APPENDIX 1

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Supporting Methods. All reactions using diphenylzinc and titanium(IV) isopropoxide were carried out in a Vacuum Atmospheres dry box, with an attached MO-40 Dritrain or by using standard Schlenk and vacuum line techniques with oven dried glassware. All chemicals were obtained from Acros Organics or Aldrich. All solvents were purchased from Fischer Scientific. Diphenylzinc was purchased from Strem and stored as a solid in a Vacuum Atmospheres dry box. All liquid ketone substrates were distilled prior to use. Toluene and hexanes were dried through alumina columns. The progress of all reactions was monitored by TLC to ensure the reactions had reached completion. ^{1}H NMR and ^{13}C NMR spectra were obtained on a Brüker AM-500 Fourier transform NMR spectrometer at 500 and 125 MHz, respectively. Chemical shifts are reported in units of parts per million downfield from tetramethylsilane, and all coupling constants are reported in Hertz. Silica gel (230-400 mesh, Silicycle) was used for air-flashed chromatography. TLC was performed on Whatman precoated silica gel 60 F-254 plates and visualized by ultraviolet light and/or 10% phosphomolybdic acid in ethanol with heating. The infrared spectra were obtained using a Perkin-Elmer 1600 series spectrometer. Melting points are reported in degrees Celsius and are uncorrected.

Synthesis of Substrate Ketones.

Preparation of 2-(*tert*-butyldimethylsilanyloxymethyl)-2-cyclohexenone. 2-(*tert*-Butyldimethylsilanyloxymethyl)-2-cyclohexenone was prepared by mixing 2-hydroxymethyl-2-cyclohexenone (493.0 mg, 3.9 mmol), *tert*-butyldimethylsilylchloride (648 mg, 4.3 mmol), 4-dimethylaminopyridine (97.7 mg, 0.8 mmol) and imidazole (398 mg, 5.85 mmol) in *N,N*-dimethylformamide at room temperature. After the reaction was done, the residue was purified by silica gel column chromatography to give the product (539 mg, 58% yield) as an oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 0.047 (s, 3H), 0.053 (s, 3H), 0.90 (s, 9H), 1.96-1.98 (m, 2H), 2.37-2.41 (m, 4H), 4.32-4.33 (m, 2H), 6.97 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ –5.1, 18.7, 23.3, 26.0, 26.3, 38.8, 60.4, 138.8, 144.3, 199.4 ppm; IR (film) 2954, 1673, 1339, 1256, 837 cm^{-1}; high resolution MS (HRMS) calcd for $C_{13}H_{24}O_2Si$ $(M + H)^+$: 241.1624; found: 241.1619.

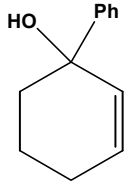
Preparation of 8,9-dihydro-benzocyclohepten-5-one. 8,9-Dihydro-benzocyclohepten-5-one was prepared according to literature procedure (1).

Preparation of 2-iodo-2-cyclopentenone. 2-Iodo-2-cyclopentenone was prepared according to literature procedure (2).

Preparation of 2-bromo-2-cyclohexenone. 2-Bromo-2-cyclohexenone was prepared according to literature procedure (3).

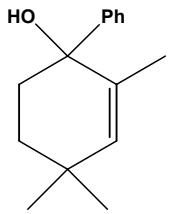
Preparation of 2-iodo-2-cyclohexenone. 2-Iodo-2-cyclohexenone was prepared according to literature procedure (2).

General Procedure A for the Preparation of Tertiary Alcohols. Preparation of 1-phenyl-cyclohex-2-enol (S1) (4)**.** The bis(sulfonamide) ligand (5.4 mg, 10 mol %) was



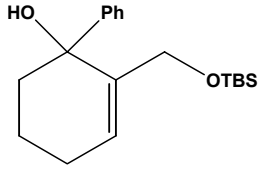
weighed into the well dried Schlenk flask and put into the dry box. Diphenylzinc (43.9 mg, 0.2 mmol) was added, previously dissolved in toluene (1 ml), followed by titanium(IV) isopropoxide (50 µl, 1.2 M hexanes solution, 0.06 mmol). The homogeneous reaction mixture was stirred at room temperature for 15 min. 2-Cyclohexenone (9.7 µl, 0.1 mmol) was then added as a solution in toluene (0.5 ml). The flask was sealed and removed from the dry box. The reaction mixture was stirred at room temperature until TLC showed complete consumption of the ketone. The reaction was quenched with a few drops of water, diluted by dichloromethane, dried using anhydrous $MgSO_4$, and filtered. The solvent was removed under vacuum. The residue was purified by flash silica gel column chromatography (hexanes/EtOAc, 95:5) to give **S1** [14 mg, 81% yield, 1% enantiomeric excess (ee)] as an oil: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.66-1.67 (m, 1H), 1.88-1.89 (m, 2H), 2.12-2.15 (m, 1H), 2.17-2.18 (m, 2H), 5.83 (d, J = 10 Hz, 1H), 6.06-6.08 (m, 1H), 7.29-7.31 (m, 1H), 7.37-7.40 (m, 2H), 7.53-7.55 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.6, 25.4, 40.0, 72.6, 125.9, 126.3, 127.2, 128.5, 131.0, 132.7, 148.3 ppm; IR (film) 3389, 1490, 1049, 961cm^{-1}; HRMS calcd for $C_{12}H_{14}O$ $(M)^+$: 174.1045; found: 174.1041.

Preparation of 2,4,4-trimethyl-1-phenyl-cyclohex-2-enol (S2). The general procedure A was applied to 2,4,4-trimethyl-2-cyclohexenone (15 µl, 0.1 mmol). The residue was



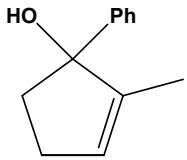
purified by silica gel column chromatography (hexanes/EtOAc, 95:5) to give **S2** (20 mg, 92% yield, 97% ee) as a white solid: m.p. 37-39℃; $[\alpha]_D^{20}$ = –83.9 (*c* 0.88, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 1.10 (s, 3H), 1.11 (s, 3H), 1.40-1.44 (m, 1H), 1.52-1.57 (m, 4H), 2.00-2.04 (m, 2H), 5.51 (s, 1H), 7.26-7.29 (m, 1H), 7.36-7.39 (m, 2H), 7.46-7.48 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 14.5, 29.5, 30.0, 32.0, 33.9, 38.7, 76.1, 126.3, 127.1, 128.4, 134.3, 138.1, 146.7 ppm; IR (film) 3442, 2950, 1446, 1359 cm^{-1}; HRMS calcd for $C_{15}H_{20}O$ $(M)^+$: 216.1514; found: 216.1508.

Preparation of 2-(*tert*-butyldimethylsilanyloxymethyl)-1-phenyl-cyclohex-2-enol (S3). The general procedure A was applied to 2-(*tert*-butyldimethylsilanyloxymethyl)-2-cyclohexenone (25 μl, 0.1 mmol). The residue was purified by silica gel column



chromatography (hexanes/EtOAc, 99:1) to give **S3** (20.2 mg, 64% yield, 80% ee) as an oil: $[\alpha]_D^{20}$ = +2.4 (*c* 0.93, $CHCl_3$); ^1H NMR (C_6D_6, 500 MHz) δ –0.24 (s, 3H), –0.17 (s, 3H), 0.83 (s, 9H), 1.44-1.48 (m, 1H), 1.73-1.77 (m, 1H), 1.90-1.97 (m, 3H), 2.10-2.14 (m, 1H), 3.82 (d, *J* = 11 Hz, 1H), 4.17 (d, *J* = 11 Hz, 1H), 5.78 (dd, *J* = 4.1, 4.0 Hz, 1H), 7.09-7.12 (m, 1H), 7.22-7.25 (m, 2H), 7.58-7.60 (m, 2H) ppm; ^{13}C NMR (C_6D_6, 125 MHz) δ –5.8, –5.7, 19.4, 25.97, 25.99, 41.7, 67.4, 74.9, 126.3, 126.7, 128.4, 129.3, 139.3, 148.9 ppm; IR (film) 3498, 2929, 2856, 1464, 1445 cm^{-1}; HRMS calcd for $C_{19}H_{30}O_2SiNa$ $(M + Na)^+$: 341.1813; found: 341.1910.

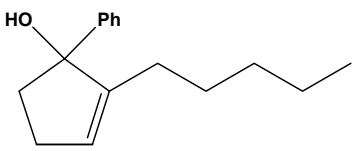
Preparation of 2-methyl-1-phenyl-cyclopent-2-enol (S4). The general procedure A was



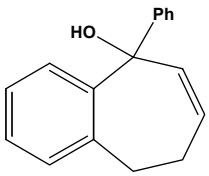
applied to 2-methyl-2-cyclopenten-1-one (20 μl, 0.2 mmol). The residue was purified by flash silica gel column chromatography (hexanes/EtOAc, 94:6) to give **S4** (20.7 mg, 60% yield, 84% ee) as an oil: $[\alpha]_D^{20}$ = –72.8 (*c* 0.92, $CHCl_3$); ^1H NMR (C_6D_6, 500 MHz) δ 1.46 (s, 3H), 2.02-2.26 (m, 4H), 5.40 (m, 1H), 7.07-7.11 (m, 1H), 7.20-7.23 (m, 2H), 7.45-7.47 (m, 2H) ppm; ^{13}C NMR (C_6D_6, 125 MHz) δ 12.1, 29.7, 43.9, 87.9, 125.4, 126.8, 128.1, 128.5, 144.9, 146.8 ppm; IR (film) 3406, 2923, 2852, 2361, 1491 cm^{-1}; HRMS calcd for $C_{12}H_{14}O$ $(M)^+$:

174.1045; found: 174.1040; HRMS calcd for $C_{12}H_{14}ONa$ $(M - H_2O)^+$: 156.0940; found: 156.0938.

