# Pyrrolidine-thiourea as a Bifunctional Organocatalyst: Highly Enantioselective Michael Addition of Cyclohexanone to Nitroolefins

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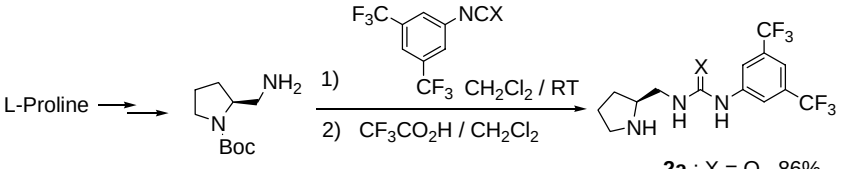
## *Supporting Information*

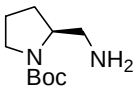
Contents

**General Methods.** Commercial Reagents were used as received, unless otherwise noted. All reactions unless otherwise noted were carried out directly under air. Benzene and THF was distilled over calcium hydride. *i*-PrOH, MeOH and cyclohexanone were distilled before use. Nitroolefins were prepared according to literature procedures.[1, 2]

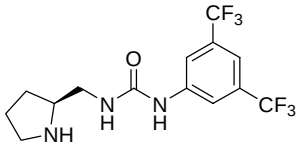
**Procedures for the preparation of pyrrolidine-urea (thiourea) organocatalysts 2a and 2b**.




**2a** : X = O 86%
**2b** : X = S 80%



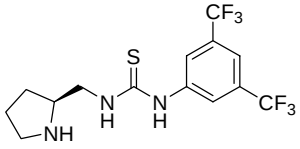

**(S)-tert-butyl 2-(aminomethyl)pyrrolidine-1-carboxylate 1**. Compound **1** is prepared according to the known procedures in 4 steps from L-proline. [3]




**(S)-1-(3,5-bis(trifluoromethyl)phenyl)-3-(pyrrolidin-2-ylmethyl)urea (2a)**：

To a stirred solution of **1** (888.6 mg, 4.35 mmol) in dry $CH_2Cl_2$ (30 mL) was added 3,5-bis(trifluoromethyl)phenyl isocyanate (0.8 mL, 4.35 mmol) at room temperature. The reaction mixture was allowed to stir for 12 h. The solvent was removed under reduced pressure to give the crude product (1.737 g, 86%), which was used directly in the following step without further purification.

The N-Boc-derivative (145 mg, 0.32 mmol) was dissolved in a mixture of trifluoroacetic acid and dichloromethane (8 mL, V/V = 1:1) and the solution was stirred for 2 h at room temperature, at which time the solvent was removed under reduced pressure. The pH was adjusted to 8 with aqueous $NaHCO_3$. The mixture was extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried with $Na_2SO_4$. Concentrated and purified by flash column chromatography (MeOH/EtOAc = 7/1) to give thiourea **2a** as a yellow solid (114 mg, 99%). Mp. 205 ºC; $[\alpha]_D^{20}$ = +4.4 (c = 0.885, $CHCl_3$); $^1$H NMR (400 MHz, $CD_3OD$): δ 8.07 (s, 2H), 7.47 (s, 1H), 3.80-3.68 (m, 1H), 3.54-3.50 (m, 2H), 3.35-3.27 (m, 2H), 2.16-2.00 (m, 3H), 1.82-1.77 (m, 1H). $^{13}$C NMR (100 MHz, $CD_3OD$): 158.8, 143.7, 133.6 (q, *J* = 32.7 Hz), 125.3 (q, *J* = 270.2 Hz), 119.7 (d, *J* = 3.2 Hz), 116.2 (t, *J* = 3.8 Hz), 63.1, 47.1, 42.7, 28.9, 25.0; IR (neat): 3306, 2970, 1680, 1573, 1388, 1279, 1127, 882, 682 $cm^{-1}$; MS (ESI, *m/z*): 356 ($M+H^+$)；HRMS (ESI): Calcd for $C_{14}H_{16}N_3OF_6^+$: 356.1190. Found: 356.1192.




**(S)-1-(3,5-bis(trifluoromethyl)phenyl)-3-(pyrrolidin-2-ylmethyl)thiourea 2b** ：

yield : 80%. Mp. 102 ºC; $[\alpha]_D^{20}$ = -26.7 (c = 0.695, $CHCl_3$); $^1$H NMR (400 MHz, $CD_3OD$): δ 8.10 (s, 2H), 7.51 (s, 1H), 3.86-3.78 (m, 3H), 3.25-3.20 (m, 1H), 3.17-3.13 (m, 1H), 2.10-2.00 (m, 1H), 1.97-1.87 (m, 2H), 1.72-1.55 (m, 1H). $^{13}$C NMR (100 MHz, $CD_3OD$): 184.6, 143.2, 133.0 (q, *J* = 33.2 Hz), 125.0 (q, *J* = 270.3 Hz), 124.5 (d, *J* = 3.1 Hz), 118.6 (t,

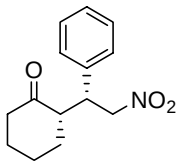
*J* = 3.5 Hz), 62.0, 46.9, 46.0, 28.9, 24.7; IR (neat): 3244, 2956, 1605, 1553, 1538, 1473, 1385, 1278, 1131, 682 $cm^{-1}$; MS (ESI, *m/z*): 372 ($M+H^+$)；HRMS (ESI): Calcd for $C_{14}H_{16}N_3SF_6^+$: 372.0963. Found: 372.0964.

**Typical Procedure for Michael Addition Reactions**:

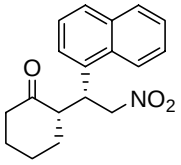
**Procedure A** (in solvent): To a solution of catalyst **2a** or **2b** (0.05 mmol) and *n*-butyric acid (0.0025 mmol) in solvent (1 mL) was added cyclohexanone (0.25 mL, 10 eq) at required temperature and the resulting mixture was stirred for 15 min, nitroolefin (0.25 mmol, 1 eq) was added. After the reaction is complete monitored by TLC, the mixture was concentrated under reduced pressure, the resulting residue was then purified by flash chromatography (PE: EtOAc = 1/5) to give the product.

**Procedure B** (in neat): The catalyst **2a** or **2b** (0.05 mmol) and n-butyric acid (0.0025 mmol) in cyclohexanone (0.5 mL, 20 eq) at required temperature and the resulting mixture was stirred for 15 min, nitroolefin (0.25 mmol, 1 eq) was added. After the reaction is complete monitored by TLC, the mixture was concentrated under reduced pressure, the resulting residue was then purified by flash chromatography (PE: EtOAc = 1/5) to give the product.

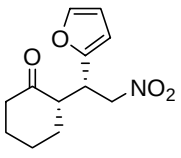
Relative and absolute configurations of the products were determined by comparison with the known $^1$H NMR, chiral HPLC analysis, and optical rotation values. Compounds reported in Table 2, entries **1**[4], **2**[5], **3**[6], **6**[7], **8**[8], **9**[9], **10**[10] are known.




**(S)-2-((R)-2-nitro-1-phenylethyl)cyclohexanone 5a**[6]：**(Procedure B)** reaction time: 38 h (99% yield); ee was determined by HPLC analysis (Chiralcel AS-H, *i*-PrOH/hexane = 10/90, 0.7 mL/min, 238 nm; $t_r$ (minor) = 23.88 min, $t_r$ (major) = 36.38 min), 90% ee, $[\alpha]_D^{20}$ = -26.4 (c = 0.945, $CHCl_3$); syn/anti = 96/4; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.34-7.23 (m, 3H), 7.16 (d, *J* = 6.9 Hz, 2H), 4.95 (dd, *J* = 4.5, 12.6 Hz, 1H), 4.63 (dd, *J* = 9.9, 12.3 Hz, 1H), 3.76 (dt, *J* = 4.5, 9.9 Hz, 1H), 2.68 (ddd, *J* = 8.1, 8.4, 11.7 Hz, 1H), 2.49-2.33 (m, 2H), 2.10-2.04 (m, 1H), 1.79-1.52 (m, 4H), 1.52-1.19 (m, 1H).

