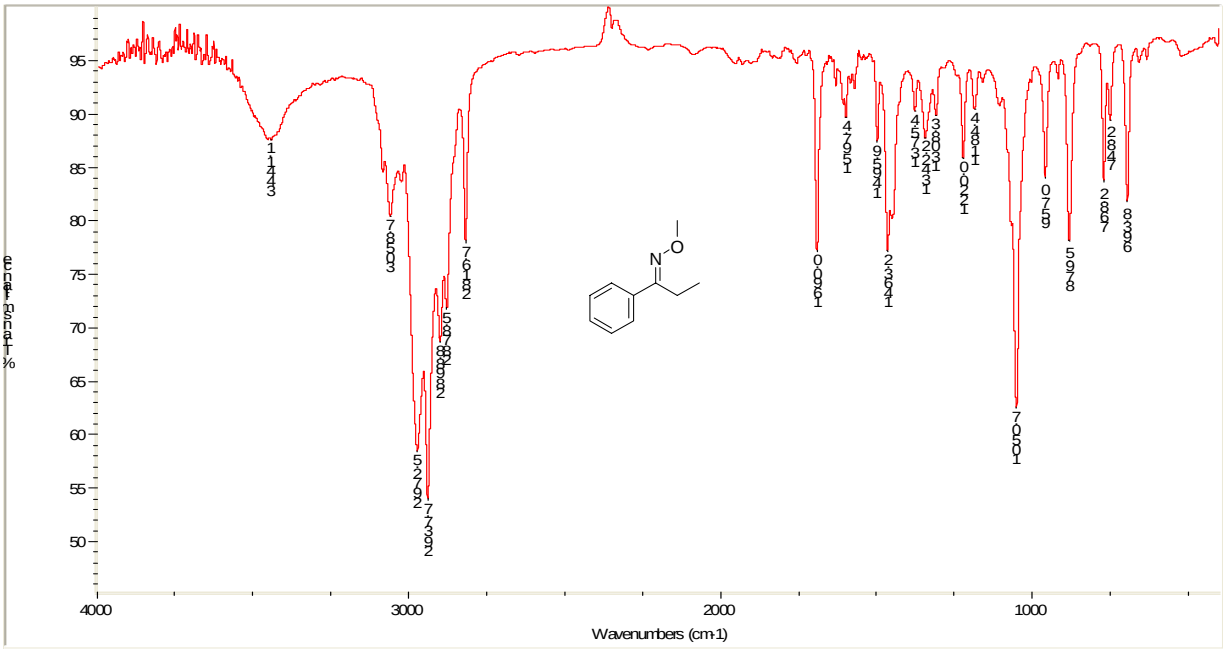
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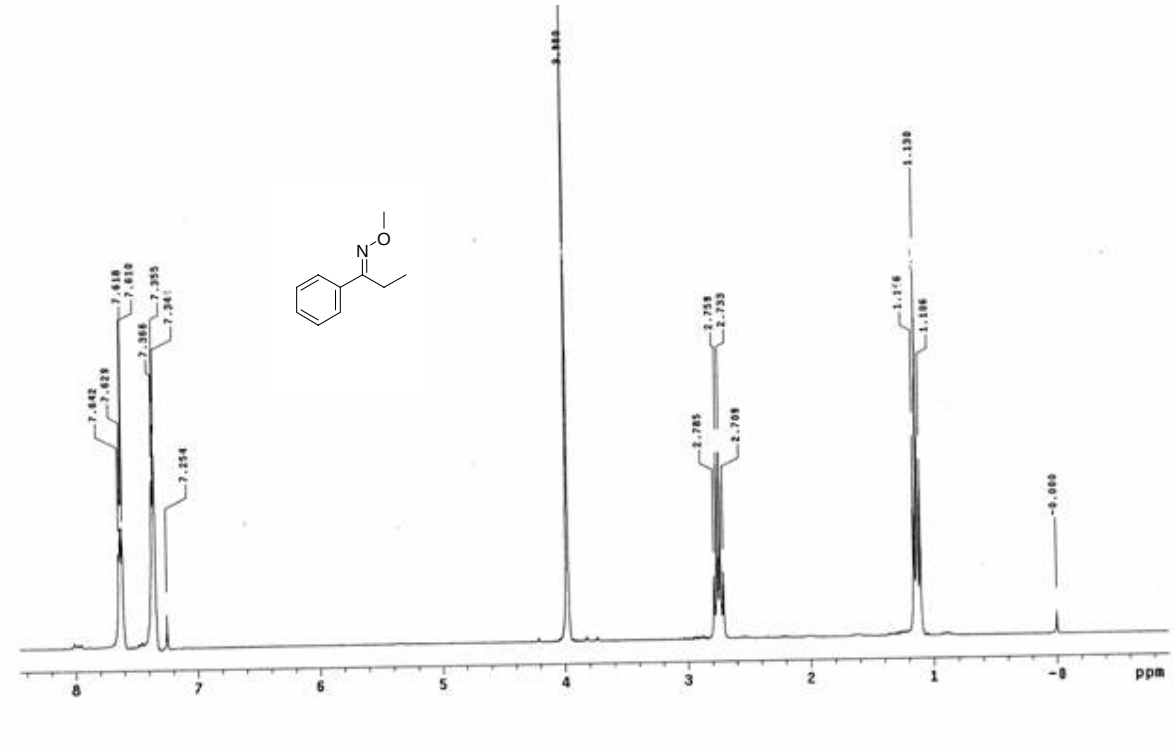
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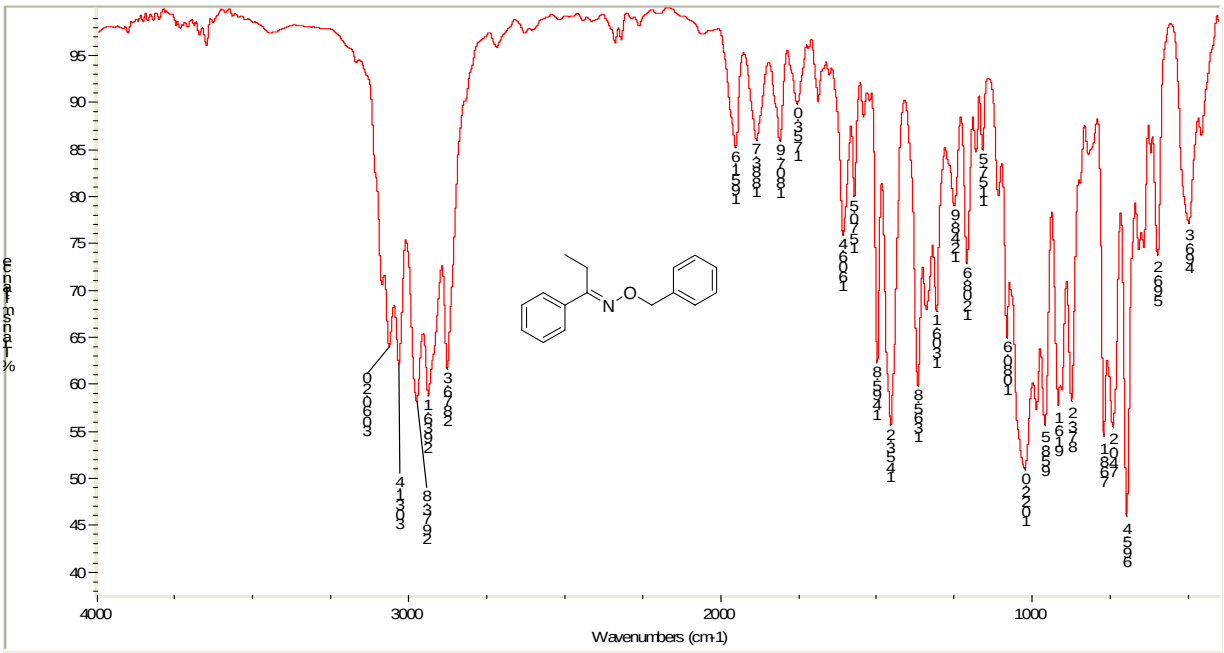
Wavenumbers (cm-1)
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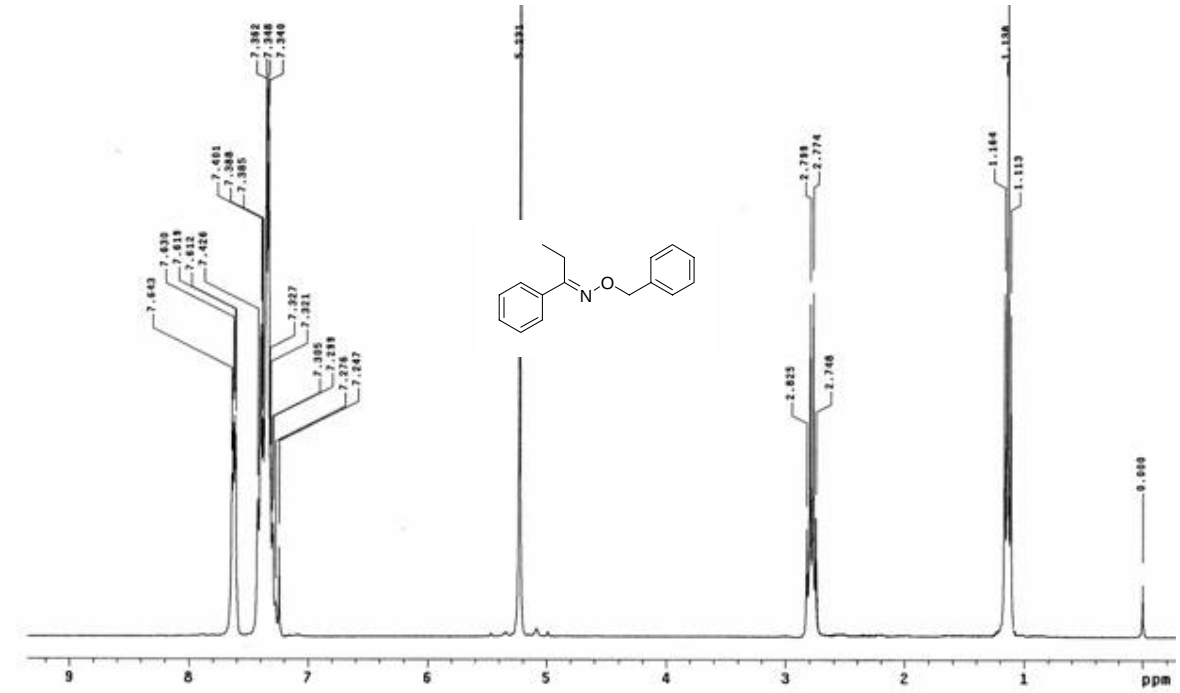
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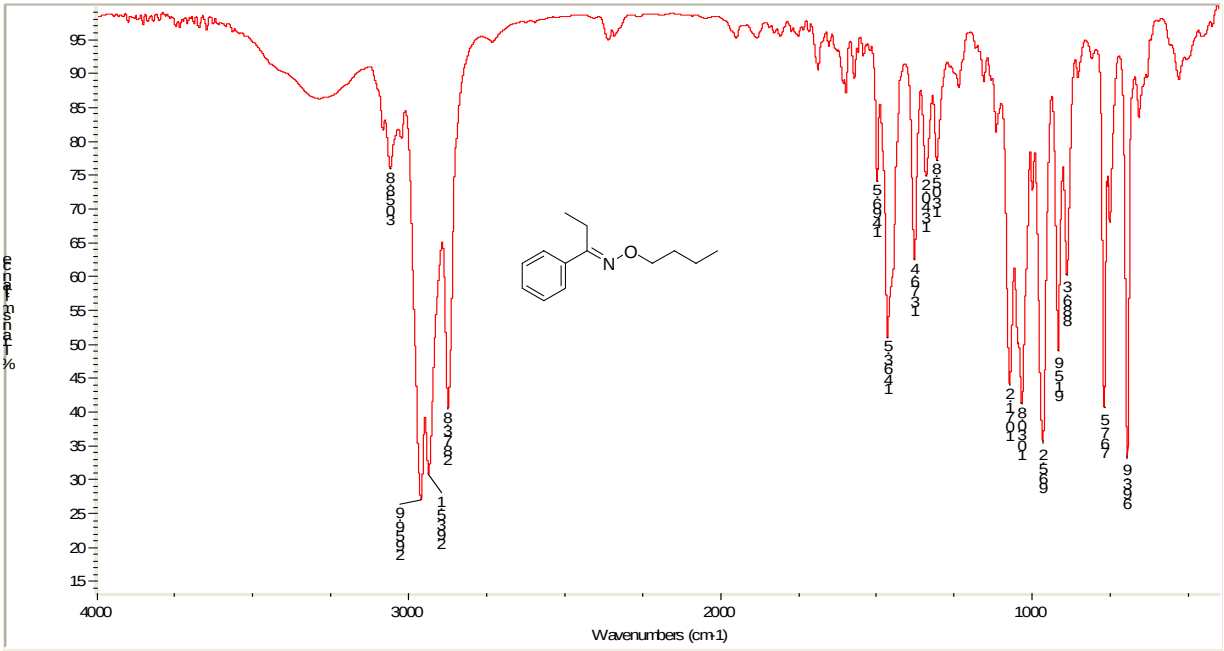
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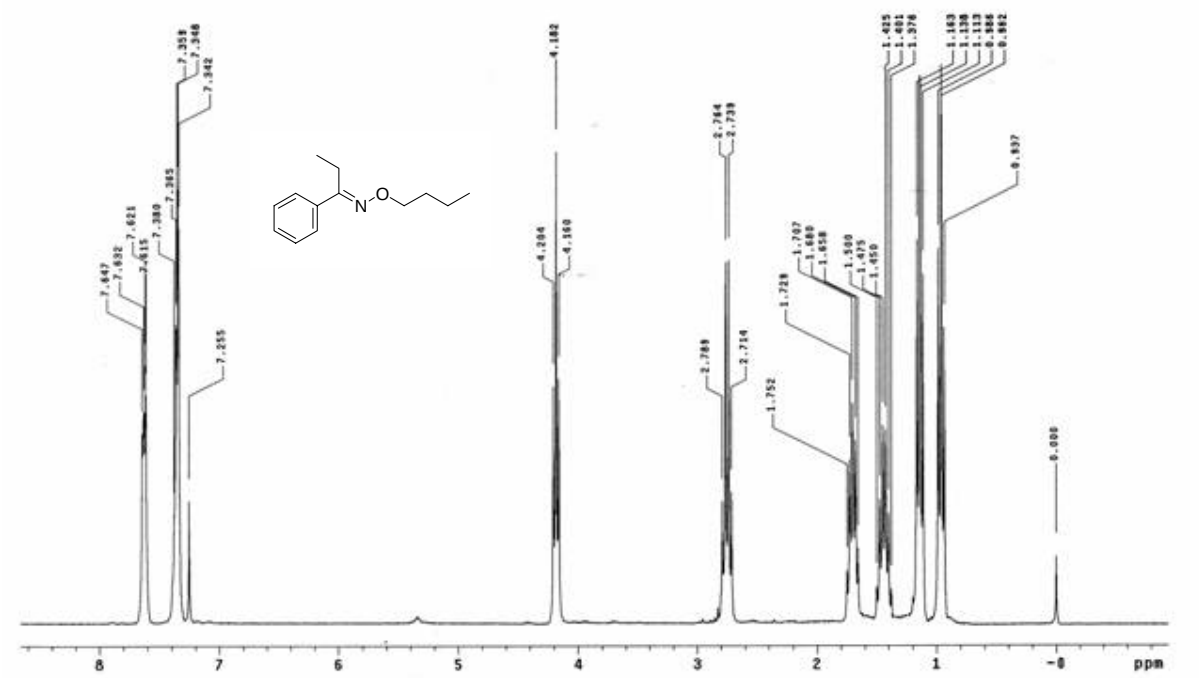
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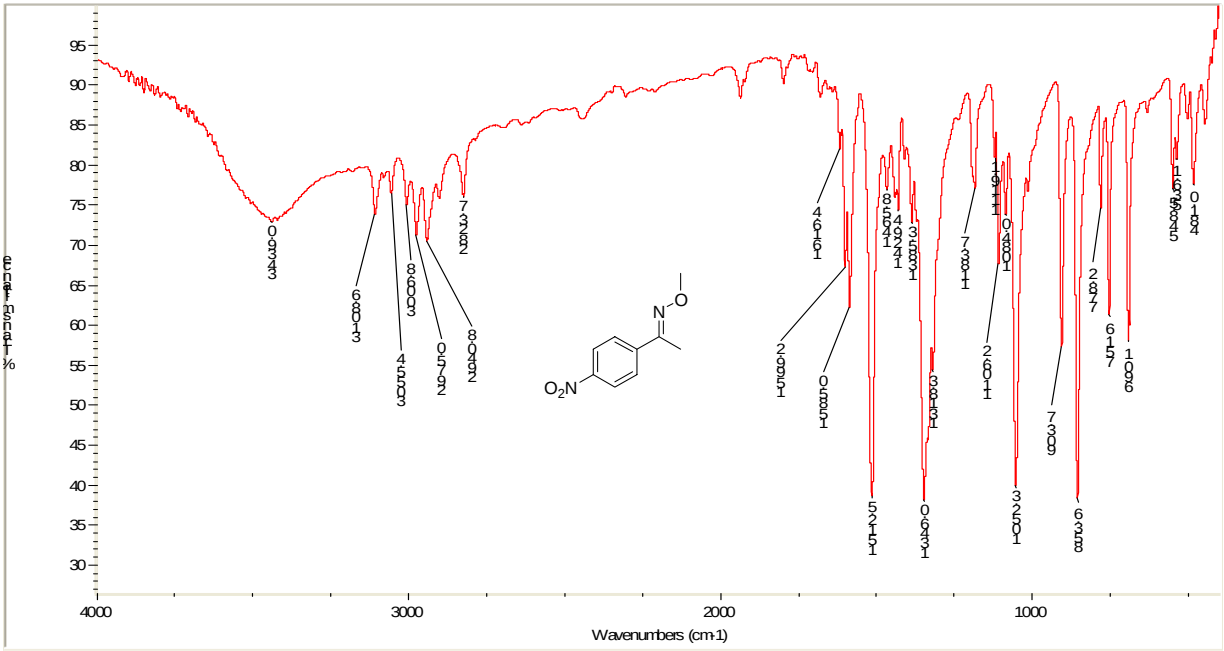
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45
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35
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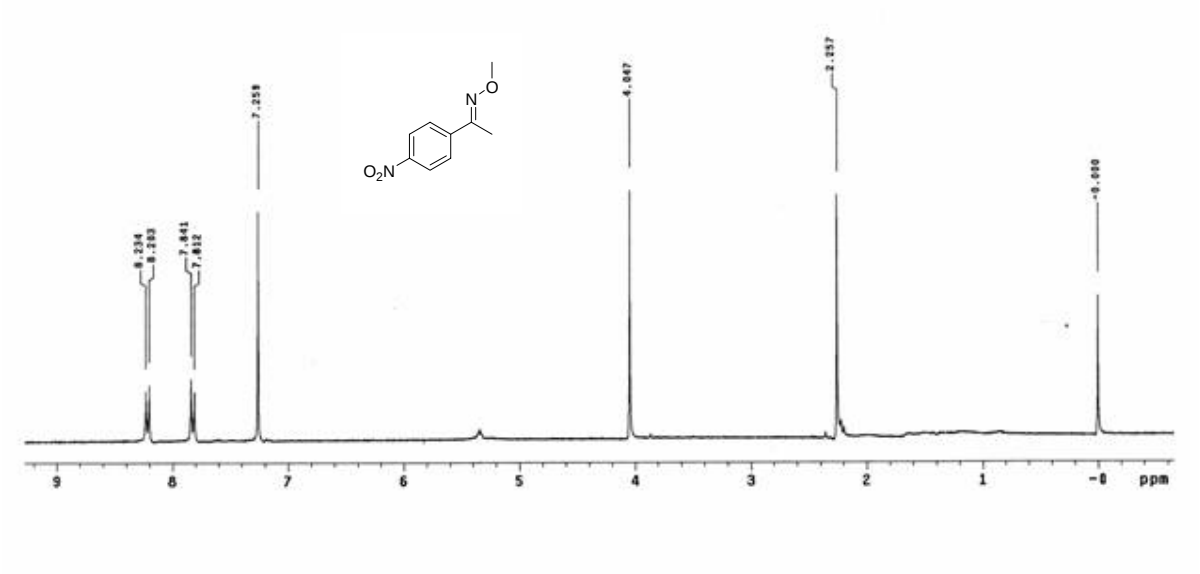
O_2N
N
O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7.412
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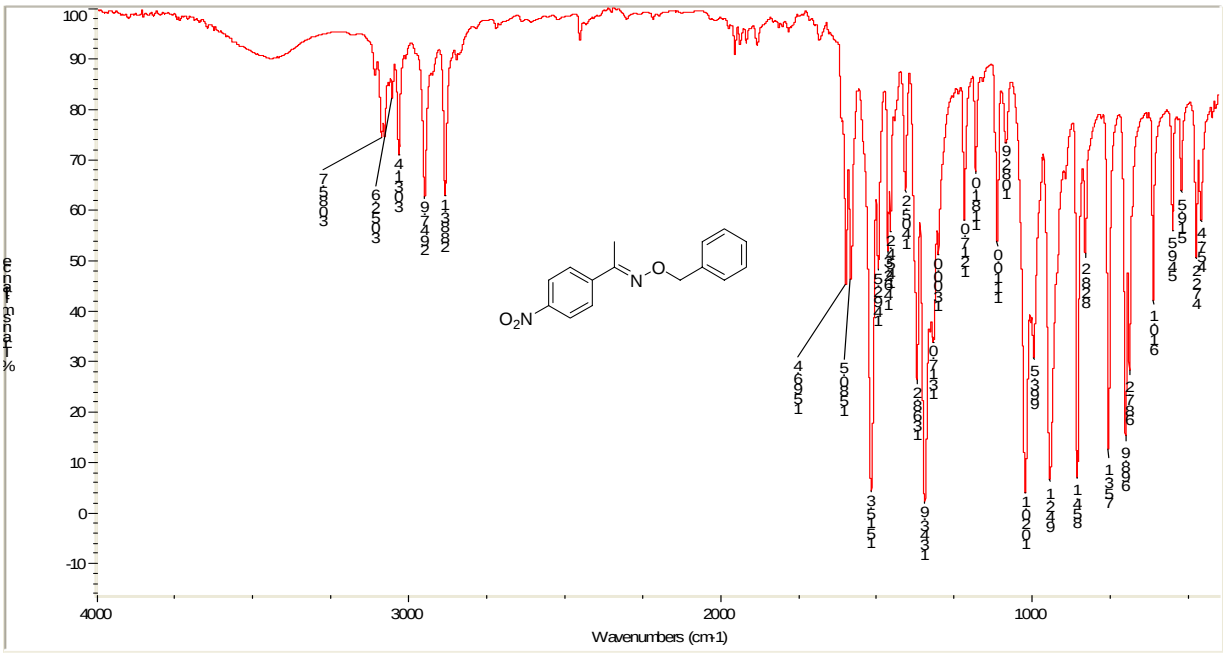
6
5
4
3
2
1
-0
ppm