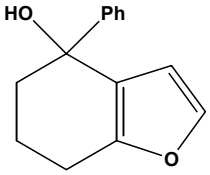
Preparation of 2-pentyl-1-phenyl-cyclopent-2-enol (S5). The general procedure A was applied to 2-pentyl-2-cyclopenten-1-one (16.5 µl, 0.1 mmol). The residue was purified by silica gel column chromatography (hexanes/EtOAc, 96:4) to give **S5** (17 mg, 74% yield, 97% ee) as an oil: $[\alpha]_D^{20}$ = +38.2 (*c* 1.26, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 0.89 (t, *J* = 7.5Hz, 3H), 1.27-1.31 (m, 4H), 1.48-1.50 (m, 2H), 1.74-1.76 (m, 1H), 1.95-1.97 (m, 1H), 2.24-2.28 (m, 1H), 2.39-2.41 (m, 2H), 2.50-2.52 (m, 1H), 5.76 (dd, *J* = 2.1, 1.9 Hz, 1H), 7.26-7.29 (m, 1H), 7.37-7.38 (m, 2H), 7.41-7.43 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 14.4, 22.9, 26.7, 28.0, 29.9, 32.3, 44.1, 88.6, 125.4, 126.9, 127.2, 128.5, 146.7, 149.3 ppm; IR (film) 3400, 2955, 2920, 2855 cm^{-1}; HRMS calcd for $C_{16}H_{22}O$ $(M^+$: 230.1671; found: 230.1664; HRMS calcd for $C_{16}H_{21}$ $(M–OH)^+$: 213.1644; found: 213.1641.



Preparation of 5-phenyl-8,9-dihydro-5*H*-benzocyclohepten-5-ol (S6). The general procedure A was applied to 8,9-dihydro-benzocyclohepten-5-one (14.3 µl, 0.1 mmol). The residue was purified by silica gel column chromatography (hexanes/EtOAc, 96:4) to give **S6** (22.1 mg, 94% yield, 84% ee) as an oil: $[\alpha]_D^{20}$ = –210.9 (*c* 0.97, $CHCl_3$); ^1H NMR (C_6D_6, 500 MHz) δ 1.90-1.97 (m, 2H), 2.04-2.08 (m, 1H), 2.76-2.82 (dd, *J* = 13, 4.5Hz, 1H), 5.47-5.50 (m, 1H), 6.02 (dd, *J* = 6.5, 2.4 Hz, 1H), 6.89-6.90 (dd, *J* = 3.5, 0.65Hz, 1H), 7.01-7.07 (m, 3H), 7.12-7.13 (m, 1H), 7.236-7.238 (m, 1H), 7.38-7.40 (m, 2H), 8.04 (dd, *J* = 4, 1.2 Hz, 1H) ppm; ^{13}C NMR (C_6D_6, 125 MHz) δ 29.4, 32.5, 78.0, 125.3, 126.4, 127.4, 127.6, 127.7, 128.4, 128.5, 129.7, 129.8, 136.3, 139.3, 147.2, 148.4 ppm; IR (film) 3420, 3020, 2930, 2888 cm^{-1}; HRMS calcd for $C_{17}H_{14}$ $(M - H_2O)^+$: 218.1096; found: 218.1089.

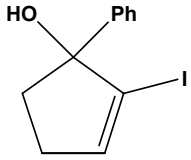


Preparation of 4-phenyl-4,5,6,7-tetrahydro-benzofuran-4-ol (S7). The general procedure A was applied to 6,7-dihydro-*5H*-benzofuran-4-one (12 µl, 0.1 mmol). The

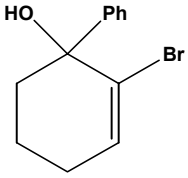


residue was purified by silica gel column chromatography (hexanes/EtOAc, 94:6) to give **S7** (18.5 mg, 81% yield, 71% ee) as an oil: $[\alpha]_D^{20}$ = +1.5 (*c* 1.05, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 1.88-1.91 (m, 1H), 1.98-2.02 (m, 1H), 2.08-2.15 (m, 2H), 2.70-2.77 (m, 2H), 6.16 (d, *J* = 1.8 Hz, 1H), 7.29-7.32 (m, 2H), 7.35-7.38 (m, 2H), 7.44-7.45 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.2, 23.5, 41.8, 73.3, 108.9, 123.0, 126.3, 127.4, 128.4, 141.6, 147.5, 153.6 ppm; IR (film) 3430, 2926, 1772, 1669, 1447 cm^{-1}; HRMS calcd for $C_{14}H_{12}O$ $(M - H_2O)^+$: 196.0888; found: 196.0893.

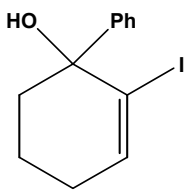
Preparation of 2-iodo-1-phenyl-cyclopent-2-enol (S8). The general procedure A was applied to 2-iodo-2-cyclopenten-1-one (52 mg, 0.25 mmol). The residue was purified by flash silica gel column chromatography (hexanes/EtOAc, 96:4) to give **S8** (32.7 mg, 46% yield, 93% ee) as a wax: $[\alpha]_D^{20}$ = +17.9 (*c* 1.2, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 2.40-2.43 (m, 2H), 2.51-2.55 (m, 1H), 2.61-2.62 (m, 1H), 6.50 (dd, *J* = 2.6, 2.5 Hz, 1H), 7.29-7.34 (m, 1H), 7.38-7.44 (m, 4H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 33.7, 39.7, 89.6, 107.5, 125.4, 127.7, 128.8, 143.2, 145.5 ppm; IR (film) 3448, 2929, 1600, 1492 cm^{-1}; HRMS calcd for $C_{11}H_{11}IO$ $(M)^+$: 285.9855; found: 285,9844; HRMS calcd for $C_{11}H_9I$ $(M - H_2O)^+$: 267.97495; found: 267.9750.



Preparation of 2-bromo-1-phenyl-cyclohex-2-enol (S9). The general procedure A was applied to 2-bromo-2-cyclohexen-1-one (26.3 mg, 0.15 mmol). The residue was purified by flash silica gel column chromatography (hexanes/EtOAc, 96:4) to give **S9** (25 mg, 66% yield, 94% ee) as an oil : $[\alpha]_D^{20}$ = –1.4 (*c* 1.2, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 1.50-1.51 (m, 1H), 1.51-1.52 (m, 1H), 2.00-2.02 (m, 1H), 2.09-2.16 (m, 3H), 6.38 (dd, *J* = 4.1, 4.0 Hz, 1H), 7.20-7.23 (m, 1H), 7.28-7.31 (m, 2H), 7.41-7.42 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.2, 28.4, 41.0, 77.0, 126.3, 127.8, 128.6, 129.4, 134.3, 145.9 ppm; IR (film) 3446, 2931, 1446, 1326 cm^{-1}; HRMS calcd for $C_{12}H_{12}Br$ $(M - OH)^+$: 235.0123; found: 235.0098.



Preparation of 2-iodo-1-phenyl-cyclohex-2-enol (S10). The general procedure A was applied to 2-iodo-2-cyclohexen-1-one (22.2 mg, 0.1 mmol). The residue was purified by flash silica gel column chromatography (hexanes/EtOAc, 96:4) to give **S10** (23 mg, 77% yield, 93% ee) as an oil : $[\alpha]_D^{20}$ = +16.4 (*c* 1.24, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz) δ 1.66-1.67 (m, 1H), 1.80-1.82 (m, 1H), 2.16-2.29 (m, 4H) 6.79 (dd, *J* = 3.9, 3.8 Hz, 1H), 7.33-7.35 (m, 1H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.50-7.51 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) δ 19.2, 30.0, 40.0, 77.6, 110.8, 126.3, 127.9, 128.5, 142.5, 146.7 ppm; IR (film) 3461, 2936, 2865, 1490, 1446 cm^{-1}; HRMS calcd for $C_{12}H_{13}IO$ $(M)^+$: 300.0011; found: 299.9997.



Conditions for the Determination of ee.

The racemic alcohols were prepared by addition of phenylmagnesium bromide to the corresponding ketone. Only the tertiary alcohol **S2** was analyzed by chiral capillary GC. The specifications for the GC analyses were as follows: fused silica chiral capillary column (Supelco β-Dex 120): 30 m x 0.25 mm (inner diameter) x 0.25 μm film thickness; carrier gas, nitrogen; inlet temperature, 250℃; flame ionization detector, 270℃. The conditions for the resolution of the racemates by GC are given below.

2,4,4-Trimethyl-1-phenyl-cyclohex-2-enol (S2). T_1 = 51.6 min, T_2 = 56.6 min (140℃, 1.0 ml/min).

Chiral HPLC analyses of **S1**, **S3-S5** and **S7-S10** were performed using a Chiralcel OD-H column; the analysis of **S6** was performed using a Chiralpak AS column. The conditions for the resolution of the racemates are described below.

1-Phenyl-cyclohex-2-enol (S1). T_1 = 16.5 min, T_2 = 18.8 min (hexane/2-propanol, 97:3, 0.5 ml/min).

2-(*tert*-Butyldimethylsilanyloxymethyl)-1-phenyl-cyclohex-2-enol (S3). T_1 = 35.1 min, T_2 = 37 5 min (hexane/2-propanol, 99.98:0.02, 0.2 ml/min).

2-Methyl-1-phenyl-cyclopent-2-enol (S4). T_1 = 17.8 min, T_2 = 21.1 min (hexane/2-propanol, 98:2, 0.5 ml/min).

2-Pentyl-1-phenyl-cyclopent-2-enol (S5). T_1 = 18.9 min, T_2 = 22.5 min (hexane/2-propanol, 99:1, 0.5 ml/min).

5-Phenyl-8,9-dihydro-5*H*-benzocyclohepten-5-ol (S6). T_1 = 52.8 min, T_2 = 59.2 min (hexane/2-propanol, 99.98:0.02, 0.3 ml/min).

4-Phenyl-4,5,6,7-tetrahydro-benzofuran-4-ol (S7). T_1 = 29.9 min, T_2 = 37.4 min (hexane/2-propanol, 99.5:0.5, 1.0 ml/min).

2-Iodo-1-phenyl-cyclopent-2-enol (S8). T_1 = 17.7 min, T_2 = 21.6 min (hexane/2-propanol, 97:3, 0.5 ml/min).

2-Bromo-1-phenyl-cyclohex-2-enol (S9). T_1 = 15.9 min, T_2 = 19.9 min (hexane/2-propanol, 97:3, 0.4 ml/min).

2-Iodo-1-phenyl-cyclohex-2-en-ol (S10). T_1 = 23.0 min, T_2 = 27.9 min (hexane/2-propanol, 97:3, 0.3 ml/min).