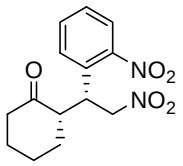



**(S)-2-((R)-1-(naphthalen-1-yl)-2-nitroethyl)cyclohexanone 5b**[5]：**(Procedure B)** reaction time: 60 h (93% yield); ee was determined by HPLC analysis (Chiralcel AS, *i*-PrOH/hexane = 30/70, 0.7 mL/min, 238 nm; $t_r$ (minor) = 11.03 min, $t_r$ (major) = 14.45 min), 95% ee, $[\alpha]_D^{20}$ = -101.2 (c = 1.174, $CHCl_3$); syn/anti = 99/1; $^1$H NMR (300 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.85 (d, *J* = 8.1 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.58-7.36 (m, 4H), 5.07 (dd, *J* = 4.2, 12.6 Hz, 1H), 4.91 (m, 1H), 4.76 (br s, 1H), 2.85 (br s, 1H), 2.52-2.39 (m, 2H), 2.10-2.04 (m, 1H), 1.69-1.47 (m, 4H), 1.30-1.19 (m, 1H).

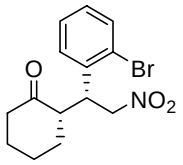



**(S)-2-((S)-1-(furan-2-yl)-2-nitroethyl)cyclohexanone 5c**[4]：**(Procedure B)** reaction time: 36 h (93% yield); ee was determined by HPLC analysis (Chiralcel AD, *i*-PrOH/hexane = 10/90, 0.7 mL/min, 220 nm; $t_r$ (major) = 15.77 min, $t_r$

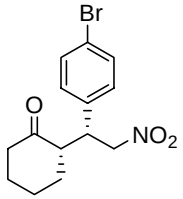
(minor) = 19.03 min), 89% ee, $[\alpha]_D^{20}$ = -10.7 (c = 1.132, $CHCl_3$); syn/anti = 91/9; $^1H$ NMR (300 MHz, $CDCl_3$): δ 7.35 (m, 1H), 6.28 (dd, *J* = 1.8, 3.3 Hz, 1H), 6.18 (d, *J* = 3.3 Hz, 1H), 4.83-4.63 (m, 2H), 3.97 (dt, *J* = 4.8, 9.3 Hz, 1H), 2.80-2.71 (m, 1H), 2.50-2.31 (m, 2H), 2.14-2.04 (m, 1H), 1.88-1.56 (m, 4H), 1.35-1.21 (m, 1H).



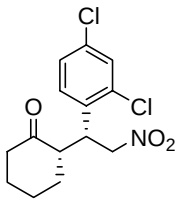

**(S)-2-((R)-2-nitro-1-(2-nitrophenyl)ethyl)cyclohexanone 5d** ：(**Procedure B**) reaction time: 38 h (95% yield); ee was determined by HPLC analysis (Chiralcel AD, *i*-PrOH/hexane = 5/95, 0.9 mL/min, 238 nm; $t_r$ (minor) = 51.63 min, $t_r$ (major) = 63.35 min), 97% ee, $[\alpha]_D^{20}$ = -153.7 (c = 1.105, $CHCl_3$); syn/anti = 96/4; $^1H$ NMR (300 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 1.2, 8.4 Hz, 1H), 7.64-7.58 (m, 1H), 7.49-7.41 (m, 2H), 4.98-4.83 (m, 2H), 4.38 (dt, *J* = 4.2, 9.0 Hz, 1H), 2.98-2.87 (m, 1H), 2.50-2.34 (m, 2H), 2.17-2.07 (m, 1H), 1.85-1.61 (m, 5H). $^{13}C$ NMR (75 MHz, $CDCl_3$):211.1, 150.7, 133.1, 132.8, 129.1, 128.6, 124.9, 77.6, 52.1, 42.8, 38.6, 33.2, 28.3, 25.3; IR (film): 2944, 2864, 1707, 1552, 1527, 1358, 855, 781 $cm^{-1}$; MS (ESI, m/z): 310 ($M+NH_4^+$); HRMS (ESI): Calcd for $C_{14}H_{16}N_2O_5Na^+$: 315.0960. Found: 315.0951.



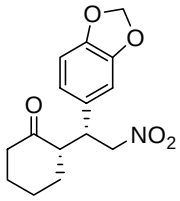

**(S)-2-((R)-1-(2-bromophenyl)-2-nitroethyl)cyclohexanone 5e** ：**(Procedure B)** reaction time: 29 h (88% yield); ee was determined by HPLC analysis (Chiralcel AS, *i*-PrOH/hexane = 10/90, 0.8 mL/min, 238 nm; $t_r$ (minor) = 16.64 min, $t_r$ (major) = 20.81 min), 96% ee, $[\alpha]_D^{20}$ = -52.9 (c = 1.610, $CHCl_3$); syn/anti = 99/1; $^1H$ NMR (300 MHz, $CDCl_3$): δ 7.47 (dd, *J* = 1.2, 8.1 Hz, 1H), 7.23-7.13 (m, 2H), 7.06-7.00 (m, 1H), 4.87-4.73 (m, 2H), 4.30-4.22 (m, 1H), 2.78 (m, 1H), 2.38-2.28 (m, 2H), 2.03-1.97 (m, 1H), 1.72-1.45 (m, 4H), 1.30-1.26 (m, 1H). $^{13}C$ NMR (75 MHz, DMSO-$d_6$): 211.0, 137.5, 132.5, 128.7, 128.4, 127.9, 125.0, 77.7, 51.3, 41.8, 41.2, 31.7, 27.7, 24.2; IR (neat): 2941, 2862, 1707, 1551, 1379, 1024, 753 $cm^{-1}$; MS (ESI, *m/z*): 327 ($M+H^+$)；Anal. Calcd for $C_{14}H_{16}BrNO_3$: C, 51.55; H, 4.94; N, 4.29. Found: C, 51.72; H, 5.01; N, 4.11.



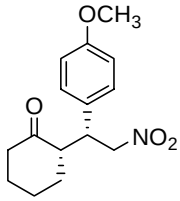

**(S)-2-((R)-1-(4-bromophenyl)-2-nitroethyl)cyclohexanone 5f**[7]: **(Procedure B)** reaction time: 38 h (90% yield); ee was determined by HPLC analysis (Chiralcel AS, *i*-PrOH/hexane = 10/90, 1.0 mL/min, 238 nm; $t_r$ (minor) = 15.52 min, $t_r$ (major) = 22.95 min), 95% ee, $[\alpha]_D^{20}$ = -22.6 (c = 1.296, $CHCl_3$); syn/anti = 95/5; $^1H$ NMR (300 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.7 Hz, 2H), 7.06 (d, *J* = 8.7 Hz, 2H), 4.93 (dd, *J* = 4.5, 12.6 Hz, 1H), 4.60 (dd, *J* = 10.2, 12.6 Hz, 1H), 3.75 (dt, *J* = 4.5, 9.9 Hz, 1H), 2.69-2.60 (m, 1H), 2.51-2.32 (m, 2H), 2.15-2.05 (m, 1H), 1.85-1.58 (m, 4H), 1.30-1.16 (m, 1H).



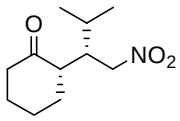
**(S)-2-((R)-1-(2,4-dichlorophenyl)-2-nitroethyl)cyclohexanone 5g** : (**Procedure B**) reaction time: 49 h (89% yield); ee was determined by HPLC analysis (Chiralcel AS, *i*-PrOH/hexane = 10/90, 0.8 mL/min, 238 nm; $t_r$ (minor) = 12.89 min, $t_r$ (major) = 19.00 min), 97% ee, $[\alpha]_D^{20}$ = -50.9 (c = 0.990, CHCl3); syn/anti = 97/3; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.40 (d, *J* = 1.8 Hz, 1H), 7.24 (dd, *J* = 2.1, 8.7 Hz, 2H), 4.95-4.80 (m, 2H), 4.27 (dt, *J* = 4.8, 9.6 Hz, 1H), 2.90-2.81 (m, 1H), 2.48-2.32 (m, 2H), 2.14-2.05 (m, 1H), 1.84-1.52 (m, 4H), 1.40-1.26 (m, 1H). $^{13}$C NMR (75 MHz, $CDCl_3$): 211.2, 135.1, 134.1, 133.8, 130.0, 127.6, 76.9, 51.5, 42.6, 40.3, 32.8, 28.3, 25.1; IR (neat): 2942, 2863, 1708, 1552, 1475, 1379, 820, 733 cm$^{-1}$; MS (ESI, m/z): 316 ($M+H^+$) ; HRMS (ESI): Calcd for $C_{14}H_{15}NO_3Cl_2Na^+$: 338.0330. Found: 338.0321.