Wavenumbers (cm-1)
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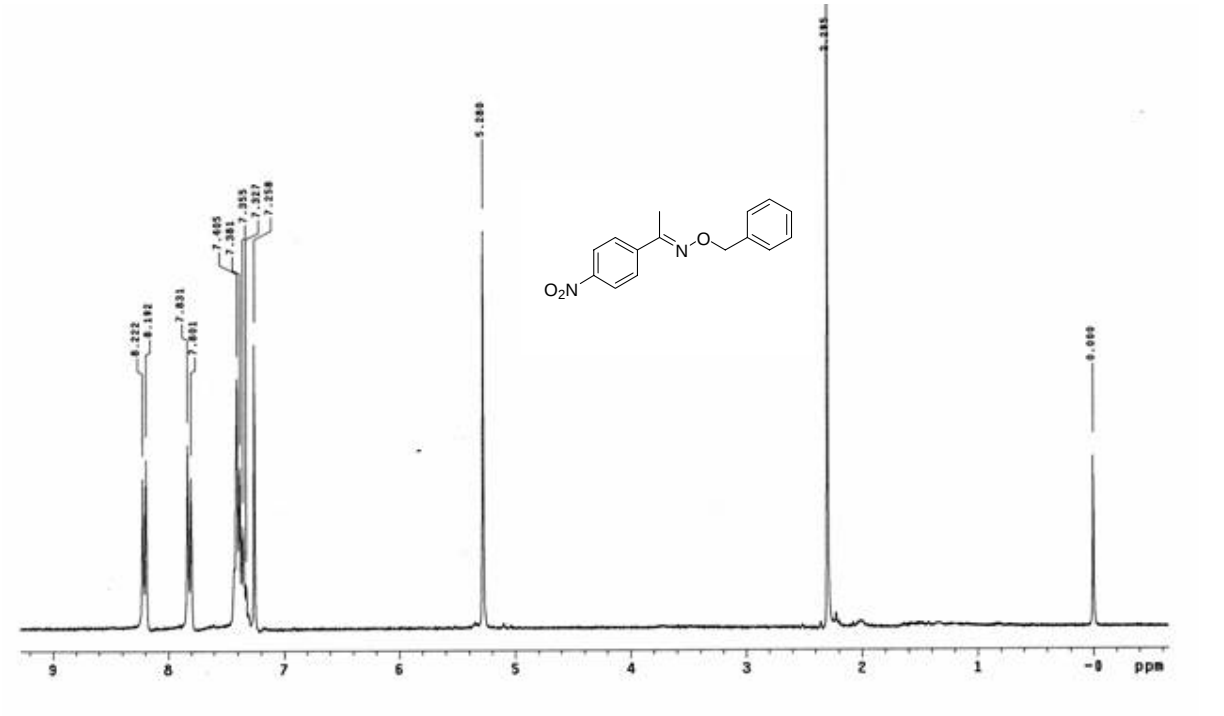
O2N
N
O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2
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O2N
N
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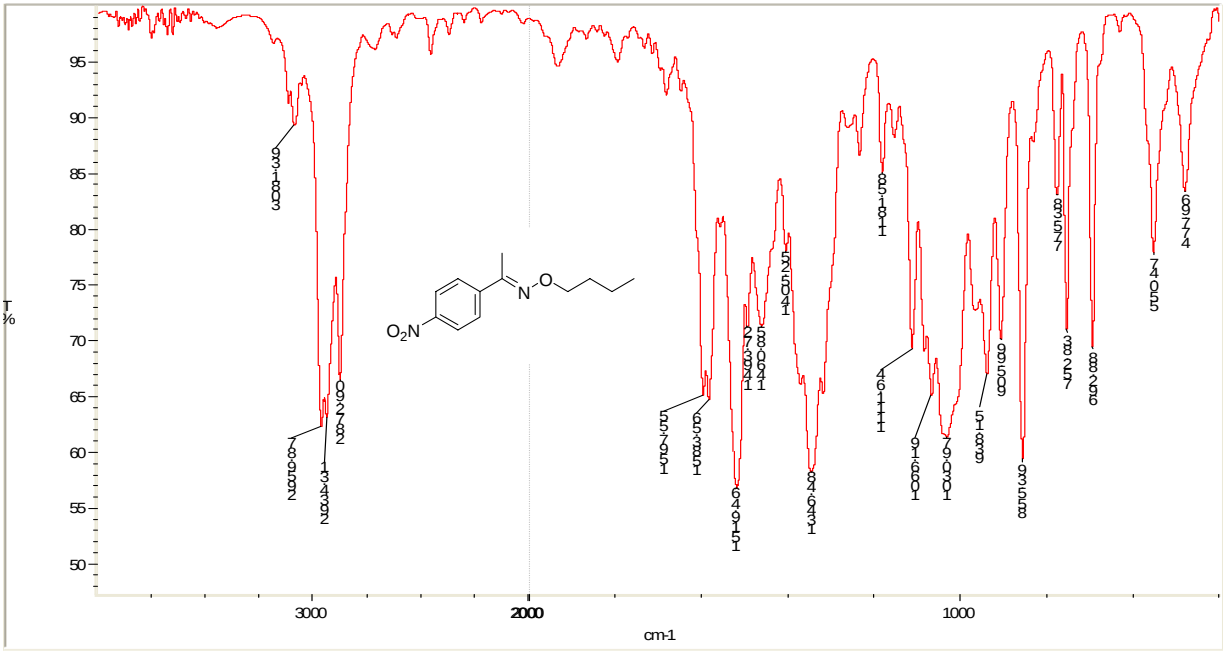
T
%
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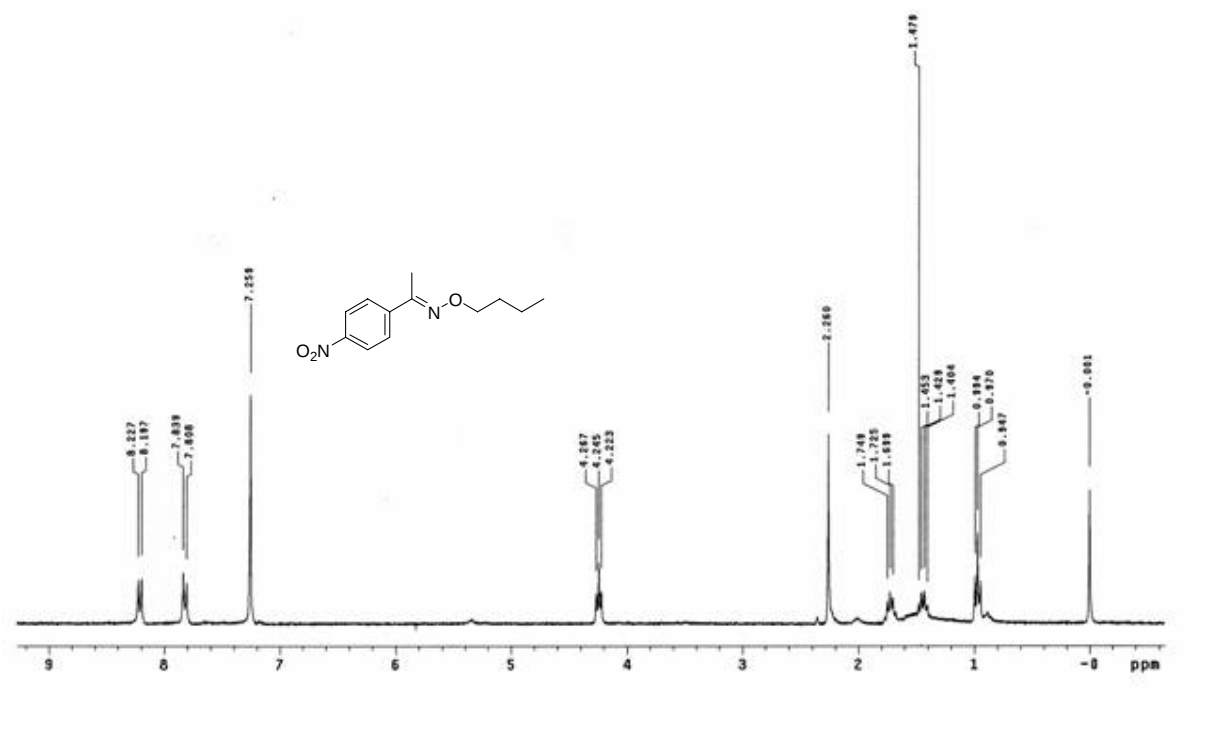
85
80
75
70
65
60
55
50
3000
2000
1000
cm-1
N
O
O_2N