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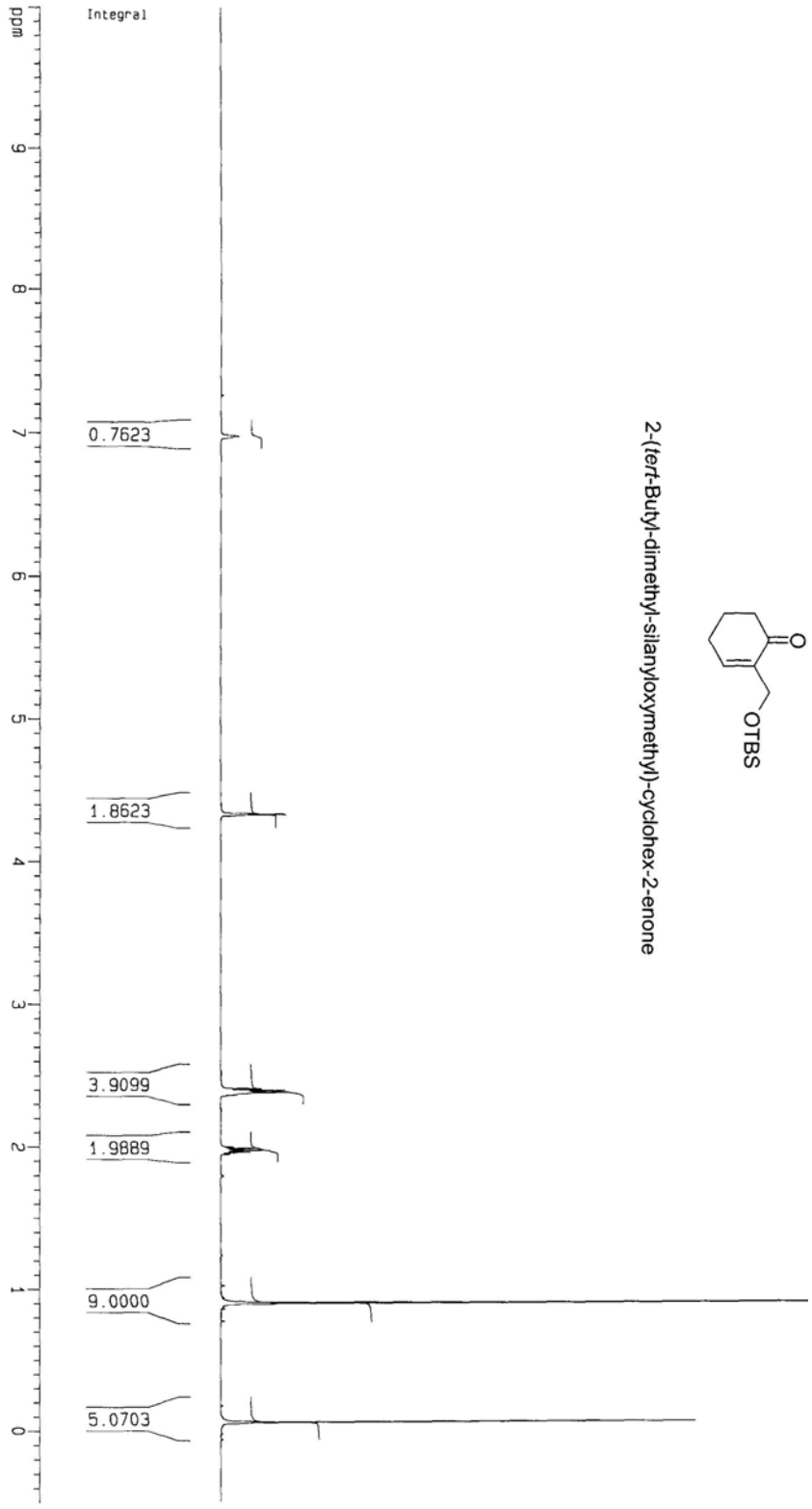
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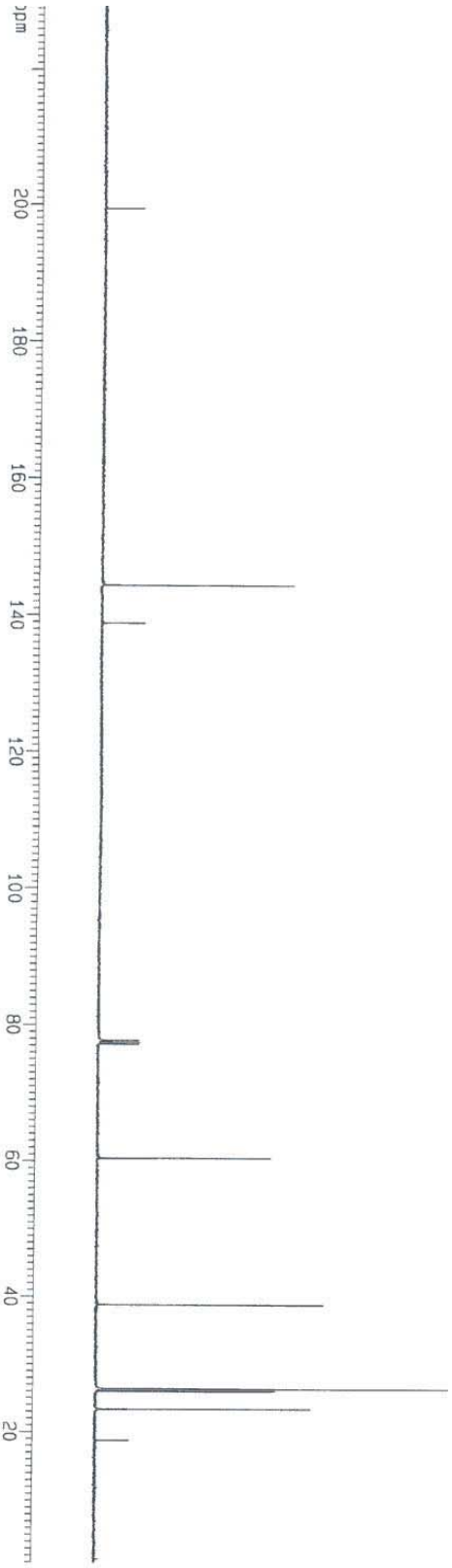
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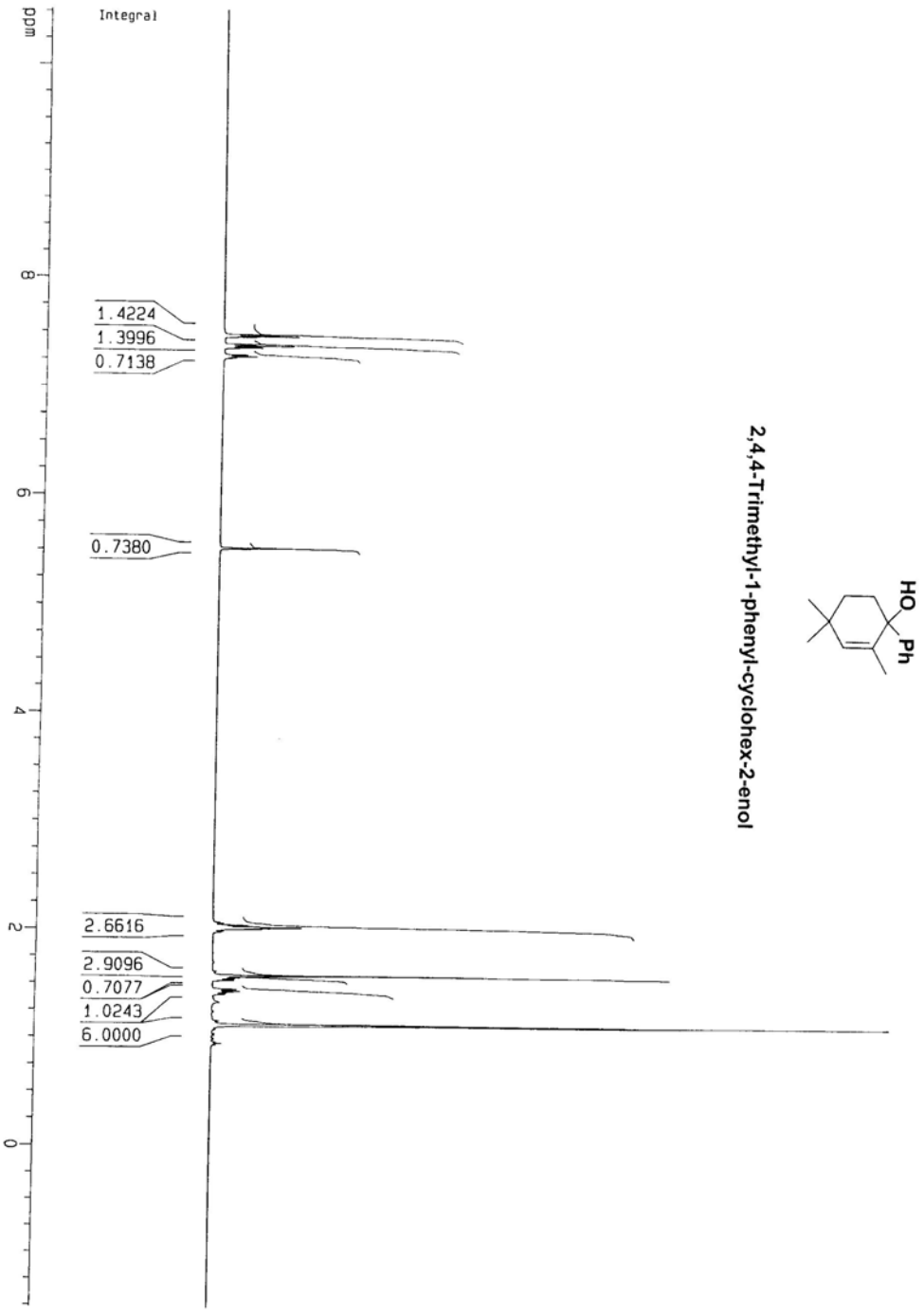
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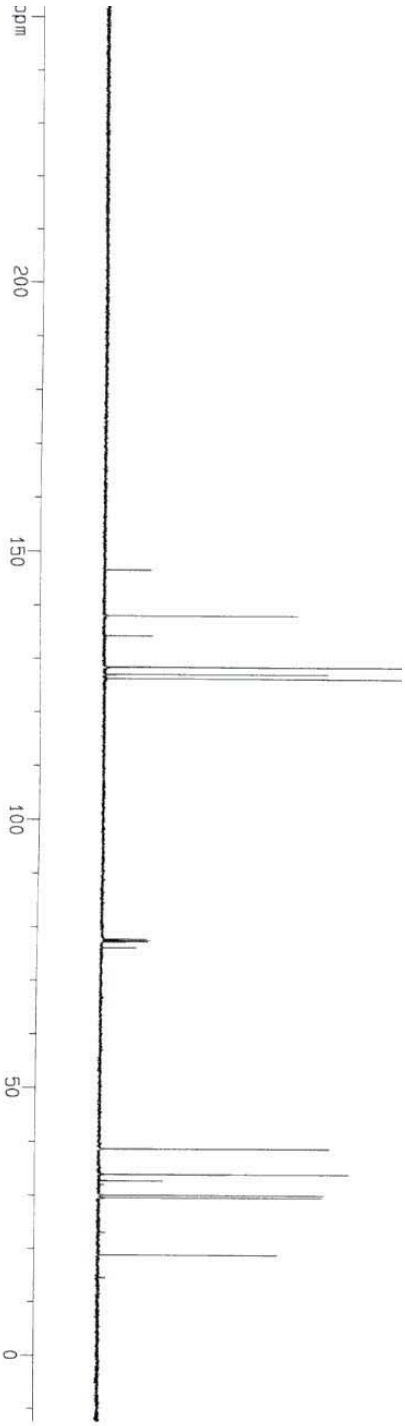
Ph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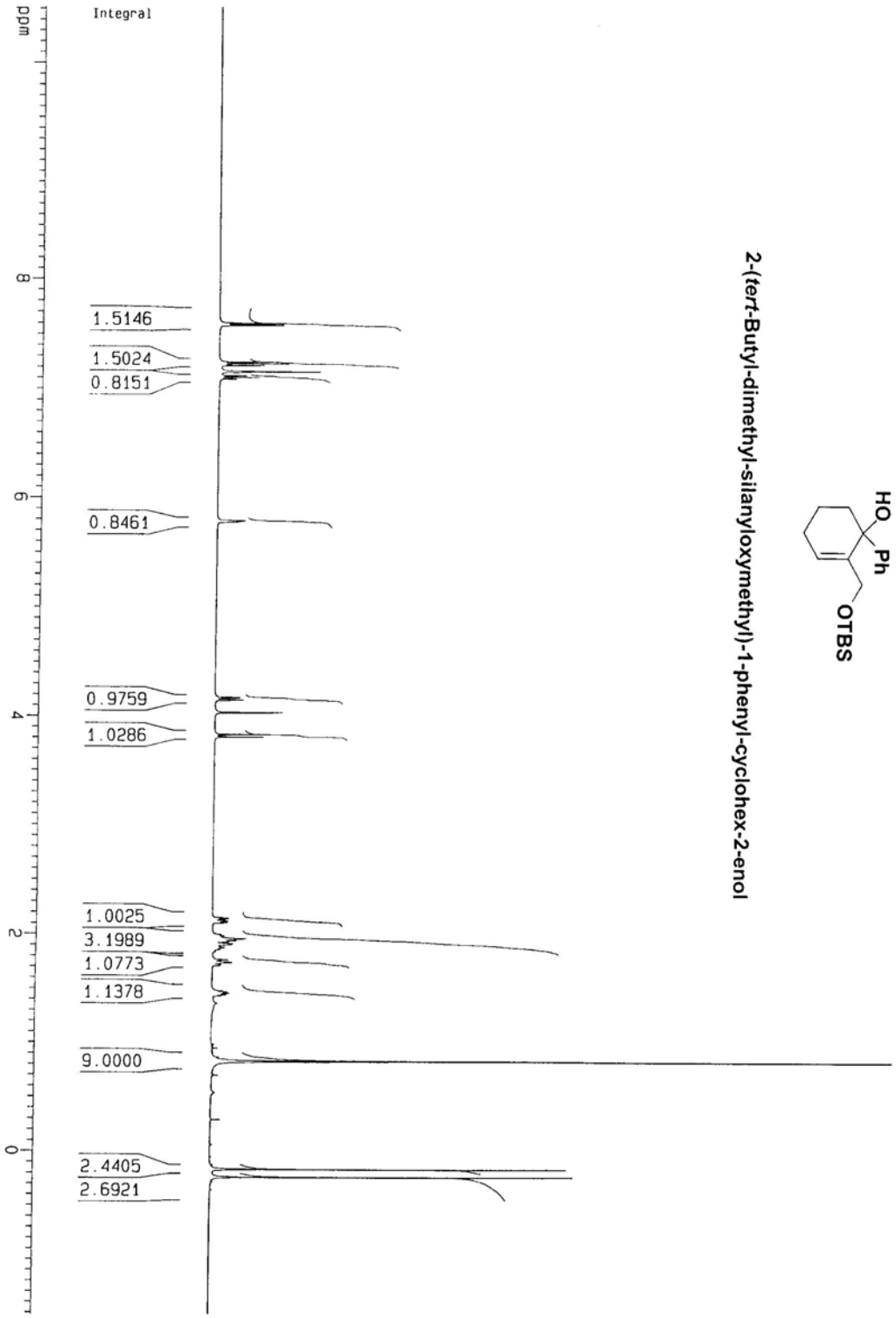
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1.0025
3.1989
1.0773
1.1378