**(S)-2-((R)-1-(benzo[d][1,3]dioxol-5-yl)-2-nitroethyl)cyclohexanone 5h**[8] : **(Procedure B)** reaction time: 38 h (87% yield); ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 5/95, 0.7 mL/min, 214 nm; $t_r$ (minor) = 48.00 min, $t_r$ (major) = 52.13 min), 98% ee, $[\alpha]_D^{20}$ = -25.6 (c = 0.776, $CHCl_3$); syn/anti = 94/6; $^1$H NMR (300 MHz, $CDCl_3$): δ 6.74 (d, *J* = 7.8 Hz, 1H), 6.64 (s, 1H), 6.62 (d, *J* = 7.8 Hz, 1H), 5.96 (s, 2H), 4.90 (dd, *J* = 4.5, 12.3 Hz, 1H), 4.55 (dd, *J* = 10.2, 12.0 Hz, 1H), 3.68 (dt, *J* = 4.2, 9.9 Hz, 1H), 2.65-2.56 (m, 1H), 2.52-2.33 (m, 2H), 2.15-2.05 (m, 1H), 1.83-1.59 (m, 4H), 1.31-1.18 (m, 1H).

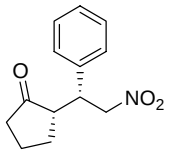


**(S)-2-((R)-1-(4-methoxyphenyl)-2-nitroethyl)cyclohexanone 5i**[9]: **(Procedure B)** reaction time: 44 h (95% yield); ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 25/75, 0.7 mL/min, 238 nm; $t_r$ (minor) = 13.46 min, $t_r$ (major) = 16.13 min), 88% ee, $[\alpha]_D^{20}$ = -20.0 (c = 0.420, $CHCl_3$); syn/anti = 97/3; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.08 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 4.91 (dd, *J* = 4.5, 12.3 Hz, 1H), 4.58 (dd, *J* = 9.9, 12.0 Hz, 1H), 3.78 (s, 3H), 3.71 (dt, *J* = 4.5, 9.9 Hz, 1H), 2.69-2.60 (m, 1H), 2.52-2.32 (m, 2H), 2.13-2.03 (m, 1H), 1.83-1.51 (m, 4H), 1.30-1.16 (m, 1H).

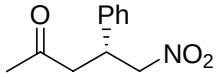


**(S)-2-((S)-3-methyl-1-nitrobutan-2-yl)cyclohexanone 5j**[10] : **(Procedure B)** reaction time: 6 d (63% yield); ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 10/90, 1.0 mL/min, 214 nm; $t_r$ (major) = 6.31 min, $t_r$ (minor) = 7.76 min), 94% ee, $[\alpha]_D^{20}$ = -28.5 (c = 1.305, $CHCl_3$); syn/anti = 99/1; $^1$H NMR (300 MHz, $CDCl_3$): δ 4.64 (dd, *J* = 5.7, 13.8 Hz, 1H), 4.35 (dd, *J* = 5.1, 13.8 Hz, 1H), 2.67-2.60 (m, 1H), 2.43-2.28 (m, 3H), 2.10 (m, 2H), 1.99-1.88 (m,

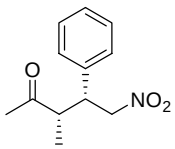
2H), 1.74-1.54 (m, 3H), 0.94 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 3H).



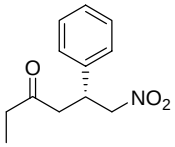
**(S)-2-((R)-2-nitro-1-phenylethyl)cyclopentanone 5l**[11] : **(Procedure B)** reaction time: 6 d (27% yield); ee was determined by HPLC analysis (Chiralcel AD, *i*-PrOH/hexane = 5/95, 1.0 mL/min, 220 nm; $t_r$ (minor) = 14.42 min, $t_r$ (major) = 20.05 min), 71% ee, $[\alpha]_D^{20}$ = -15.5 (c = 0.495, $CHCl_3$); syn/anti = 75/15; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.34-7.23 (m, 3H), 7.20-7.15 (m, 2H), 5.37-5.30 (m, 1H), 4.71 (dd, *J* = 10.2, 12.6 Hz, 1H), 3.76-3.65 (m, 1H), 2.44-2.31 (m, 2H), 2.19-2.06 (m, 1H) , 1.94-1.83 (m, 2H), 1.76-1.66 (m, 1H), 1.55-1.41 (m, 1H).



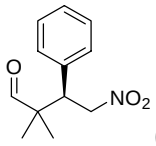
**(R)-5-nitro-4-phenylpentan-2-one 5k**[11]: **(Procedure B)** reaction time: 48 h (80% yield); ee was determined by HPLC analysis (Chiralcel AS, *i*-PrOH/hexane = 15/85, 1.0 mL/min, 220 nm; $t_r$ (minor) = 18.85 min, $t_r$ (major) = 22.13 min, 48% ee; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.28-7.13 (m, 5H), 4.67-4.49 (m, 2H), 3.98-3.89 (m, 1H), 2.84 (d, *J* = 6.9 Hz, 2H), 2.05 (s, 3H).



**(3S,4R)-3-methyl-5-nitro-4-phenylpentan-2-one 5ma**[11] : **(Procedure B)** reaction time: 48 h (90% total yield for compound **5ma** and **5mb**); ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 1.2/100, 0.6 mL/min, 238 nm; $t_r$ (minor) = 43.45 min, $t_r$ (major) = 54.10 min), 70% ee, $[\alpha]_D^{20}$ = -10.6 (c = 1.250, $CHCl_3$); syn/anti = 99/1; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.37-7.30 (m, 3H), 7.18-7.15 (m, 2H), 4.67-4.63 (m, 2H), 3.80 (dt, *J* = 5.7, 9.0 Hz, 1H), 3.03-2.93 (m, 1H), 2.24 (s, 3H), 0.98 (d, *J* = 7.2 Hz, 3H).

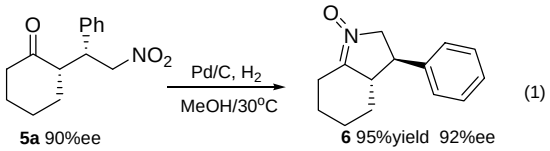


**(R)-6-nitro-5-phenylhexan-3-one 5mb**[11] : **(Procedure B)** reaction time: 48 h (90% total yield for compound **5ma** and **5mb**); ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 1.2/100, 0.6 mL/min, 238 nm; $t_r$ (minor) = 60.36 min, $t_r$ (major) = 65.47 min), 70% ee; $[\alpha]_D^{20}$ = -6.6 (c = 0.735, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$): δ 7.33-7.26 (m, 3H), 7.23-7.20 (m, 2H), 4.74-4.54 (m, 2H), 4.08-3.98 (m, 1H), 2.88 (d, *J* = 7.2 Hz, 2H), 2.43-2.33 (m, 2H), 1.00 (t, *J* = 7.2 Hz, 3H).

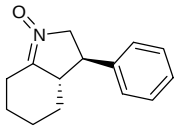


**(R)-2,2-dimethyl-4-nitro-3-phenylbutanal 5n**[12] : **(Procedure A)** reaction time: 48h (62% yield); ee was determined by HPLC analysis (Chiralcel AS-H, *i*-PrOH/hexane = 10/90, 0.7 mL/min, 238 nm; $t_r$ (minor) = 20.38 min, $t_r$ (major) = 21.96 min), 82% ee, $[\alpha]_D^{20}$ = +0.3 (c = 0.625, $CHCl_3$); $^1$H NMR (300 MHz, $CDCl_3$): δ 9.53 (s, 1H), 7.36-7.30 (m, 3H), 7.22-7.18 (m, 2H), 4.86 (dd, *J* = 11.4, 12.9 Hz, 1H), 4.69 (dd, *J* = 3.9, 12.6 Hz, 1H), 3.79 (dd, *J* = 4.2, 11.1 Hz,

1H), 1.14 (s, 3H), 1.01 (s, 3H).