N
O
O_2N
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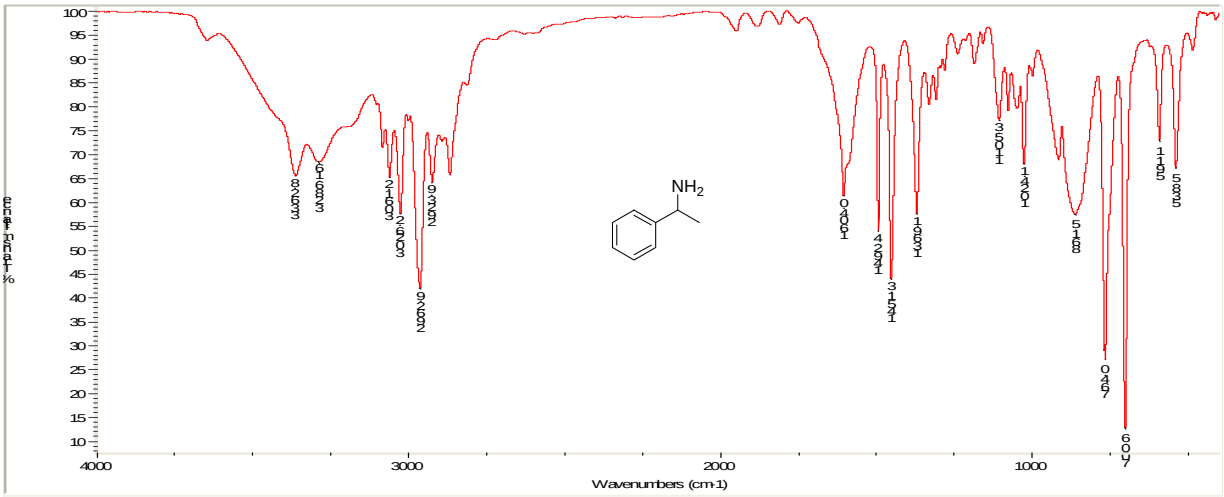
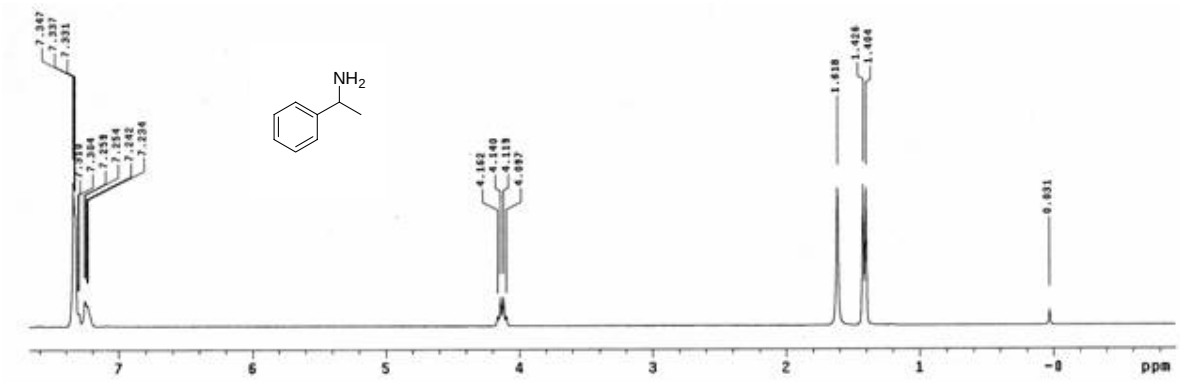

NH2
Wavenumbers (cm-1)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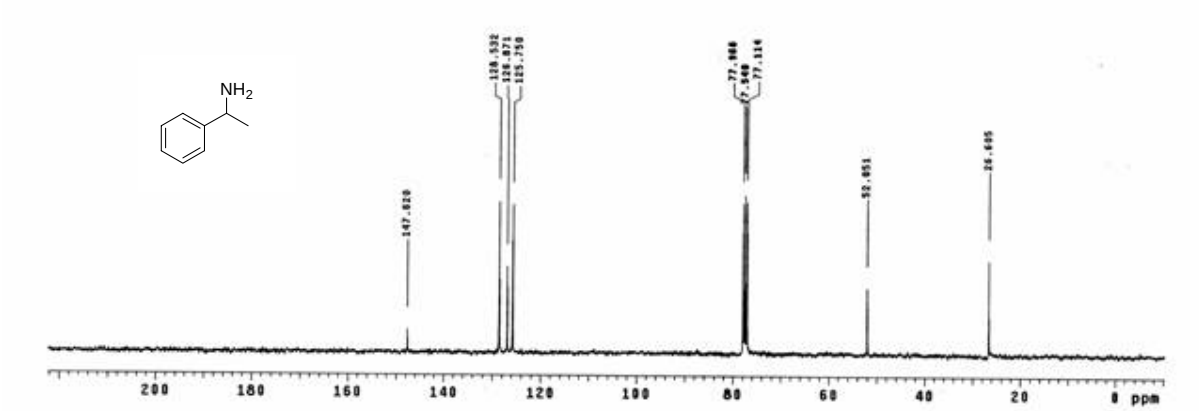
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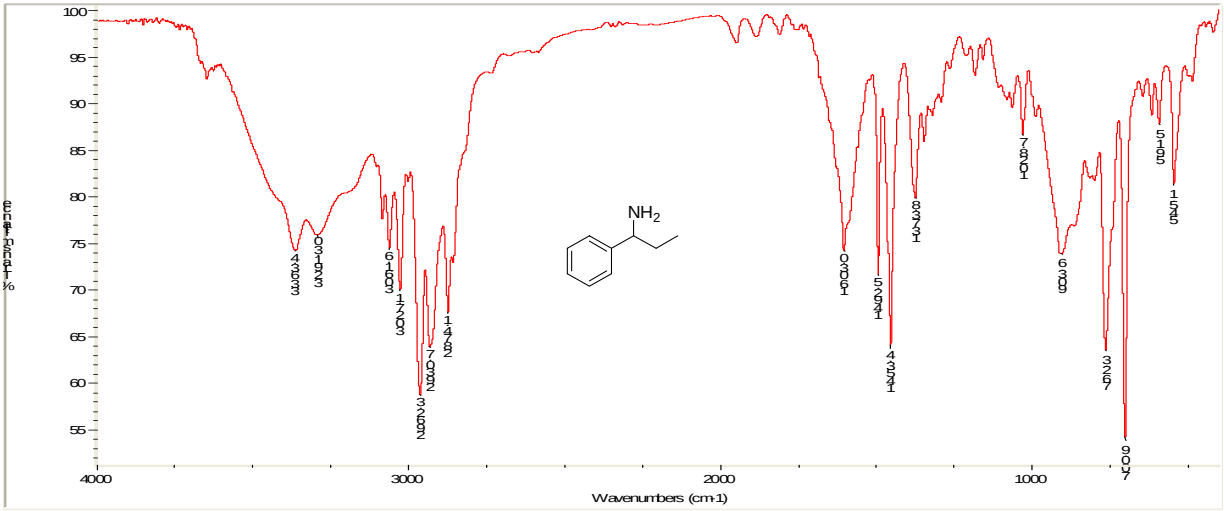
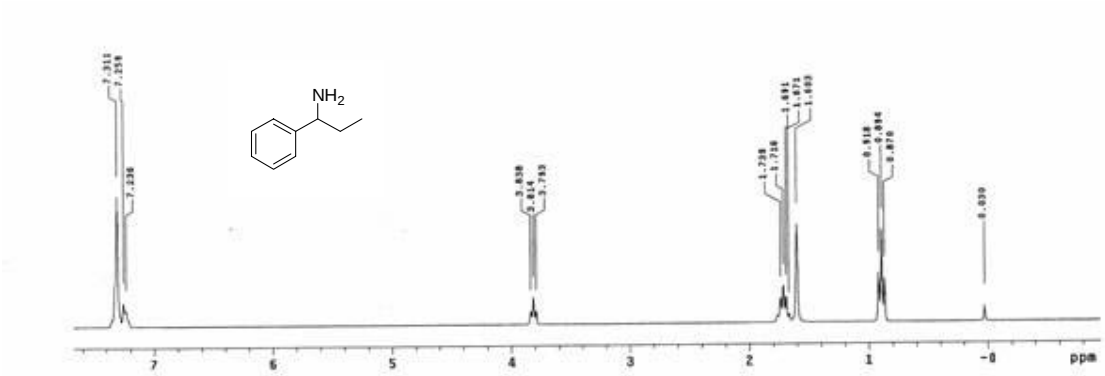