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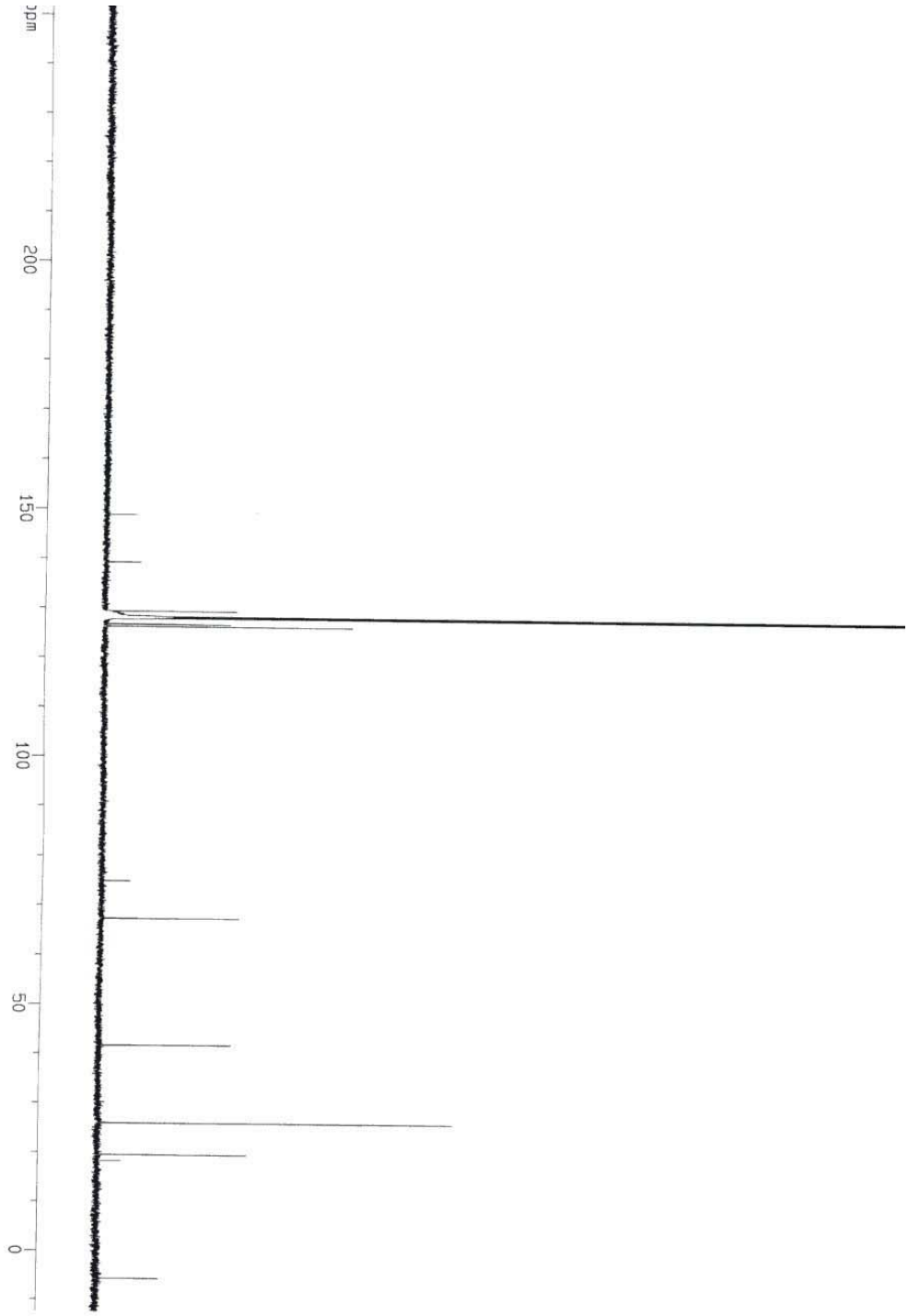
2.6921
HO
Ph
OTBS
2-(tert-Butyl-dimethyl-silanyloxymethyl)-1-phenyl-cyclohex-2-enol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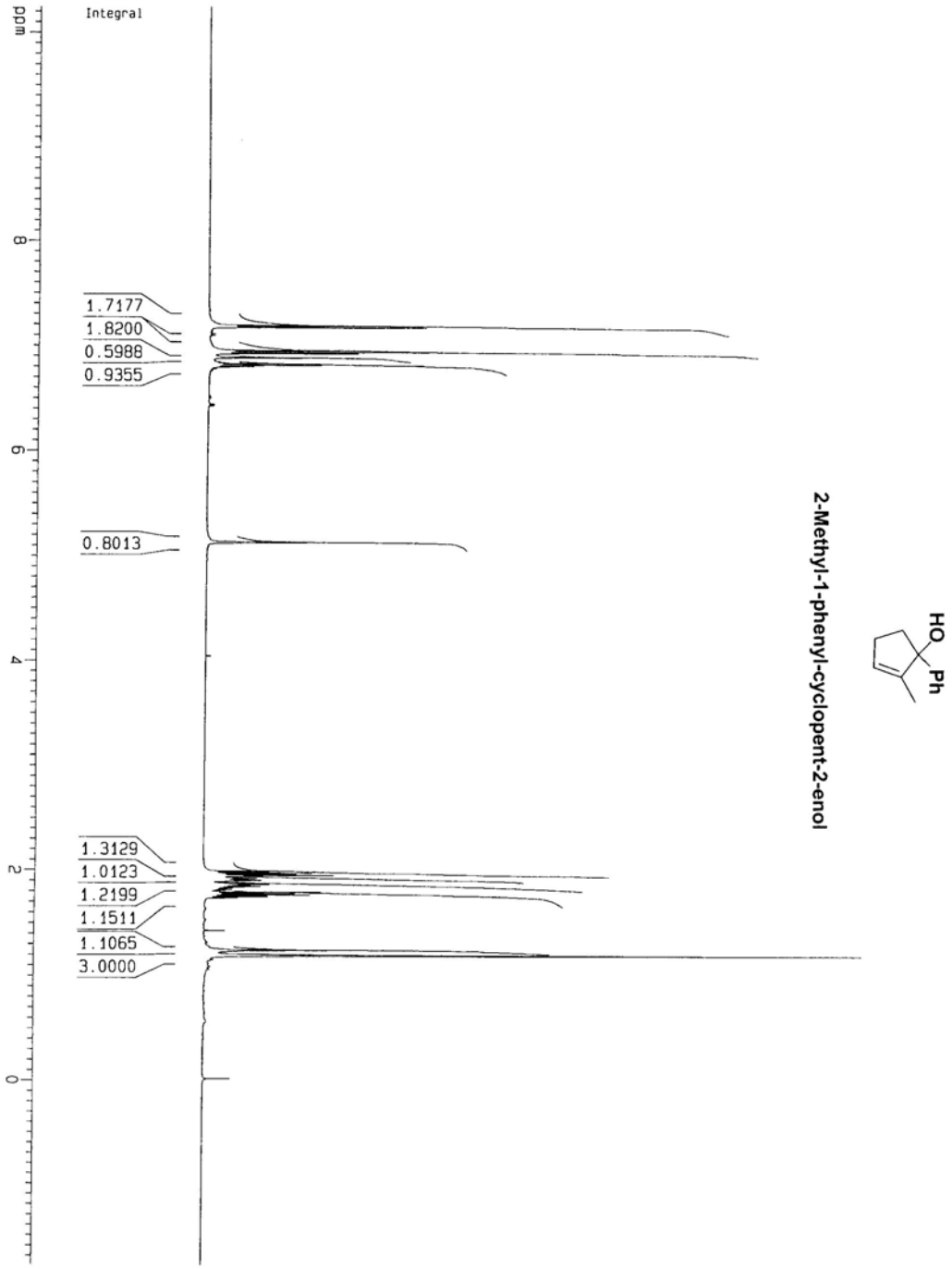
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4
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0.9355
0.8013
1.3129
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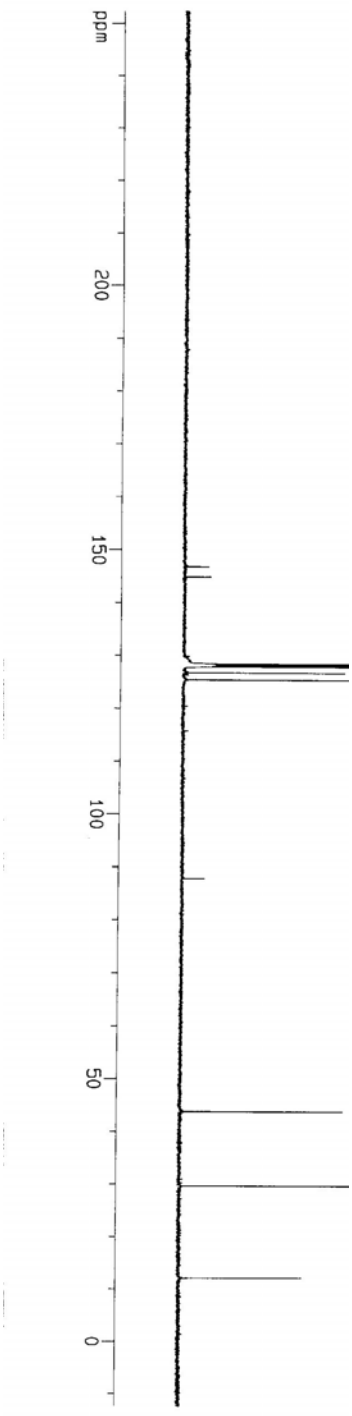
1.2199
1.1511
1.1065
3.0000
2-Methyl-1-phenyl-cyclopent-2-enol
HO