(1)




**(3R,3aS)-3-phenyl-3,3a,4,5,6,7-hexahydro-2H-indole 1-oxide 7**[13] **:** A suspension of Pd/C (8 mg) and **5a** (50 mg) in MeOH (5 mL) was stirred at room temperature under 3 atm hydrogen atmosphere. After being stirred for 12 h, the mixture was filtrated through a pad of Celite and the filtration was concentrated in vacuo, the residue was purified by column chromatography on silica gel to afford desired product **7** (43 mg, 95% yield): ee was determined by HPLC analysis (Chiralcel AD-H, *i*-PrOH/hexane = 10/90, 0.6 mL/min, 238 nm; $t_r$ (major) = 18.02 min, $t_r$ (minor) = 22.83 min), 92% ee. Mp. 86 ºC; $[\alpha]_D^{20}$ = -34.1 (c = 1.140, $CHCl_3$); syn/anti >99/1; $^1$H NMR (300 MHz, $CDCl_3$): δ 7.30-7.25 (m, 2H), 7.21-7.15 (m, 3H), 4.23-4.04 (m, 2H), 3.19-3.01 (m, 2H), 2.73 (m, 1H), 2.05-1.87 (m, 3H), 1.77 (m, 1H), 1.33-1.14 (m, 3H). $^{13}$C NMR (75 MHz, $CDCl_3$): 147.9, 139.4, 128.6, 127.0, 126.9, 67.9, 50.2, 45.5, 32.0, 23.9, 