NH2
Wavenumbers (cm-1)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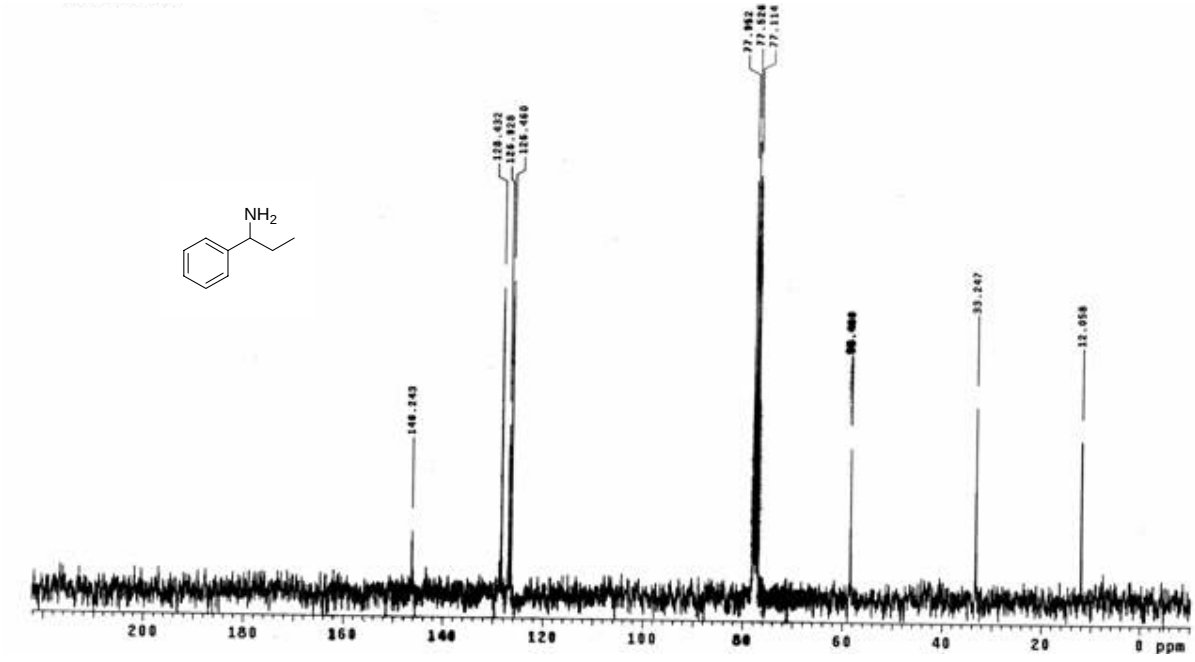
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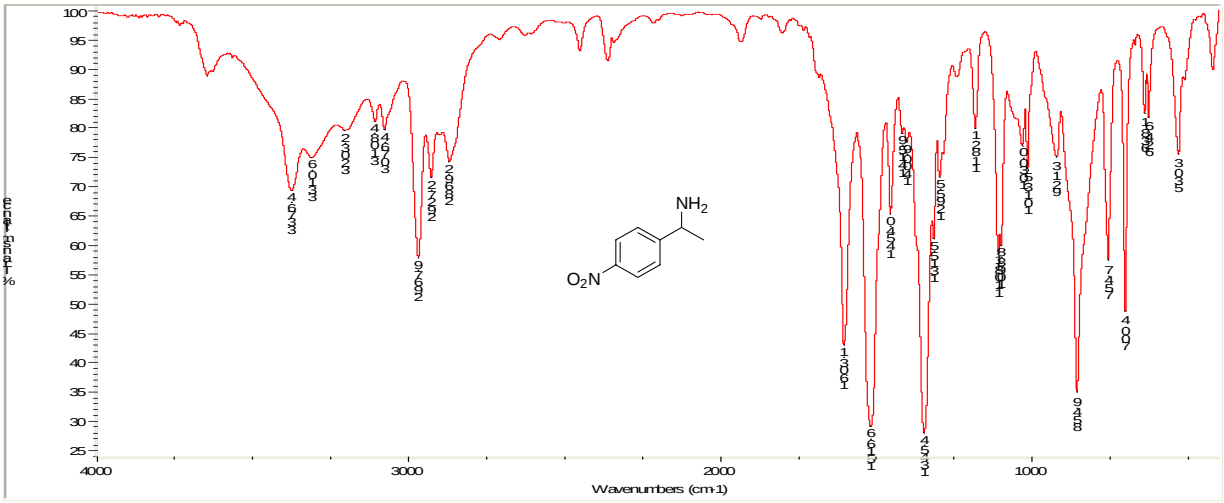
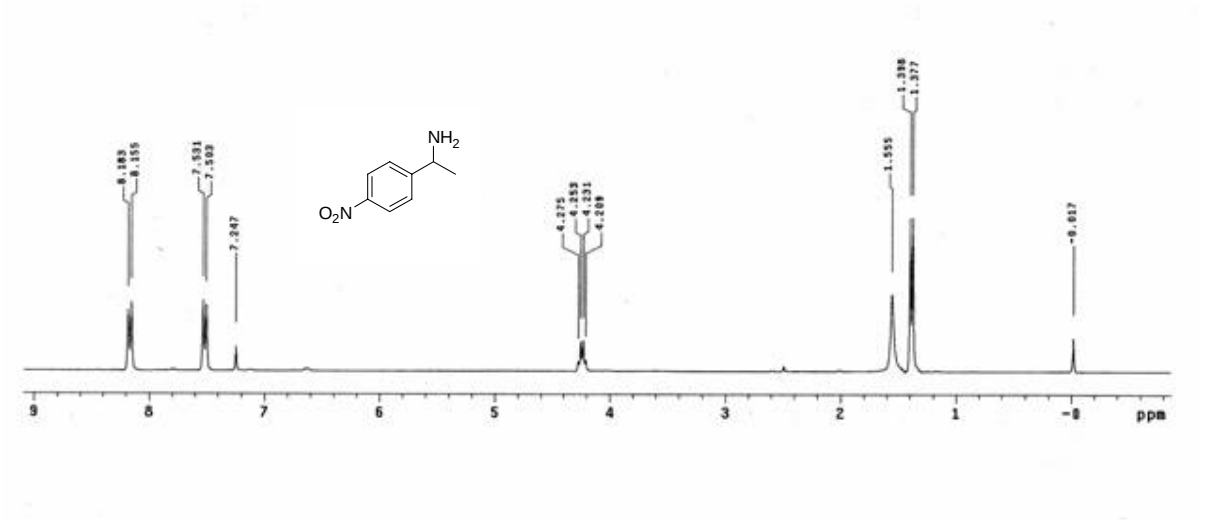

NH2
O2N
Wavenumbers (cm-1)


NH2
O2N
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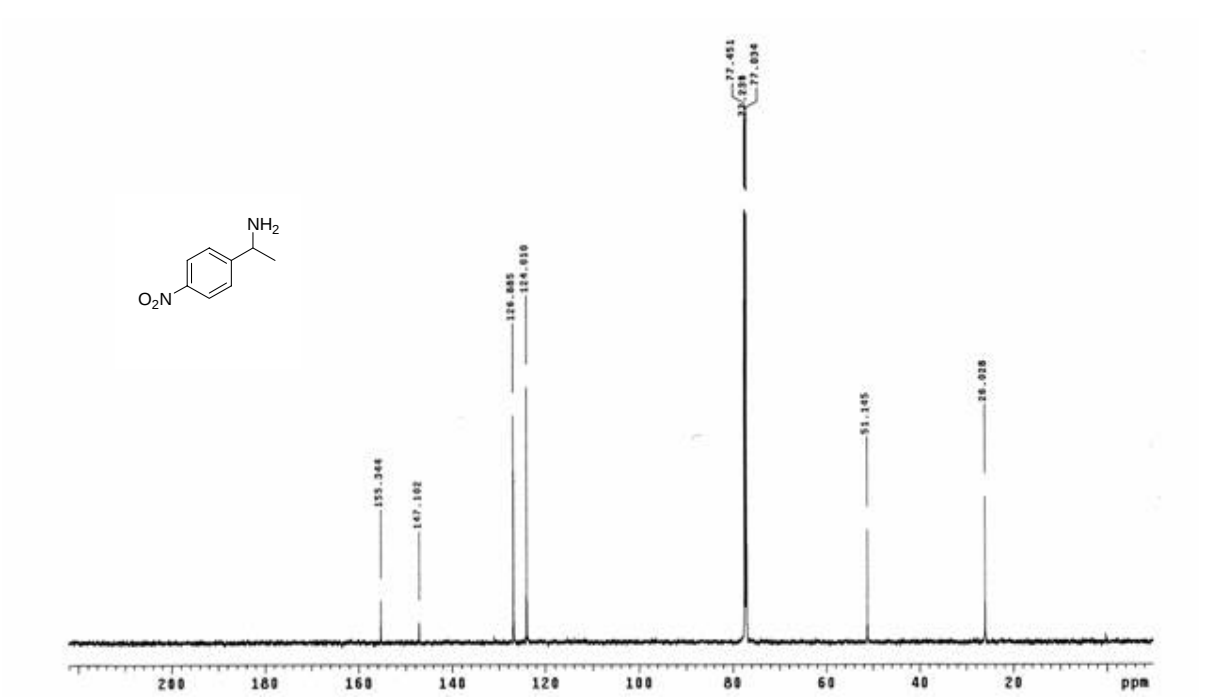
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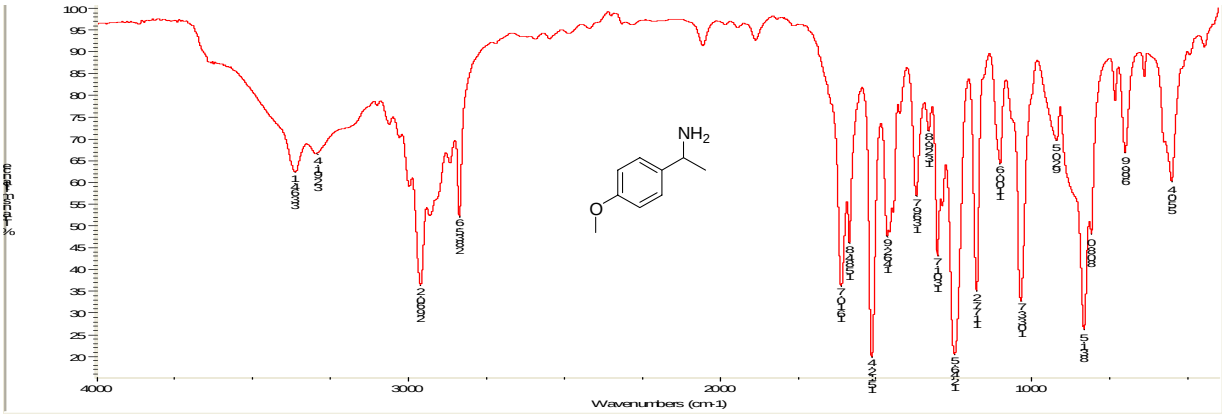
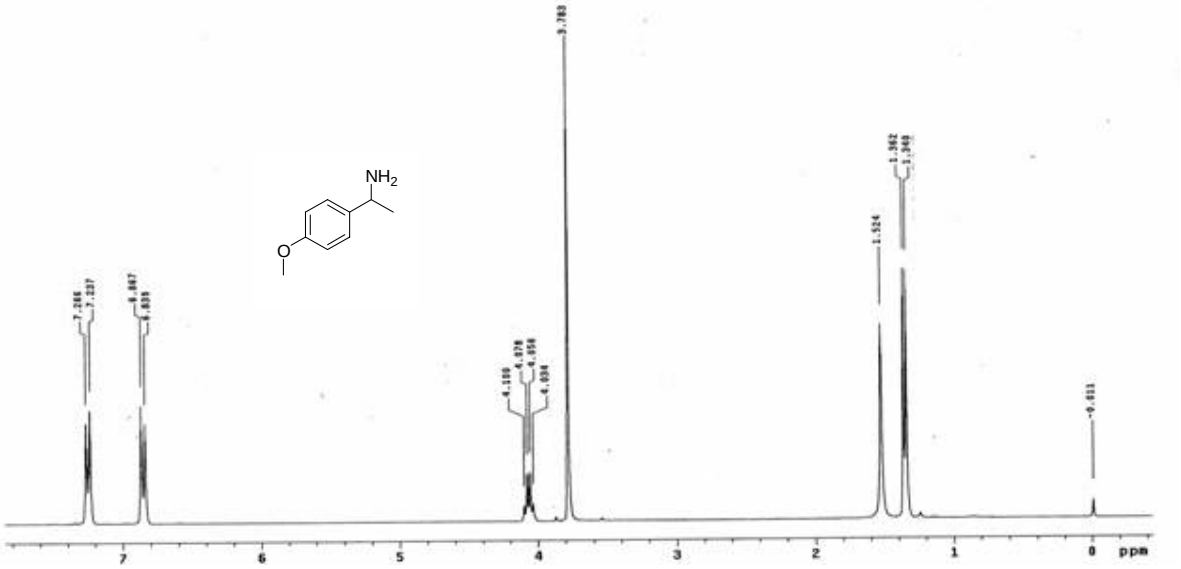
O2N
ppm