Ph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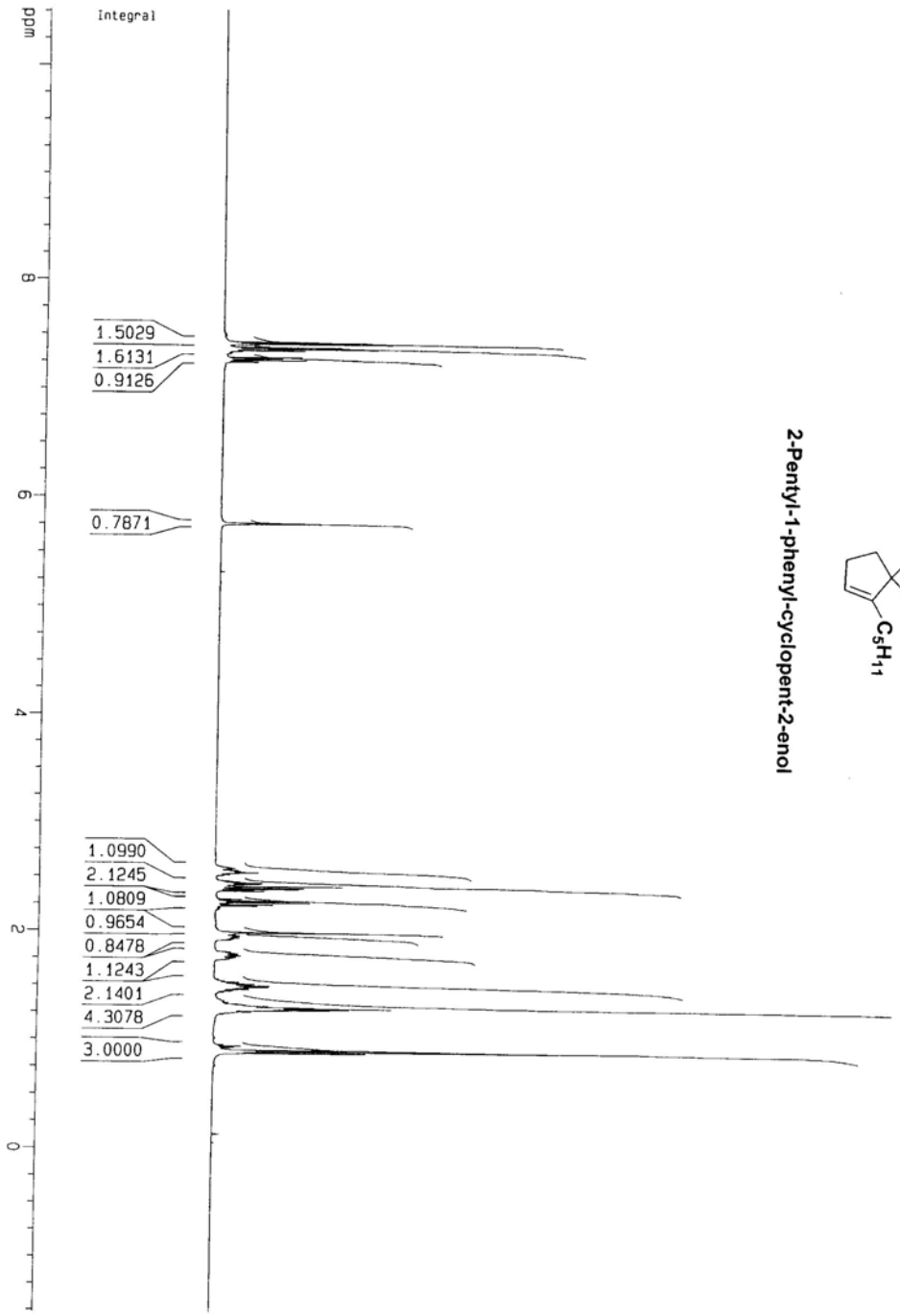
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4
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0.9126
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0.9654
0.8478
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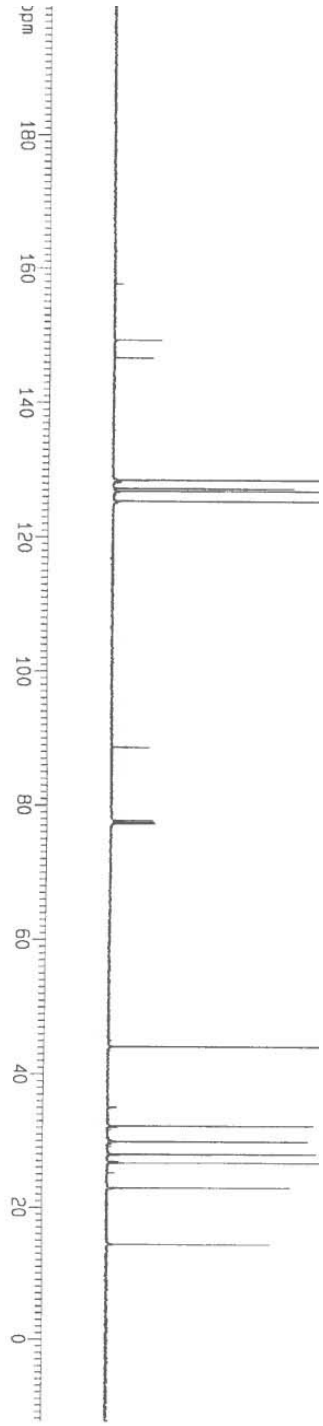
2.1401
4.3078
3.0000
2-Pentyl-1-phenyl-cyclopent-2-enol
HO
Ph
C5H11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120
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80
60
40
20