23.4, 23.1; IR (neat): 2934, 2857, 1621, 1447, 1253, 1231, 1179, 765, 702 $cm^{-1}$; MS (ESI, *m/z*): 216 ($M+H^+$); HRMS (ESI): Calcd for $C_{14}H_{17}NONa^+$: 238.1204. Found: 238.1202.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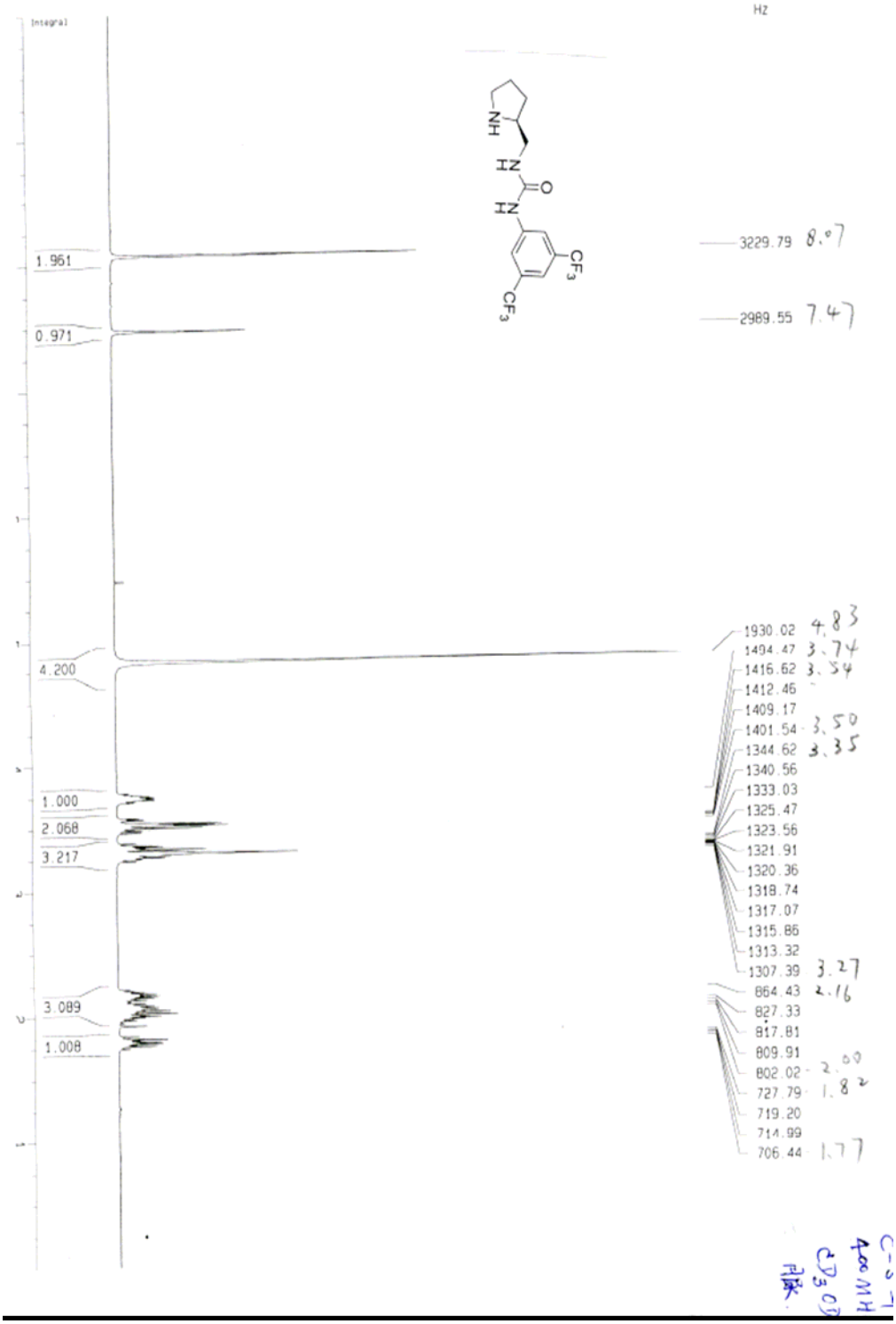
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1416.62 3.54
1412.46
1409.17
1401.54 3.50
1344.62 3.35
1340.56
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1325.47