NH2
O
Wavenumbers (cm-1)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O
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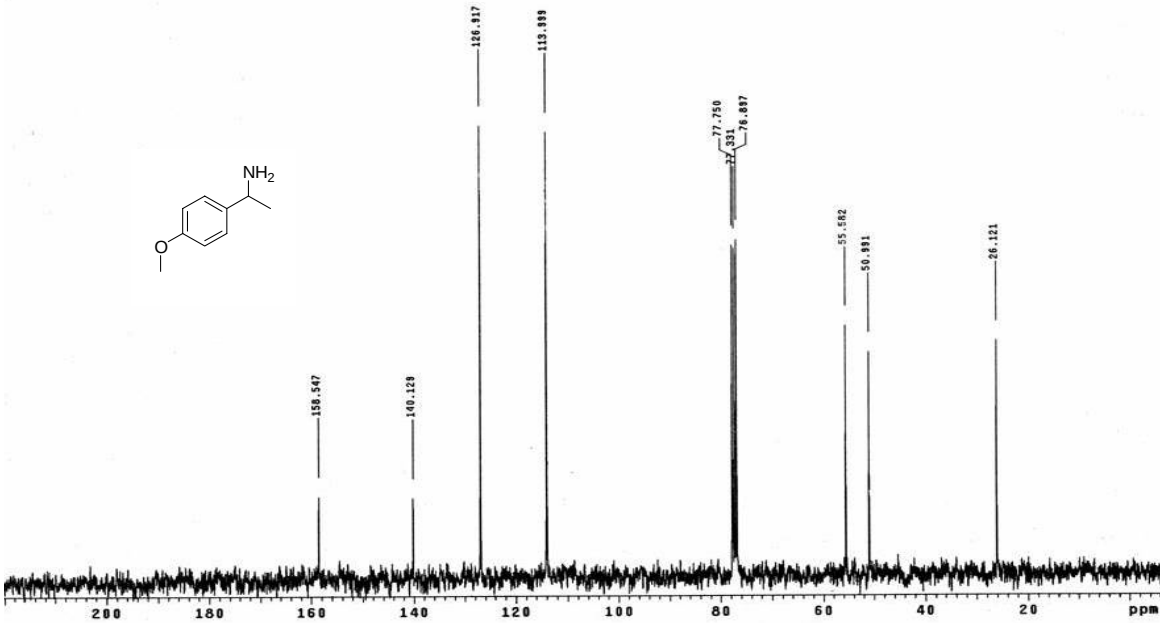
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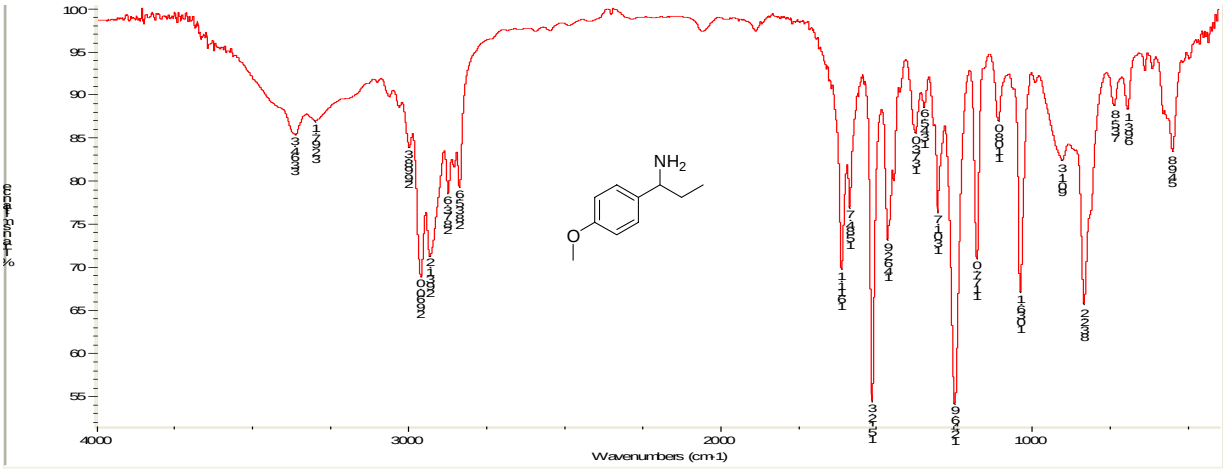
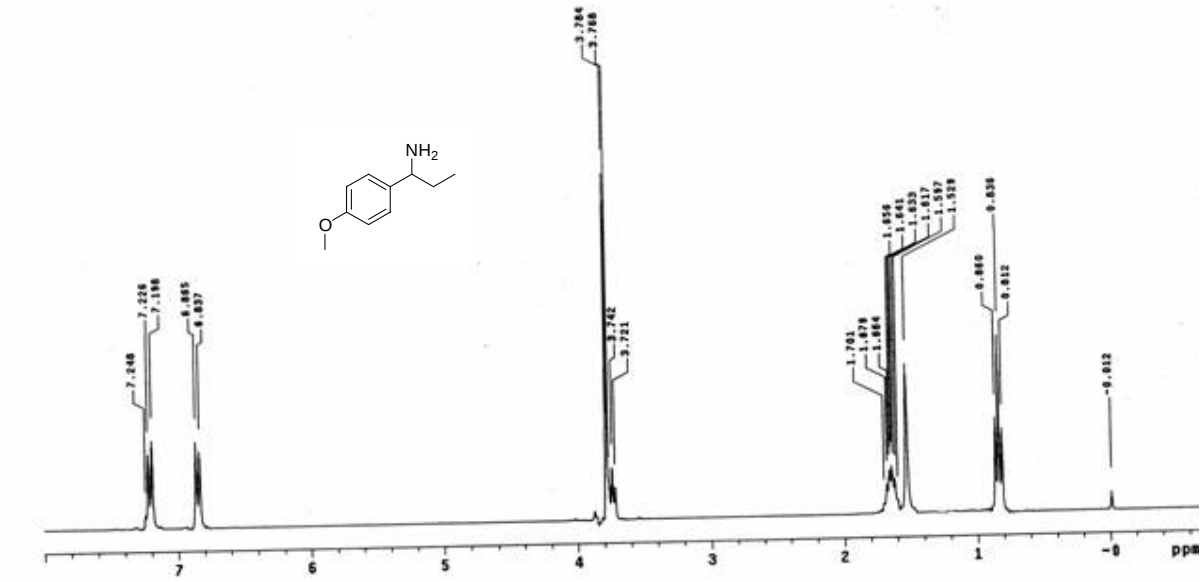

NH2
O
Wavenumbers (cm-1)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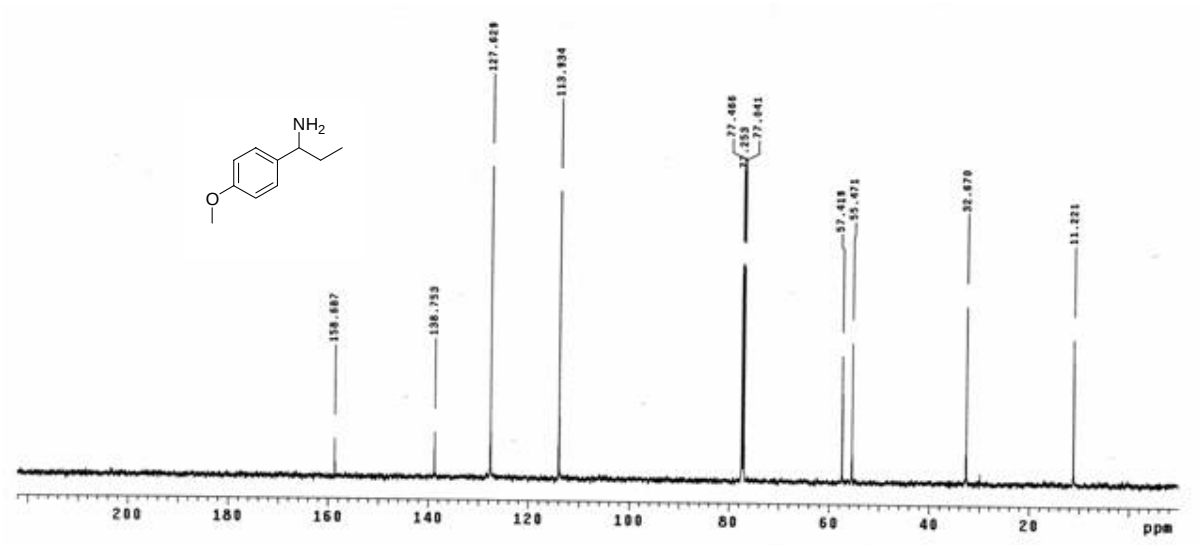
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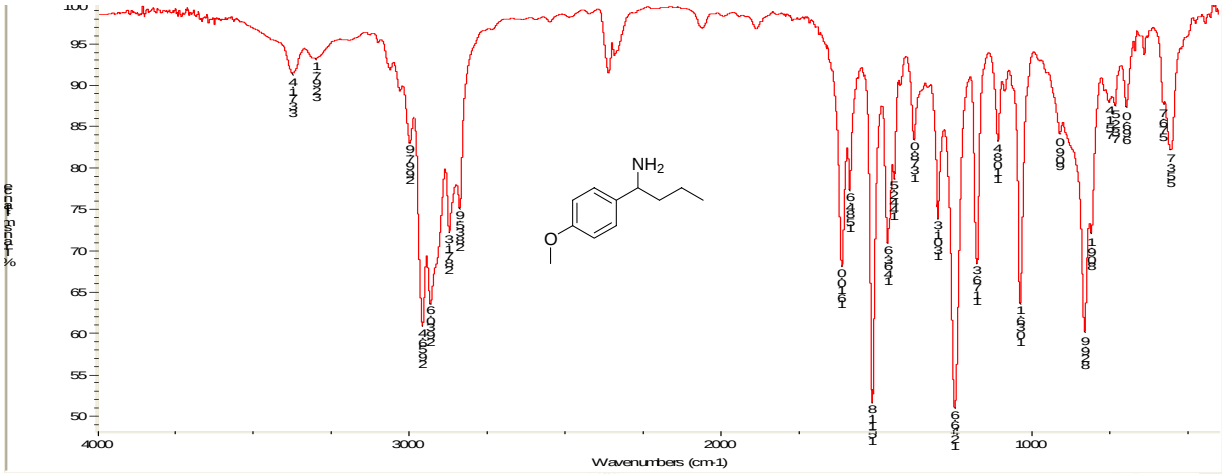
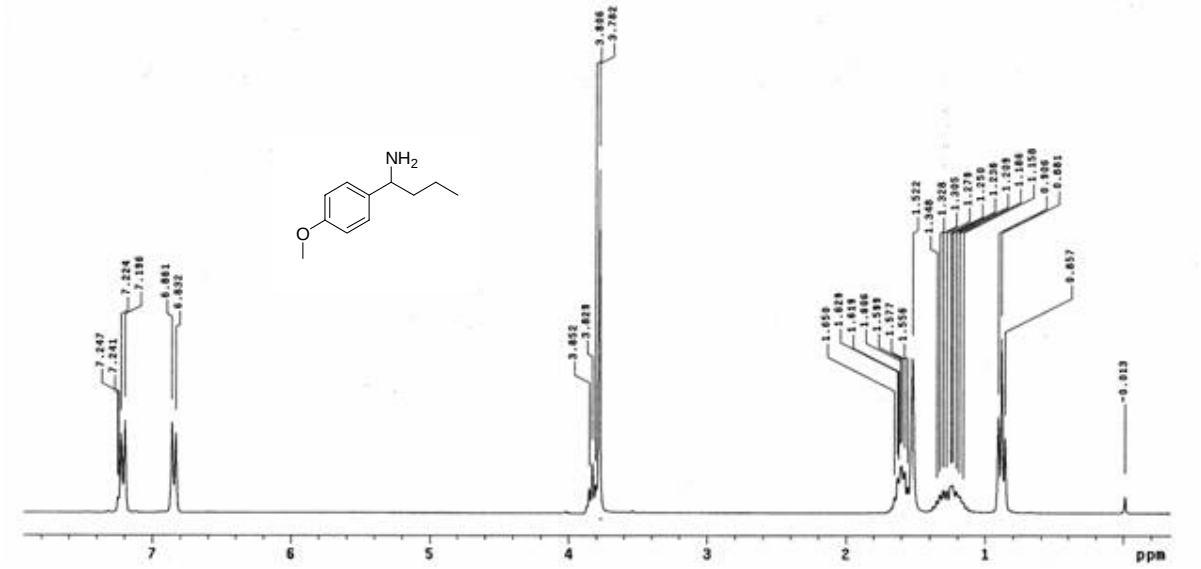