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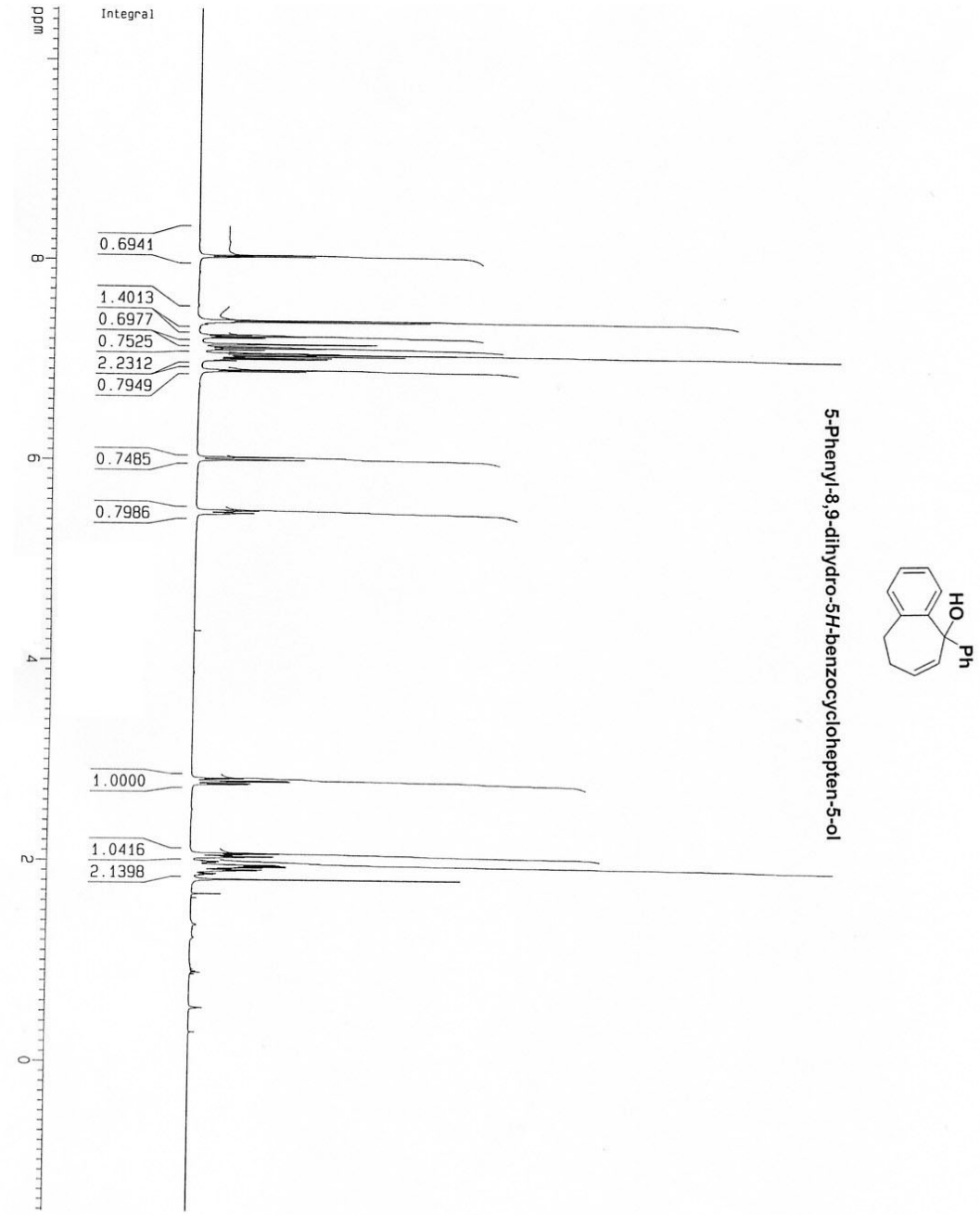
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4
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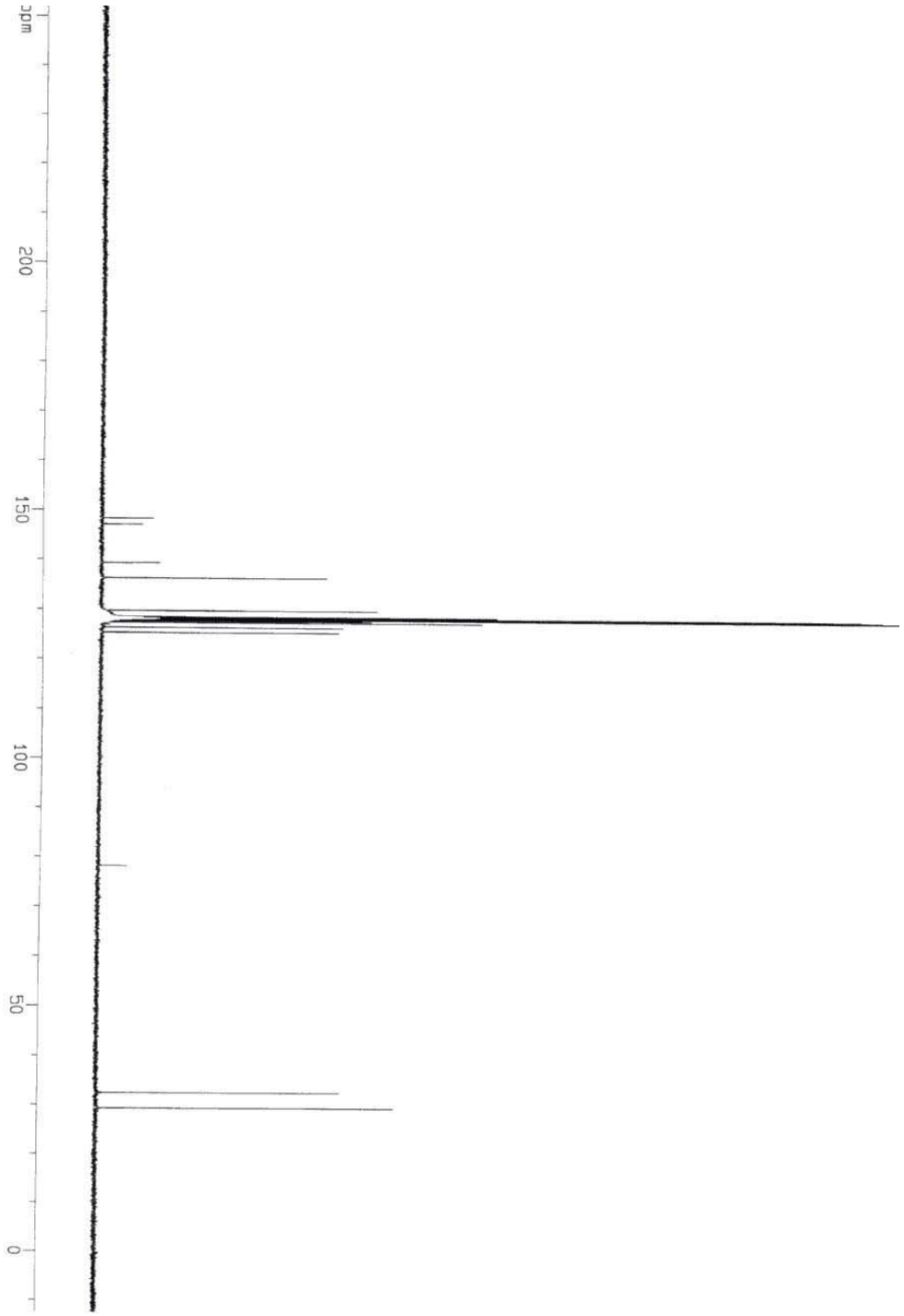
1.0416
2.1398
5-Phenyl-8,9-dihydro-5H-benzocyclohepten-5-ol
HO
Ph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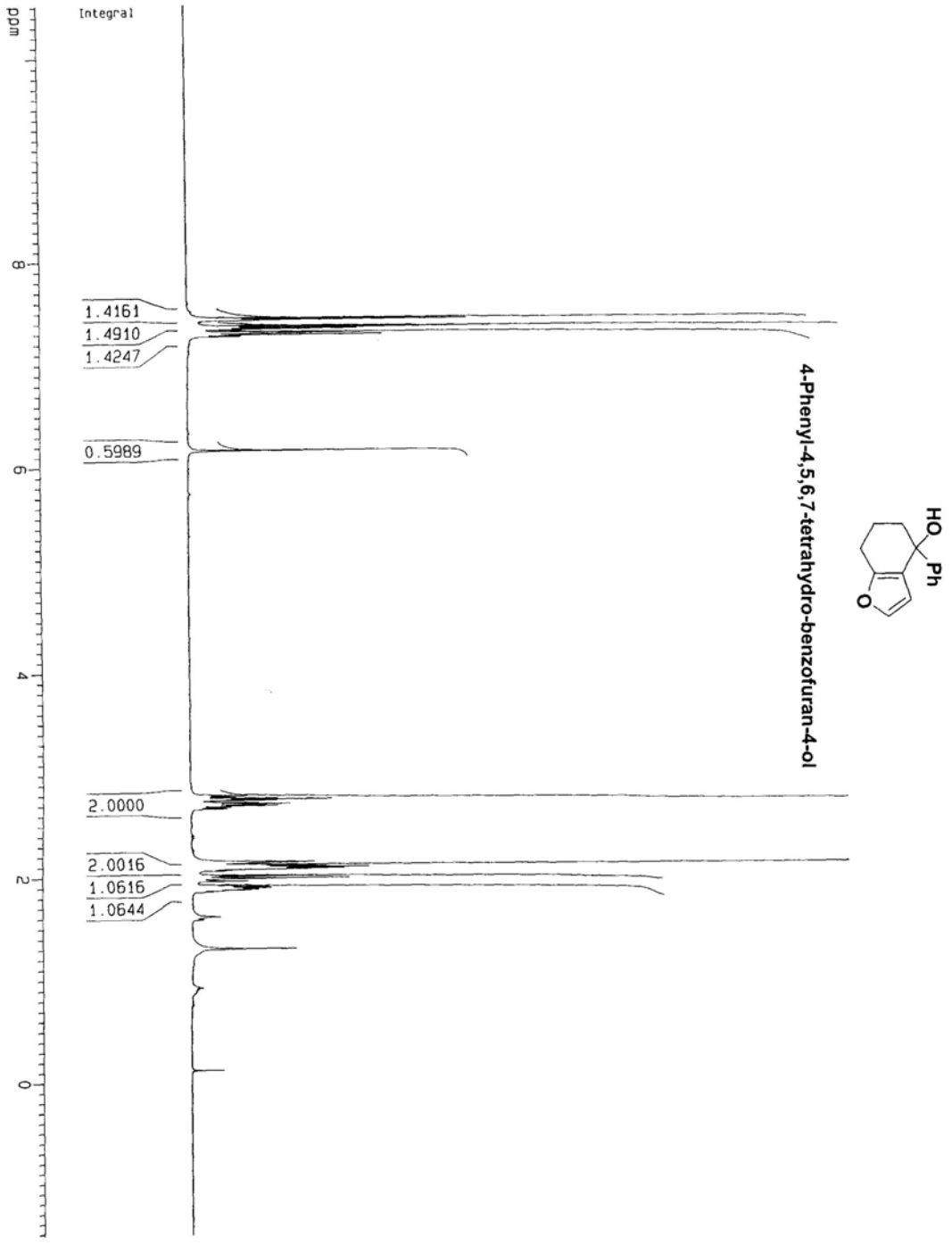
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2.0016
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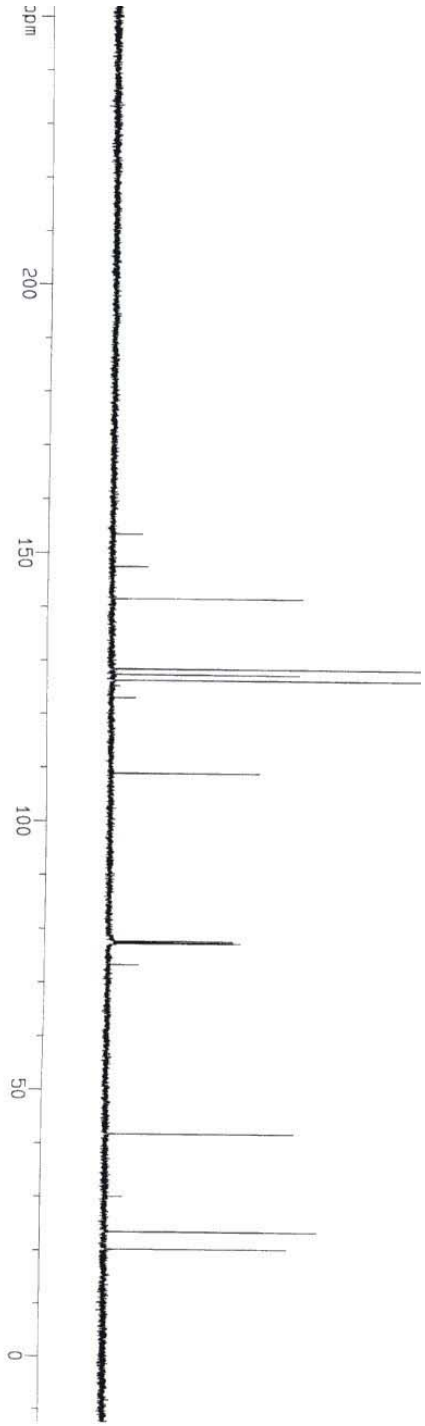
ppm
8
6
4
2
0