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1317.07
1315.86
1313.32
1307.39 3.27
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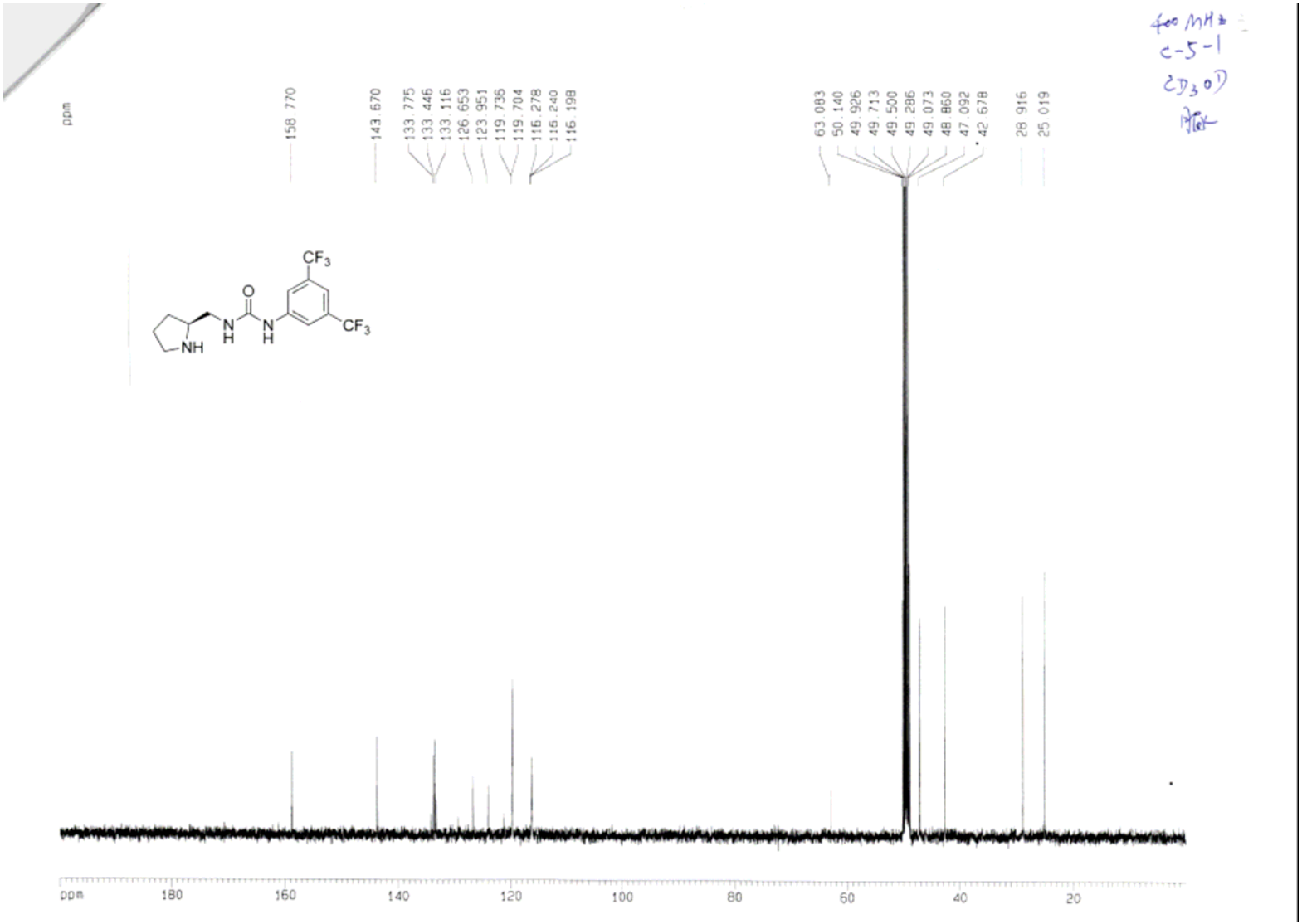
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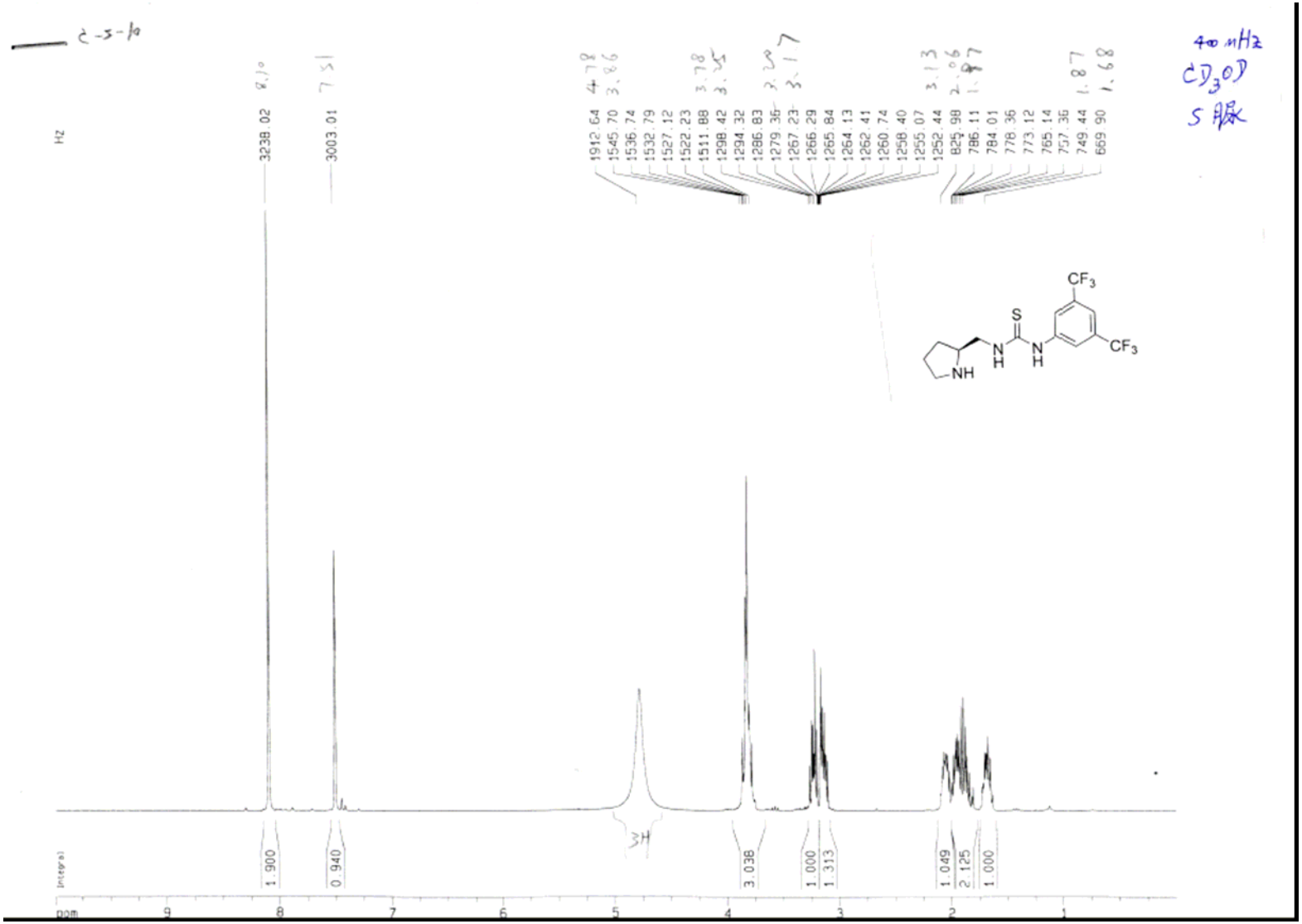
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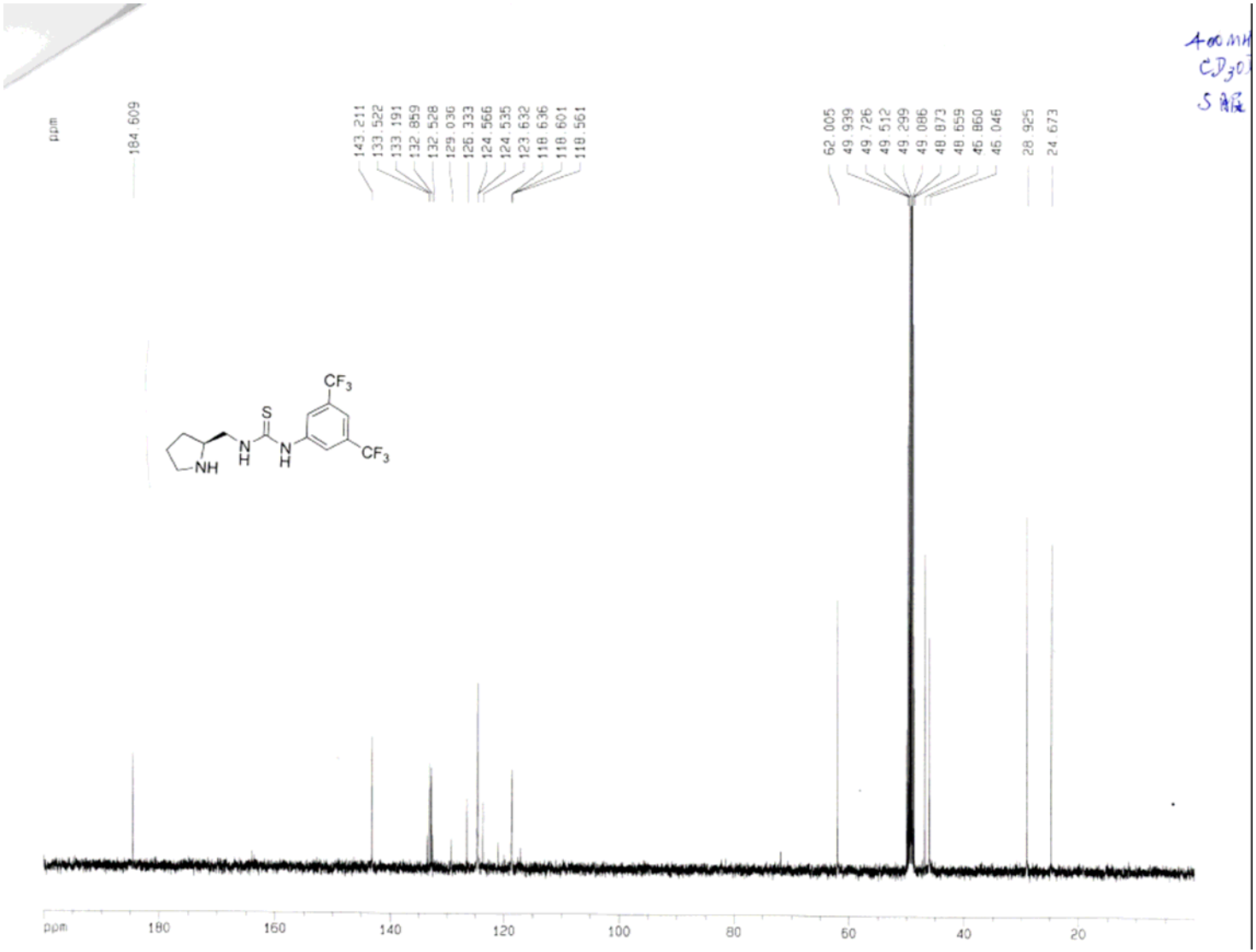
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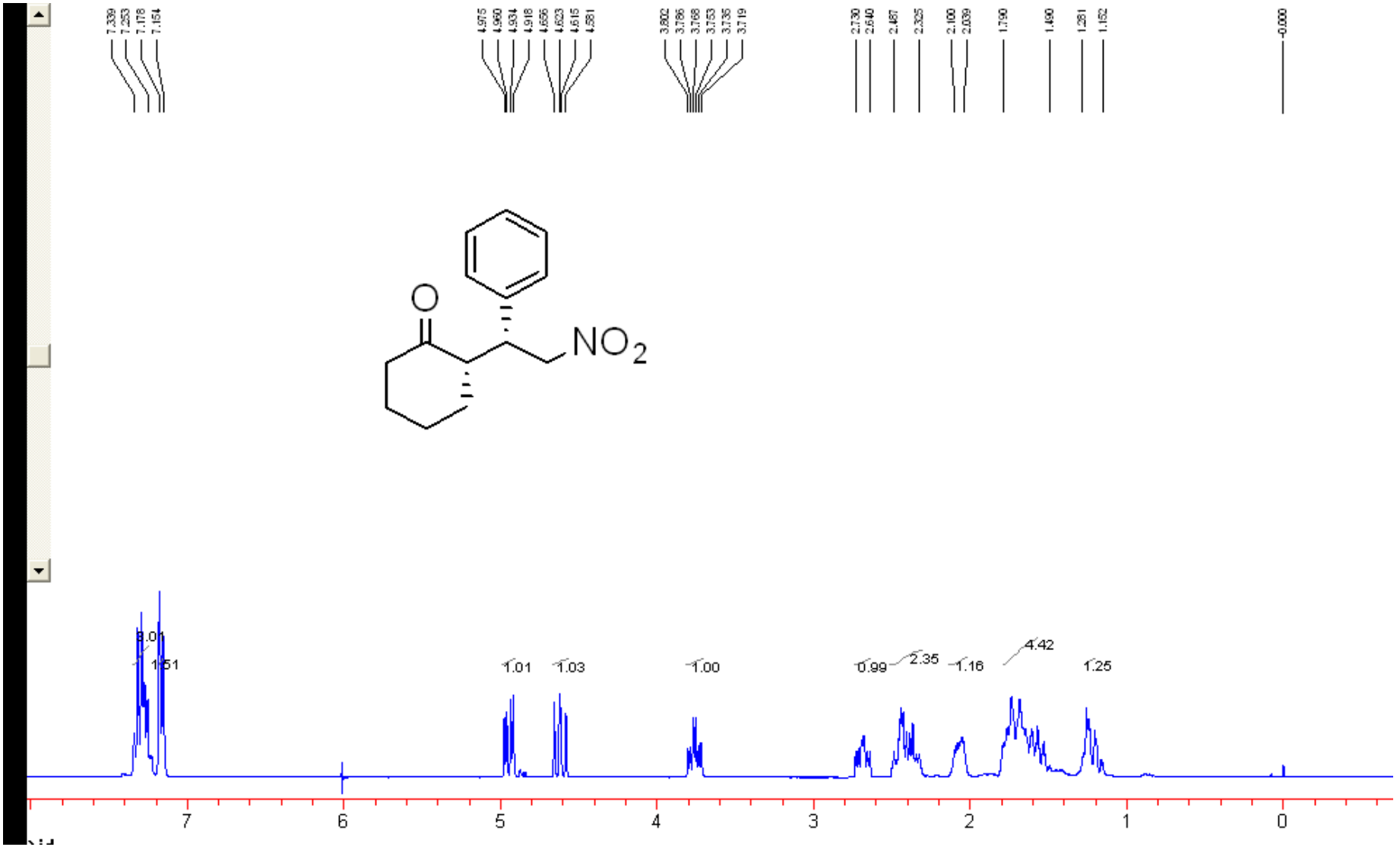