NH2
O
Wavenumbers (cm-1)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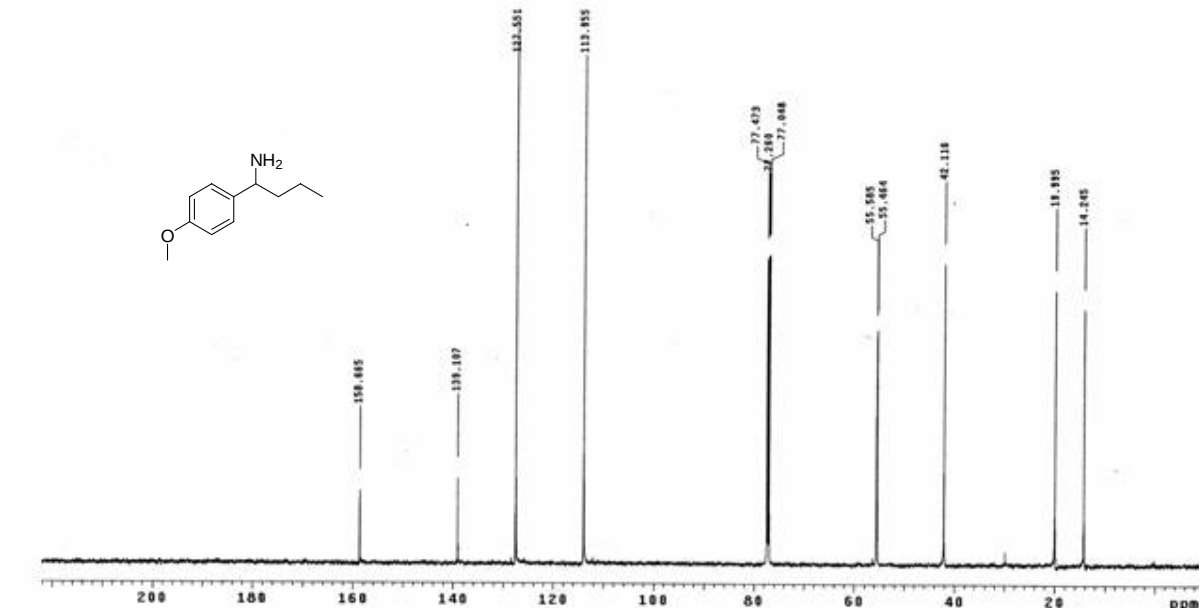
ppm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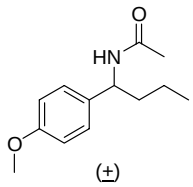
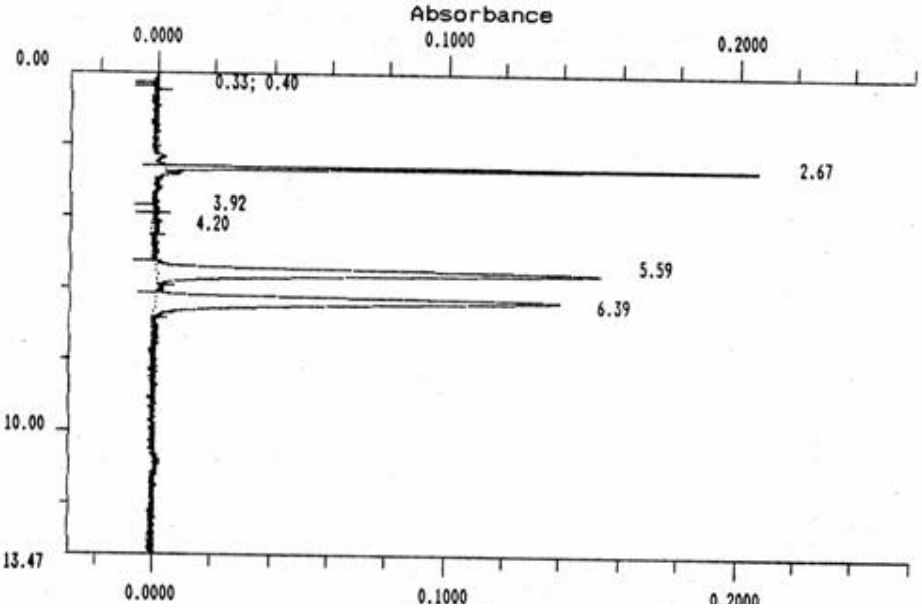


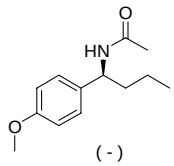
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30135849			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.326		0.0000	0.000	0.02715	0.00049	BCV	0.0000	0.0000	0.034	0.099
	2	0.397		0.0000	0.000	0.01232	0.00058	VCB	0.0000	0.0000	0.016	0.115
Q	3	2.667		0.0000	0.000	13.32660	0.20420	BCB	0.0000	0.0000	16.740	40.846
	4	3.925		0.0000	0.000	0.29265	0.00115	BMS	0.0000	0.0000	0.367	0.230
	5	4.205		0.0000	0.000	0.83721	0.00258	SCB	0.0000	0.0000	1.052	0.515
	6	5.592		0.0000	0.000	32.22078	0.15237	BCB	0.0000	0.0000	40.474	30.479
	7	6.390		0.0000	0.000	32.89188	0.13856	BCB	0.0000	0.0000	41.317	27.716
TOTALS				0.0000	0.000	79.60859	0.49993				100.000	100.000

Average Efficiency: 0



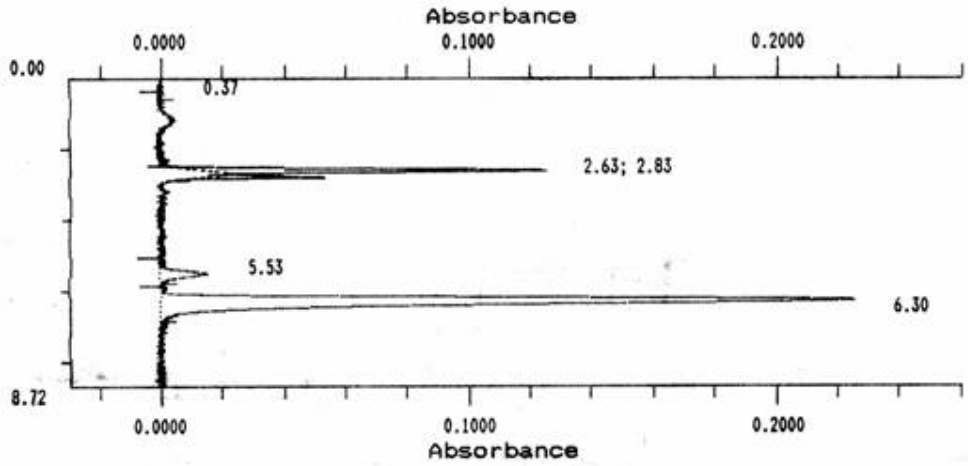
HPLC chromatogram for acetylated derivative of racemic 1-(4-methoxyphenyl)butylamine



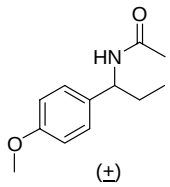
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30134B18			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.371		0.0000	0.000	0.13140	0.00085	BCB	0.0000	0.0000	0.183	0.217
Q	2	2.625		0.0000	0.000	11.00355	0.10813	BCB	0.0000	0.0000	15.353	27.724
Q	3	2.833		0.0000	0.000	3.13311	0.04112	BCB	0.0000	0.0000	4.372	10.544
	4	5.528		0.0000	0.000	3.53419	0.01497	BCB	0.0000	0.0000	4.931	3.839
	5	6.301		0.0000	0.000	53.86934	0.22494	BCB	0.0000	0.0000	75.161	57.676
TOTALS				0.0000	0.000	71.67159	0.39000				100.000	100.000

Average Efficiency: 0



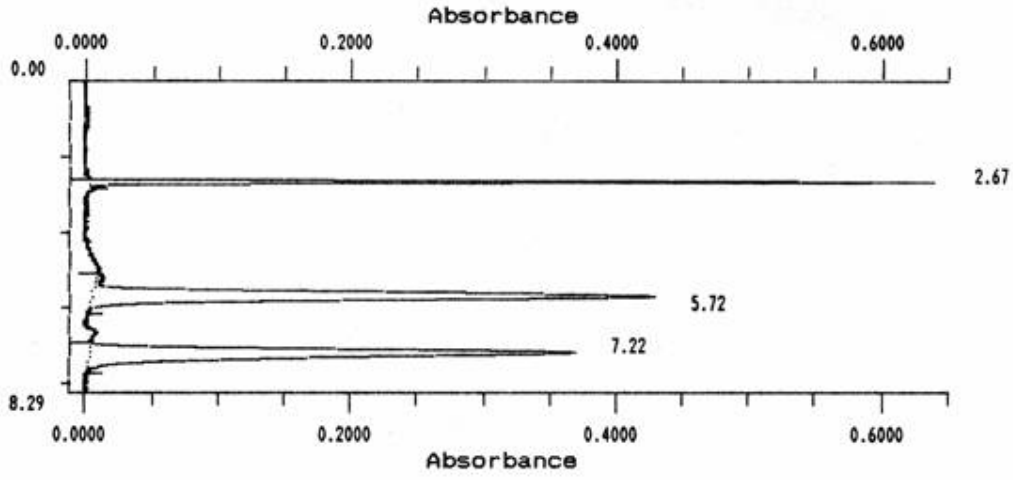
HPLC chramatogram for acetylated derivative of (*S*)-1-(4-methoxyphenyl)butylamine obtained from the reduction of O-methyl 4-methoxyphenyl propyl ketoxime



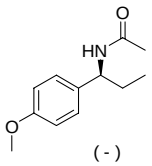
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30131218			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
Q 1	2.675		0.0000	0.000	42.68852	0.59471	BCB	0.0000	0.0000	18.527	42.960
2	5.721		0.0000	0.000	96.32253	0.42483	SCB	0.0000	0.0000	41.804	30.688
Q 3	7.217		0.0000	0.000	91.40274	0.36481	BCB	0.0000	0.0000	39.669	26.352
TOTALS			0.0000	0.000	230.41379	1.38434				100.000	100.000

Average Efficiency: 0



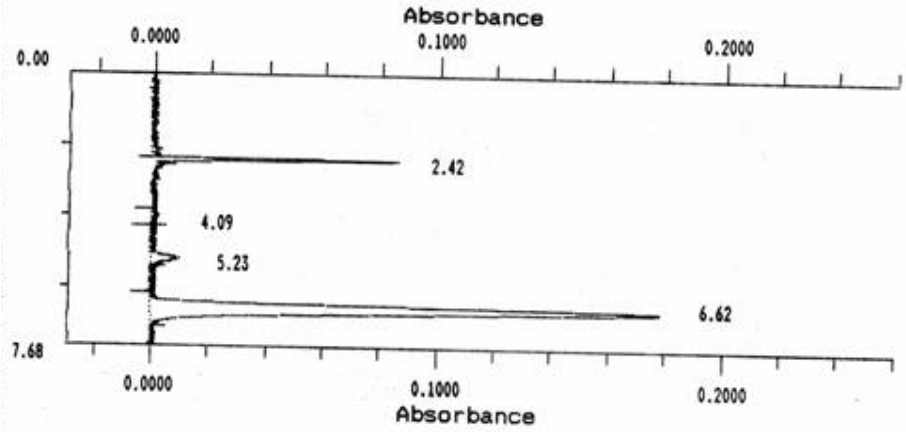
HPLC Chromatogram for acetylated derivative of racemic 1-(4-methoxyphenyl)propylamine



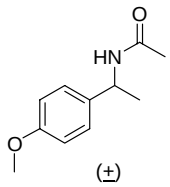
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30141657			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
Q 1	2.417		0.0000	0.000	5.35765	0.06672	BCB	0.0000	0.0000	10.163	25.901
2	4.093		0.0000	0.000	0.57727	0.00199	BCB	0.0000	0.0000	1.095	0.773
3	5.225		0.0000	0.000	2.63119	0.00982	BCB	0.0000	0.0000	4.991	3.813
Q 4	6.616		0.0000	0.000	44.15081	0.17905	BCB	0.0000	0.0000	83.751	69.513
TOTALS			0.0000	0.000	52.71692	0.25757				100.000	100.000

Average Efficiency: 0



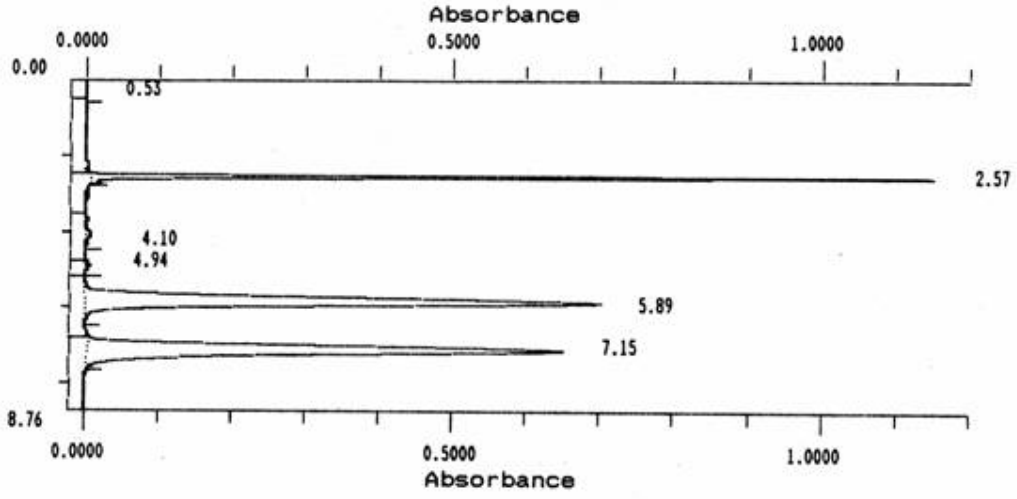
HPLC Chromatogram for acetylated derivative of (*S*)-1-(4-methoxyphenyl)propylamine obtained from the reduction of O-methyl 4-methoxyphenyl ethyl ketoxime