4-Phenyl-4,5,6,7-tetrahydro-benzofuran-4-ol
HO
Ph
O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50
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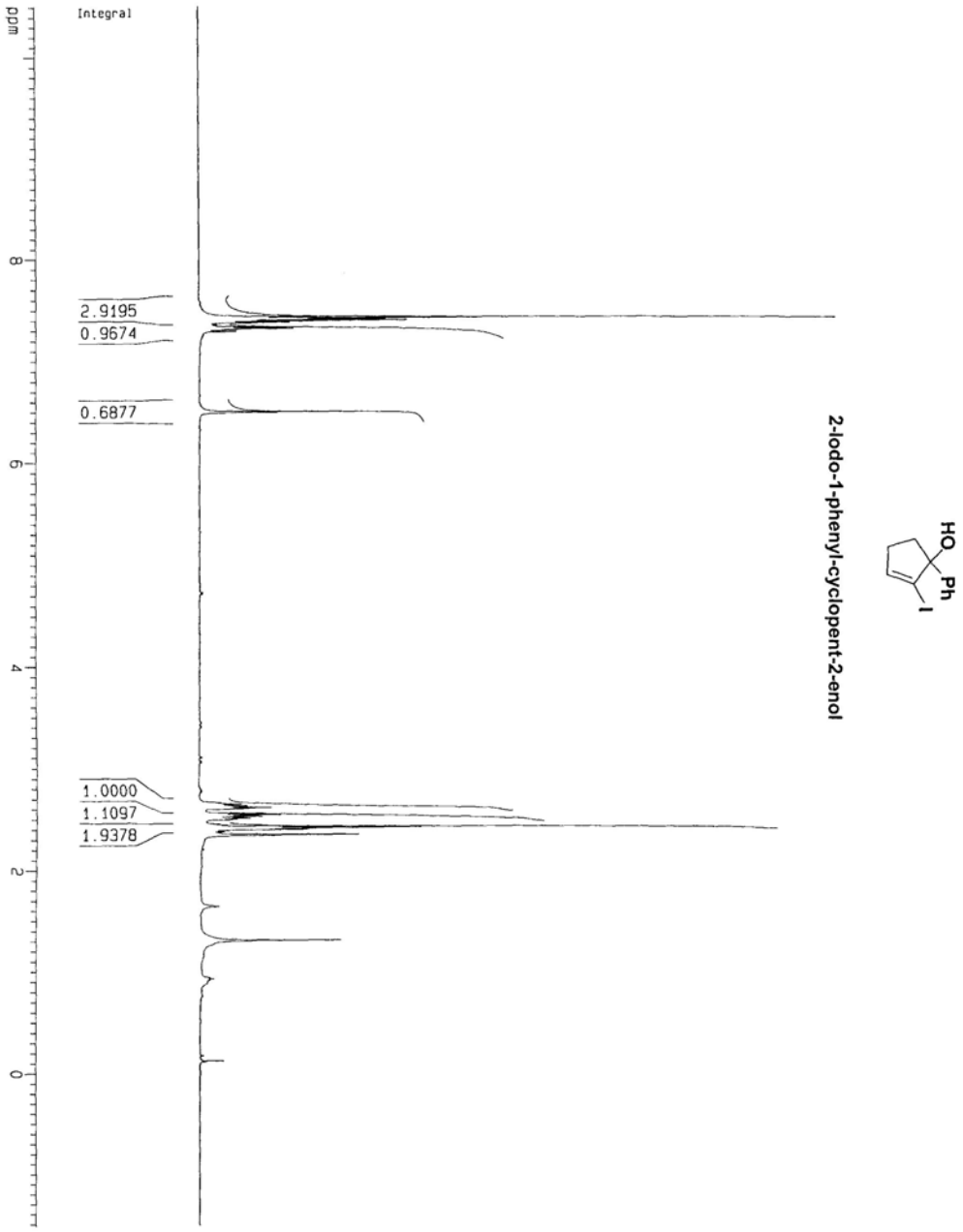
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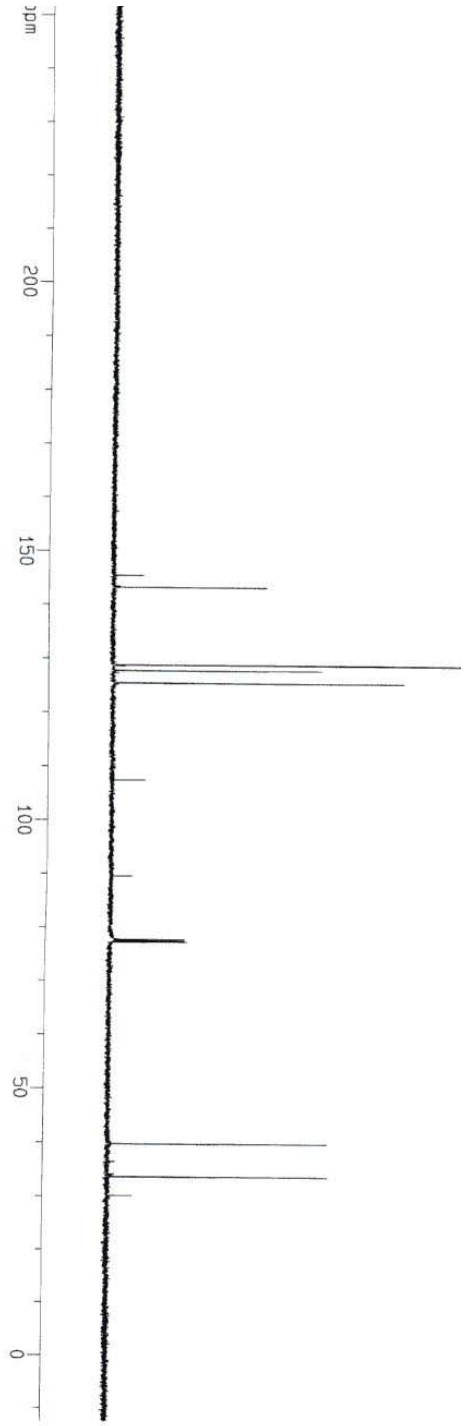
ppm
8
6
4
2
0
2-Iodo-1-phenyl-cyclopent-2-enol
HO
Ph
I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100
50
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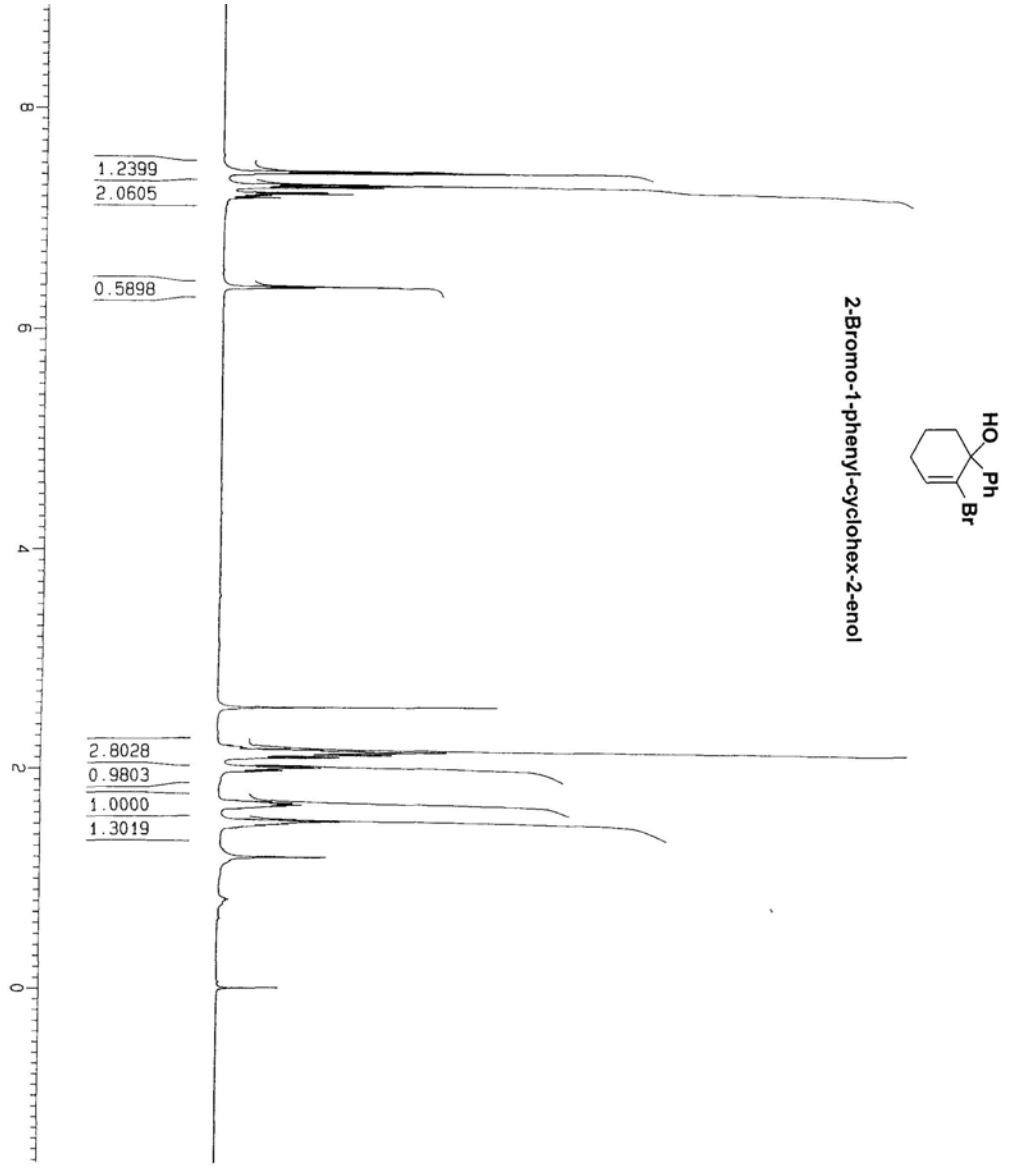
1.2399
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0.5898