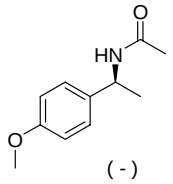
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30123259			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.529		0.0000	0.000	0.05639	0.00035	BCB	0.0000	0.0000	0.013	0.014
Q	2	2.567		0.0000	0.000	83.13570	1.11407	BCB	0.0000	0.0000	18.941	45.096
	3	4.098		0.0000	0.000	2.58745	0.00826	BCB	0.0000	0.0000	0.590	0.334
	4	4.940		0.0000	0.000	1.24920	0.00712	BCB	0.0000	0.0000	0.285	0.289
	5	5.886		0.0000	0.000	176.97403	0.70081	BCB	0.0000	0.0000	40.321	28.367
Q	6	7.150		0.0000	0.000	174.90218	0.63985	BCB	0.0000	0.0000	39.850	25.900
TOTALS				0.0000	0.000	438.90494	2.47047				100.000	100.000

Average Efficiency: 0



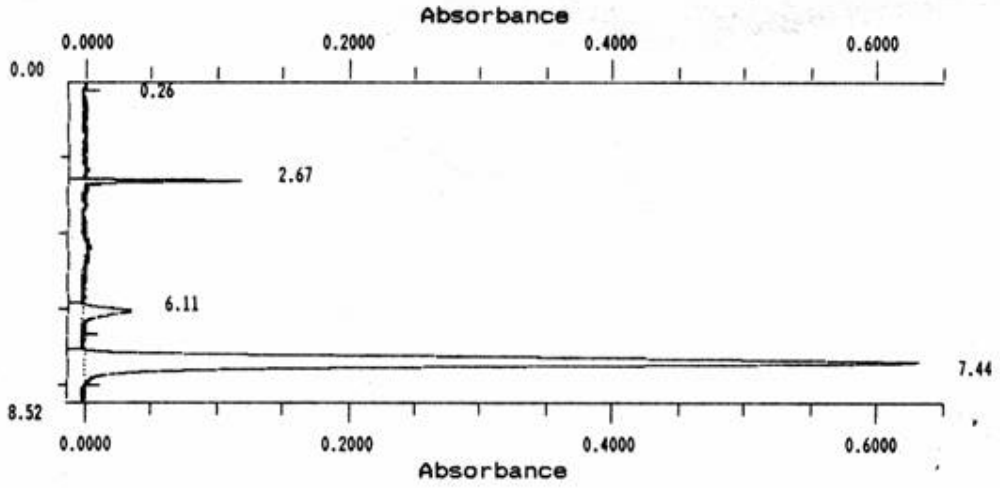
HPLC chromatogram for acetylated derivative of racemic 1-(4-methoxyphenyl)ethylamine



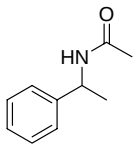
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30125300			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.257		0.0000	0.000	0.00601	0.00046	BCB	0.0000	0.0000	0.003	0.059
Q	2	2.675		0.0000	0.000	7.32024	0.11097	BCB	0.0000	0.0000	3.937	14.266
Q	3	6.107		0.0000	0.000	8.60447	0.03615	BCB	0.0000	0.0000	4.628	4.648
Q	4	7.442		0.0000	0.000	170.00163	0.63027	BCB	0.0000	0.0000	91.432	81.027
TOTALS				0.0000	0.000	185.93235	0.77784				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-(4-methoxyphenyl)ethylamine obtained from the reduction of O-methyl 4-methoxyphenyl methyl ketoxime



(±)

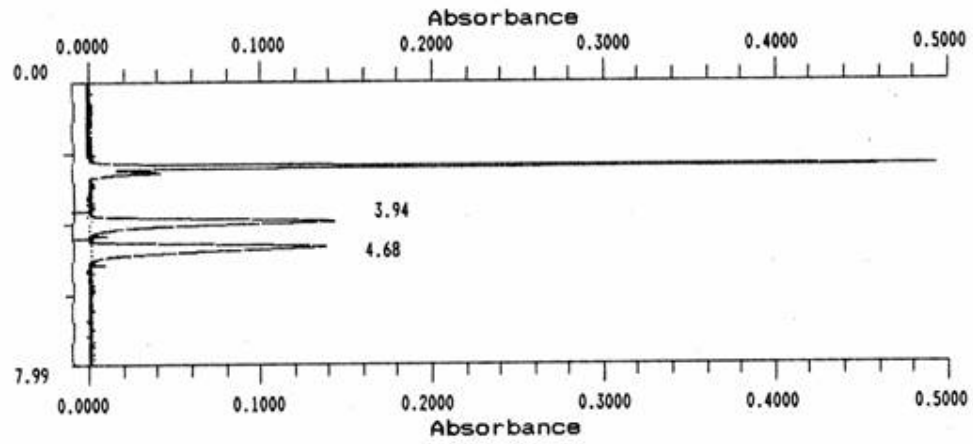
SAMPLE TABLE none
SYSTEM 1: SYSTEM1

Chart Speed 0.50 cm/min

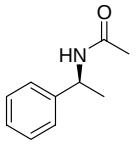
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	02091035			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
Q 1	3.936		0.0000	0.000	30.00108	0.14309	BCB	0.0000	0.0000	49.613	51.060
Q 2	4.677		0.0000	0.000	30.46896	0.13715	BCB	0.0000	0.0000	50.387	48.940
TOTALS			0.0000	0.000	60.47004	0.28025				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of racemic 1-phenylethylamine

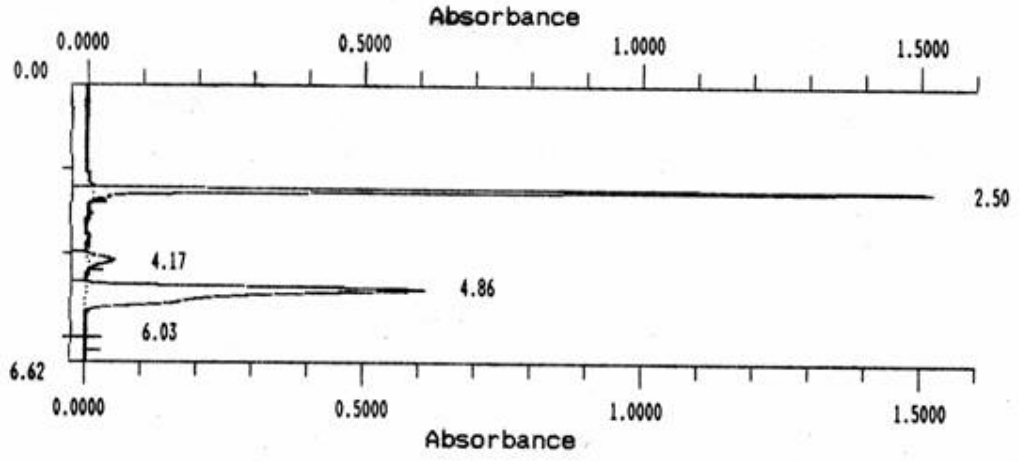


(-)

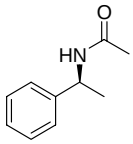
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	02095313			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
Q 1	2.500		0.0000	0.000	133.61227	1.48282	BCB	0.0000	0.0000	44.243	69.277
2	4.172		0.0000	0.000	9.47655	0.04745	BCB	0.0000	0.0000	3.139	2.217
3	4.857		0.0000	0.000	158.56706	0.60997	BCS	0.0000	0.0000	52.506	28.497
4	6.033		0.0000	0.000	0.33713	0.00019	SMB	0.0000	0.0000	0.112	0.009
TOTALS			0.0000	0.000	301.99302	2.14043				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-phenylethylamine obtained from the reduction of O-benzyl acetophenoneoxime

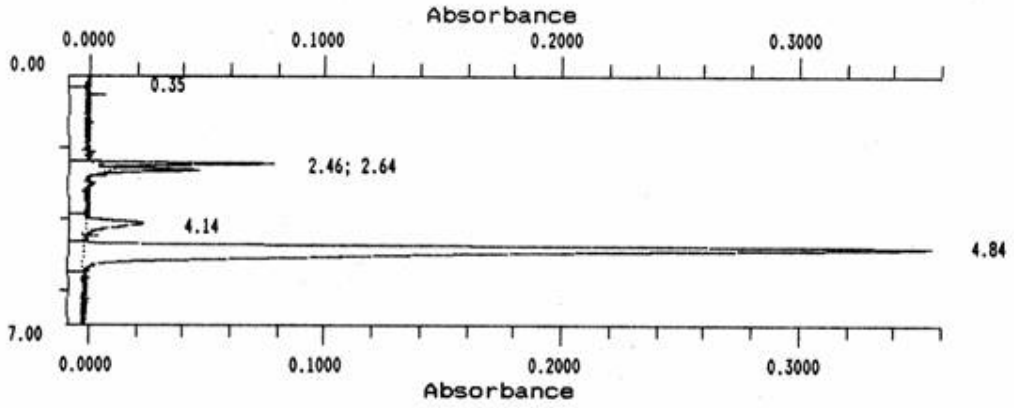


(-)

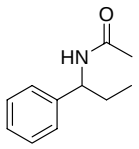
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	02093929			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.348		0.0000	0.000	0.04333	0.00071	BCB	0.0000	0.0000	0.043	0.146
Q	2	2.458		0.0000	0.000	5.27706	0.06943	BCB	0.0000	0.0000	5.234	14.282
Q	3	2.642		0.0000	0.000	3.28659	0.03374	BCB	0.0000	0.0000	3.260	6.940
Q	4	4.144		0.0000	0.000	5.43755	0.02473	BCB	0.0000	0.0000	5.393	5.087
Q	5	4.839		0.0000	0.000	86.77912	0.35753	BCV	0.0000	0.0000	86.070	73.545
TOTALS				0.0000	0.000	100.82365	0.48614				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-phenylethylamine obtained from the reduction of *O*-methyl acetophenoneoxime

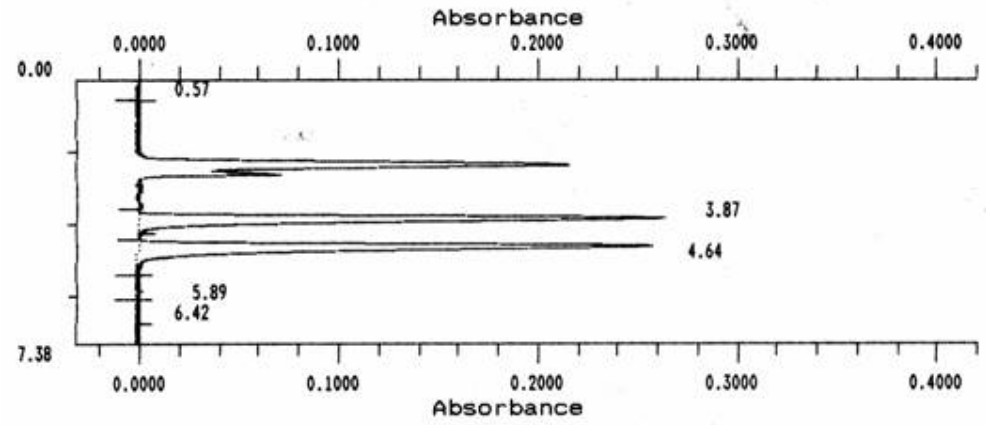


(±)

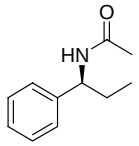
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	01173143			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
1	0.566		0.0000	0.000	0.03076	0.00092	BCB	0.0000	0.0000	0.029	0.175
2	3.871		0.0000	0.000	50.83171	0.26368	BCB	0.0000	0.0000	48.456	50.216
3	4.638		0.0000	0.000	53.12435	0.25824	BCB	0.0000	0.0000	50.642	49.182
4	5.894		0.0000	0.000	0.48372	0.00137	BCB	0.0000	0.0000	0.461	0.262
5	6.419		0.0000	0.000	0.43204	0.00087	BCB	0.0000	0.0000	0.412	0.165
TOTALS			0.0000	0.000	104.90258	0.52508				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of racemic 1-phenylpropy1amine