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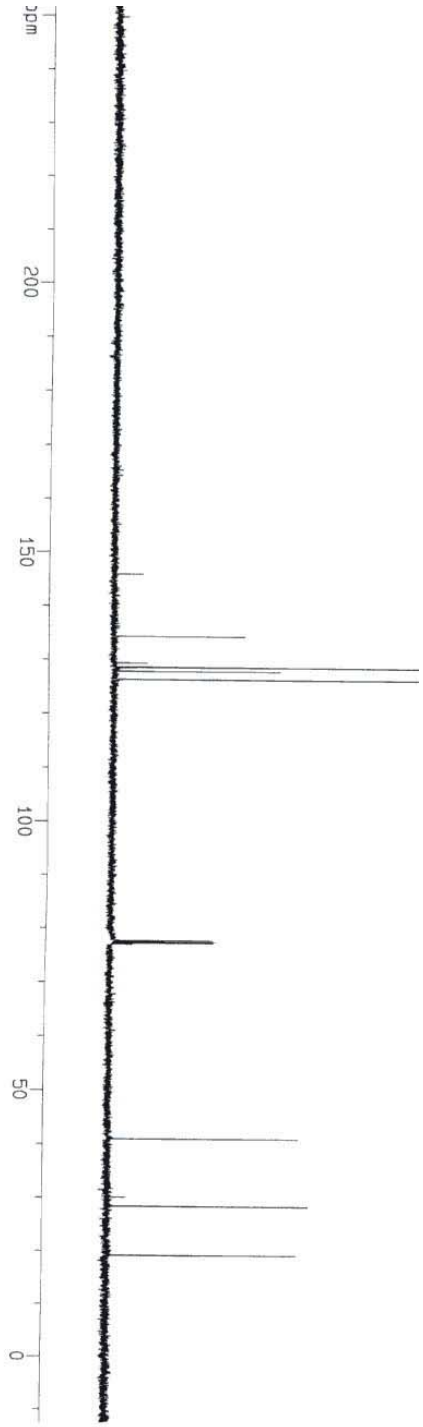
0.9803
1.0000
1.3019
2-Bromo-1-phenyl-cyclohex-2-enol
HO
Ph
Br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50
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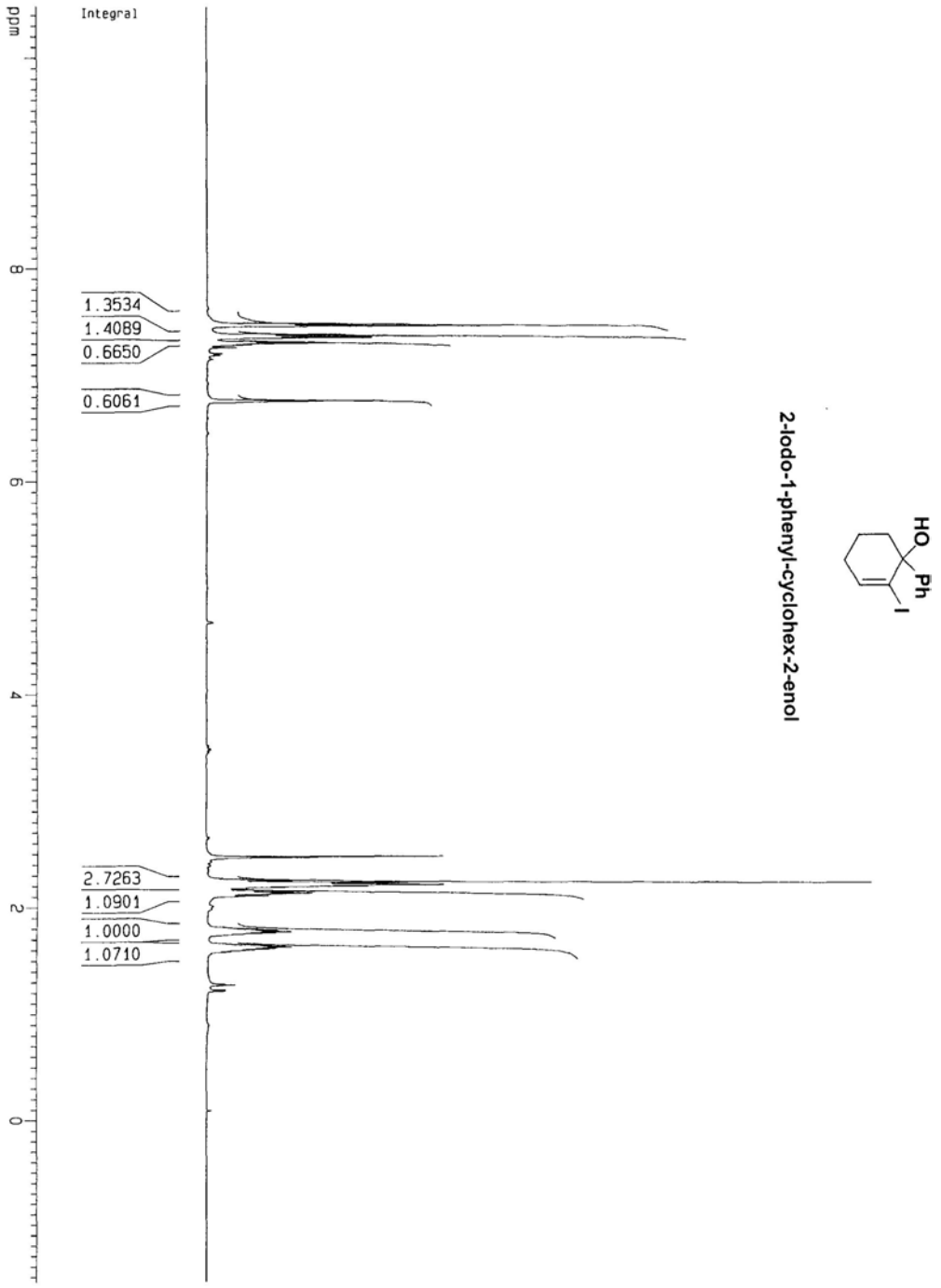
Integral
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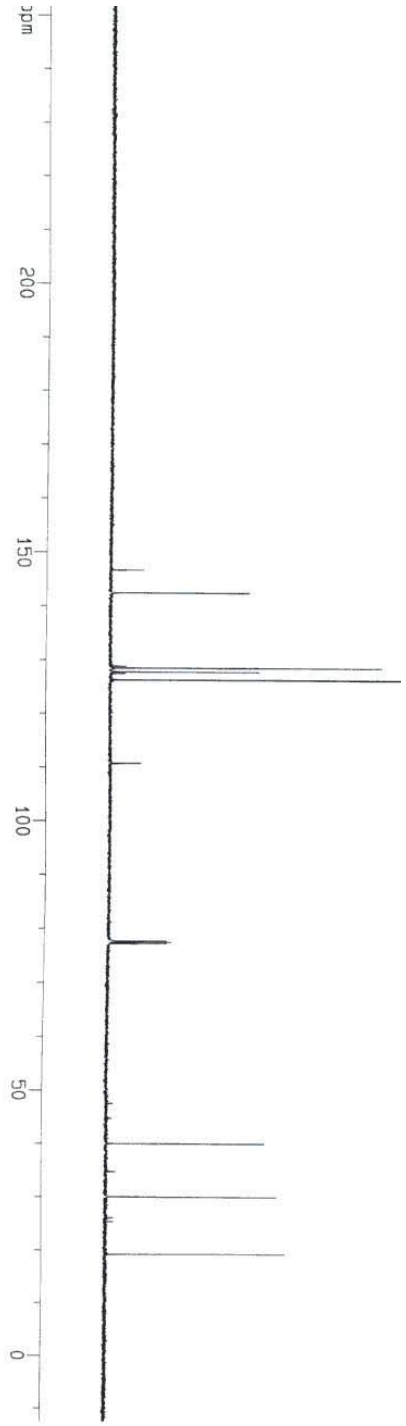
1.0901
1.0000
1.0710
2-Iodo-1-phenyl-cyclohex-2-enol
HO
Ph
I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50
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