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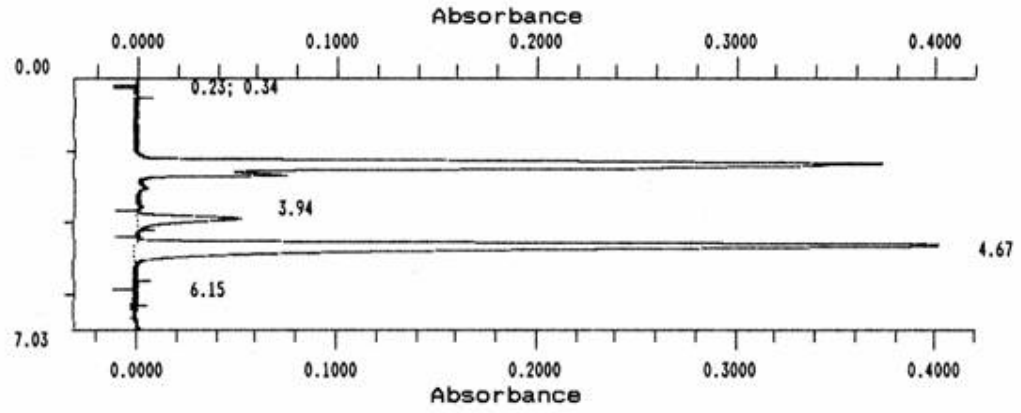
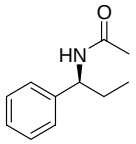
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	01173941			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
1	0.230		0.0000	0.000	0.03568	0.00053	BCV	0.0000	0.0000	0.035	0.108
2	0.343		0.0000	0.000	0.04748	0.00049	VCB	0.0000	0.0000	0.046	0.100
3	3.938		0.0000	0.000	10.03512	0.05131	BCB	0.0000	0.0000	9.720	10.386
4	4.672		0.0000	0.000	92.56631	0.43991	BCB	0.0000	0.0000	89.664	89.044
5	6.149		0.0000	0.000	0.55184	0.00179	BCB	0.0000	0.0000	0.535	0.362
TOTALS			0.0000	0.000	103.23643	0.49403				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-phenylpropy1amine obtained from the reduction of O-*n*-butyl propionylphenone oxime



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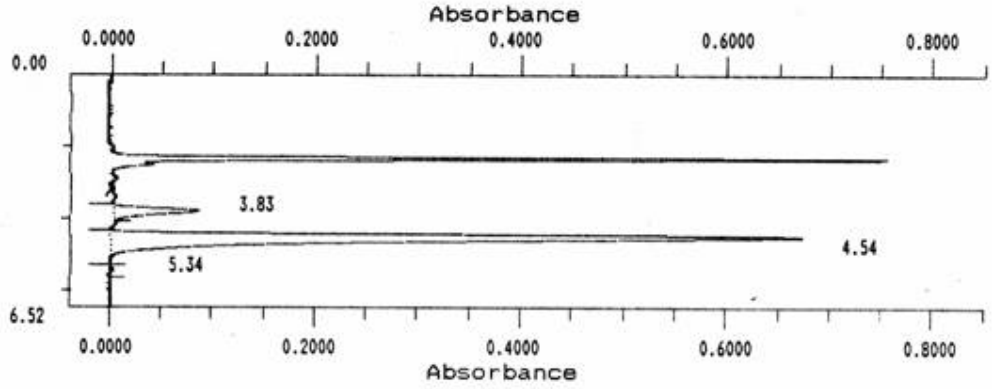
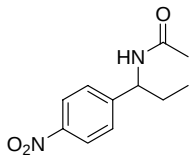
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	01174728			1.00000	1 / 1			(from File)

Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
1	3.832		0.0000	0.000	15.79762	0.08247	BCB	0.0000	0.0000	9.560	10.897
2	4.542		0.0000	0.000	148.89305	0.67392	BCS	0.0000	0.0000	90.104	89.052
3	5.342		0.0000	0.000	0.55469	0.00039	SMB	0.0000	0.0000	0.336	0.051
TOTALS			0.0000	0.000	165.24537	0.75678				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-phenylpropy1amine obtained from the reduction of O-benzyl propionylphenone oxime

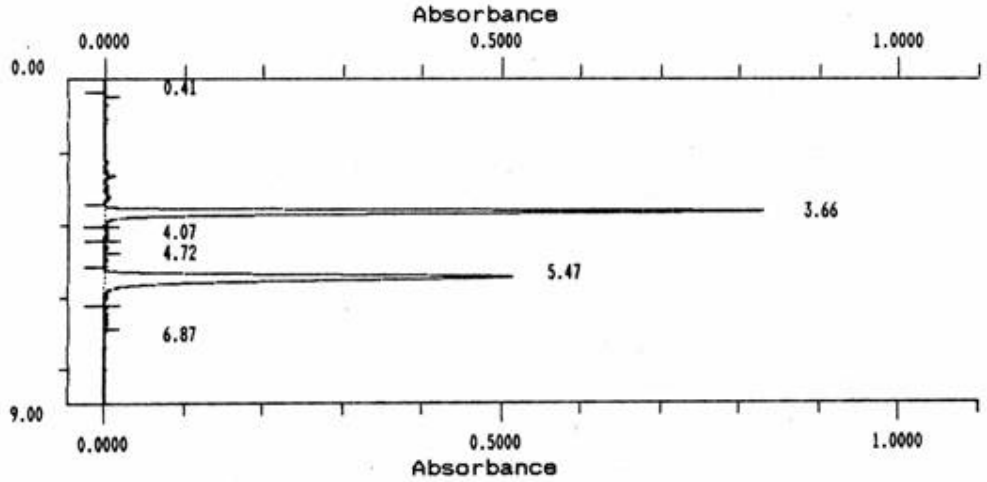


(±)

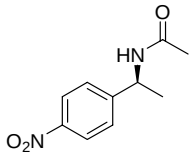
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	29140500			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.412		0.0000	0.000	0.01721	0.00035	BCB	0.0000	0.0000	0.008	0.026
Q	2	3.658		0.0000	0.000	102.55048	0.82671	BCS	0.0000	0.0000	50.037	61.473
	3	4.067		0.0000	0.000	0.80611	0.00067	SNB	0.0000	0.0000	0.393	0.050
	4	4.721		0.0000	0.000	0.19493	0.00101	BCB	0.0000	0.0000	0.096	0.075
	5	5.474		0.0000	0.000	100.46783	0.51425	BCB	0.0000	0.0000	49.020	38.239
	6	6.865		0.0000	0.000	0.91400	0.00184	BCB	0.0000	0.0000	0.446	0.137
TOTALS				0.0000	0.000	204.95057	1.34483				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of racemic 1-(4-nitrophenyl)ethylamine

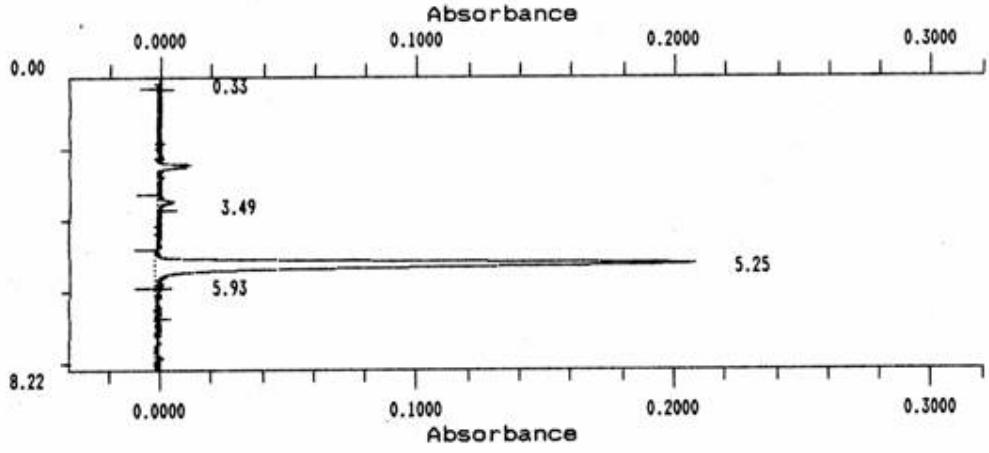


(-)

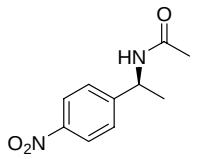
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	29141445			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.335		0.0000	0.000	0.00239	0.00008	BCB	0.0000	0.0000	0.006	0.038
Q	2	3.492		0.0000	0.000	0.80280	0.00544	BCB	0.0000	0.0000	1.893	2.530
	3	5.255		0.0000	0.000	40.72330	0.20929	BCS	0.0000	0.0000	96.028	97.300
	4	5.925		0.0000	0.000	0.87901	0.00028	SMB	0.0000	0.0000	2.073	0.132
TOTALS				0.0000	0.000	42.40750	0.21510				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-(4-nitrophenyl)ethylamine obtained from the reduction of O-methyl 4-nitrophenyl methyl ketoxime

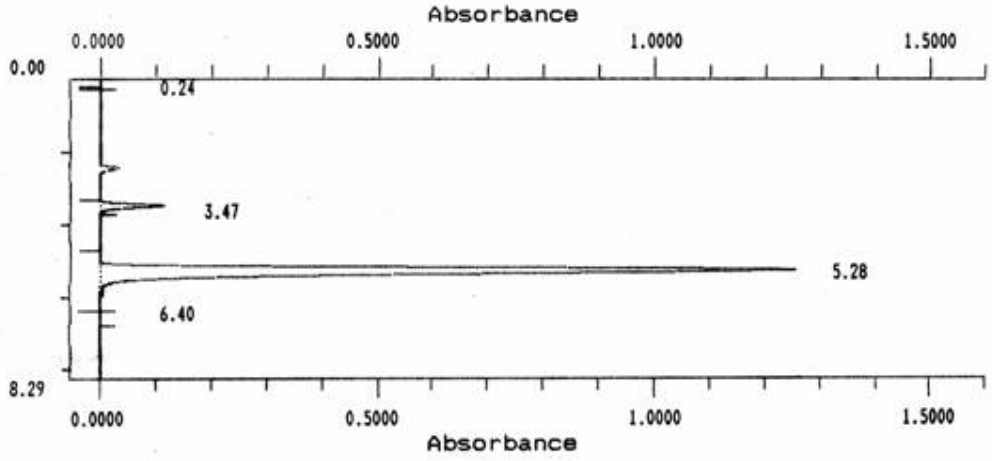


(-)

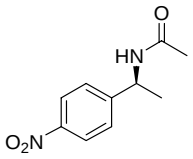
Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	29142348			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
	1	0.244		0.0000	0.000	0.04144	0.00056	BCB	0.0000	0.0000	0.015	0.041
Q	2	3.475		0.0000	0.000	14.45663	0.11348	BCB	0.0000	0.0000	5.171	8.258
	3	5.276		0.0000	0.000	264.63403	1.26002	BCS	0.0000	0.0000	94.666	91.691
	4	6.400		0.0000	0.000	0.41335	0.00014	SNB	0.0000	0.0000	0.148	0.010
TOTALS				0.0000	0.000	279.54546	1.37420				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-(4-nitrophenyl)ethylamine obtained from the reduction of O-benzyl 4-nitrophenyl methyl ketoxime

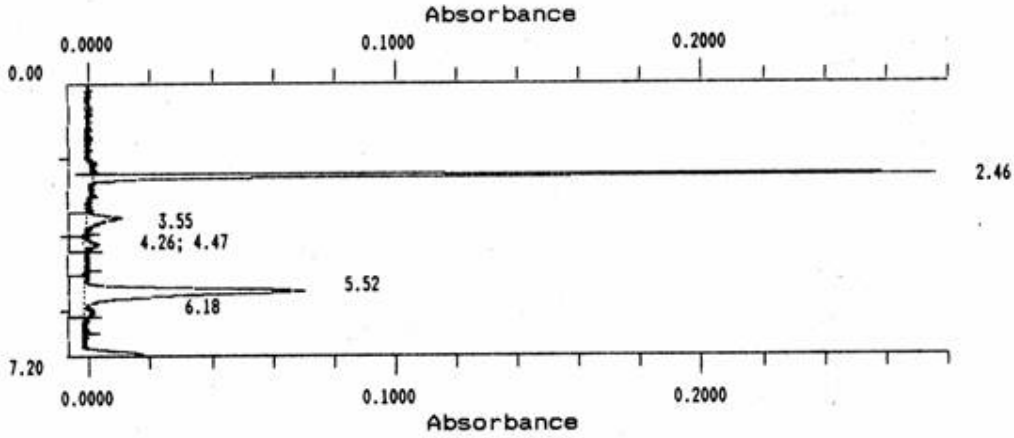


(-)

Type	Sample Name	Sample Amount	Int Std Amount	Scale Factor	Nr. Inj	Vial Nr.	Inject Vol uL	
Unk	30093753			1.00000	1 / 1			(from File)

	Peak Number	Retention Time	Component Name	Concentration mg / mL	Normalized Concentration	Peak Area	Peak Height	Base Code	Response Factor	Rel. Ret Time	Area Percent	Height Percent
Q	1	2.458		0.0000	0.000	21.01708	0.26740	BCB	0.0000	0.0000	50.629	75.172
Q	2	3.549		0.0000	0.000	2.39658	0.01148	BCB	0.0000	0.0000	5.774	3.228
	3	4.259		0.0000	0.000	0.88834	0.00418	BCS	0.0000	0.0000	2.140	1.175
	4	4.467		0.0000	0.000	0.33718	0.00002	SNB	0.0000	0.0000	0.812	0.006
	5	5.515		0.0000	0.000	16.43628	0.07167	BCS	0.0000	0.0000	39.595	20.147
	6	6.175		0.0000	0.000	0.43607	0.00097	SNB	0.0000	0.0000	1.050	0.272
TOTALS				0.0000	0.000	41.51152	0.35572				100.000	100.000

Average Efficiency: 0



HPLC chromatogram for acetylated derivative of (*S*)-1-(4-nitrophenyl)ethylamine obtained from the reduction of O-*n*-butyl 4-nitrophenyl methyl ketoxime