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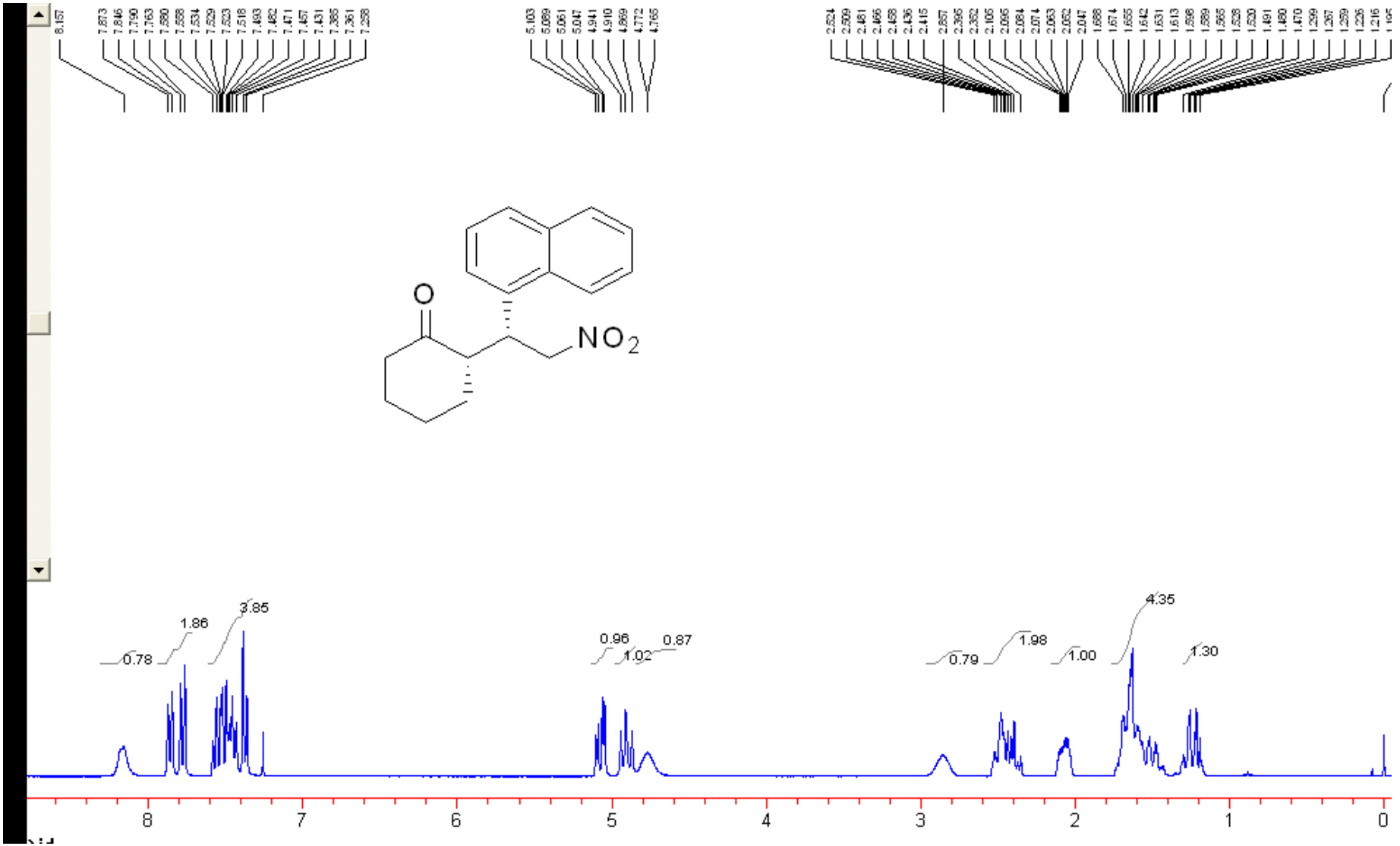
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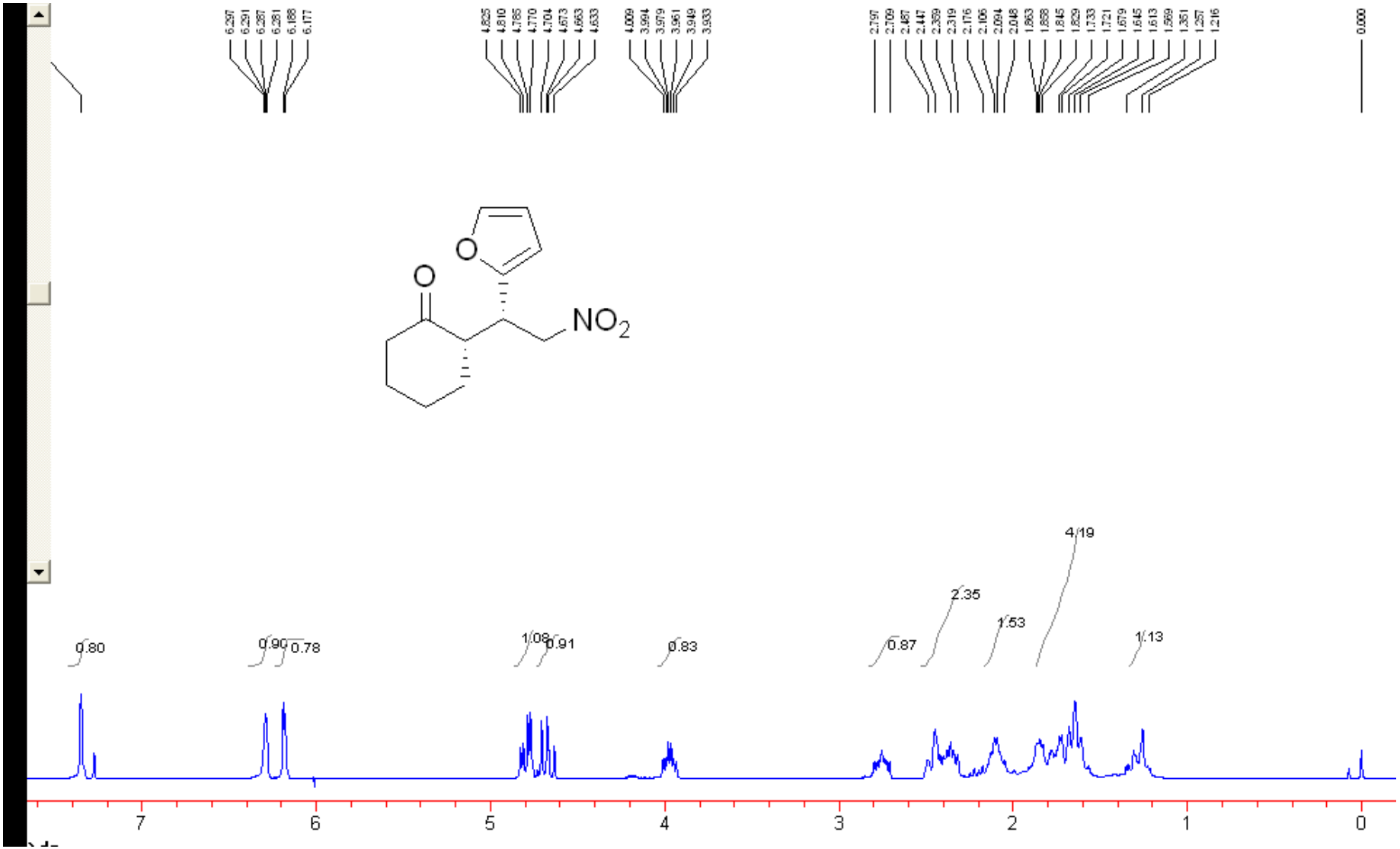
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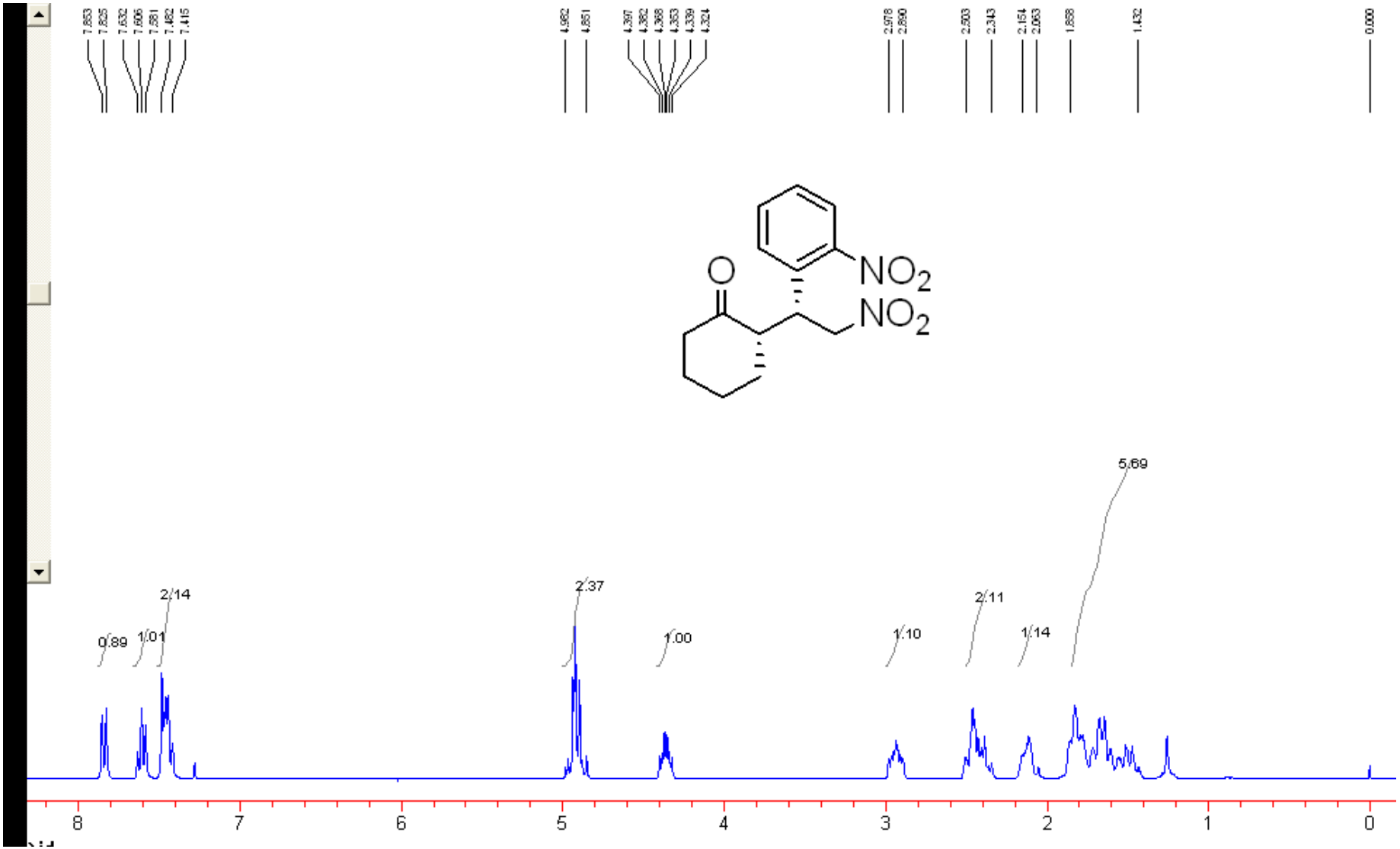
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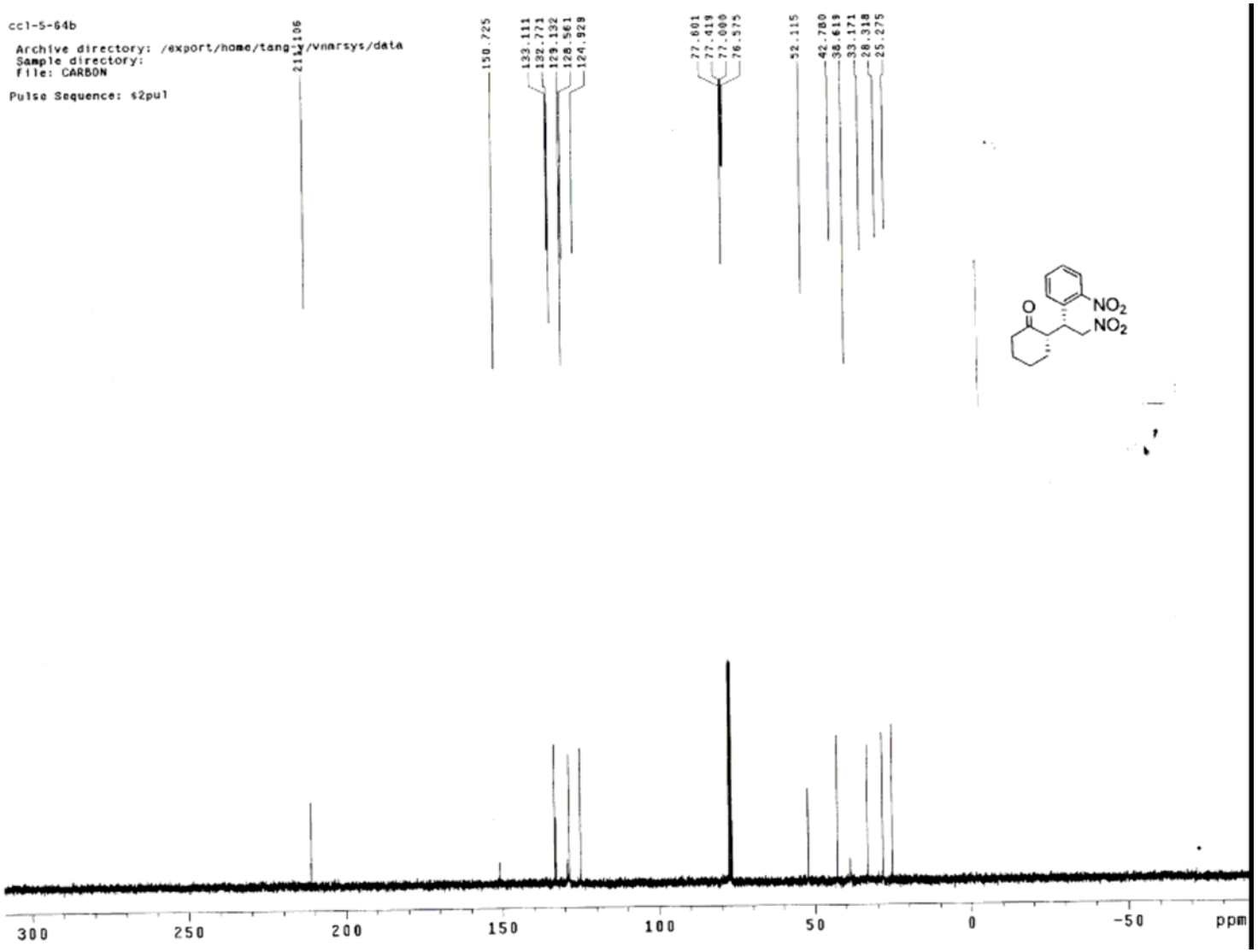
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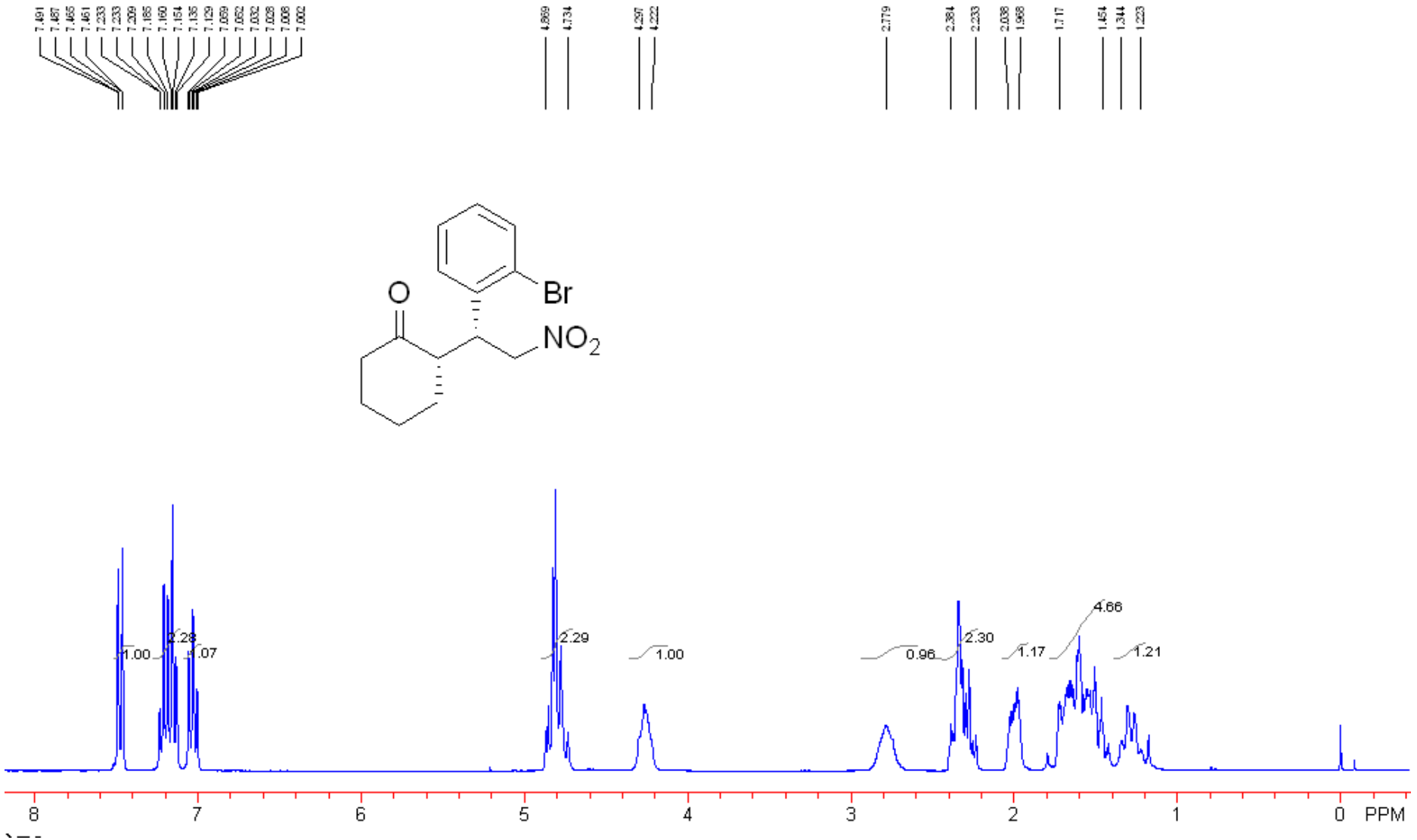
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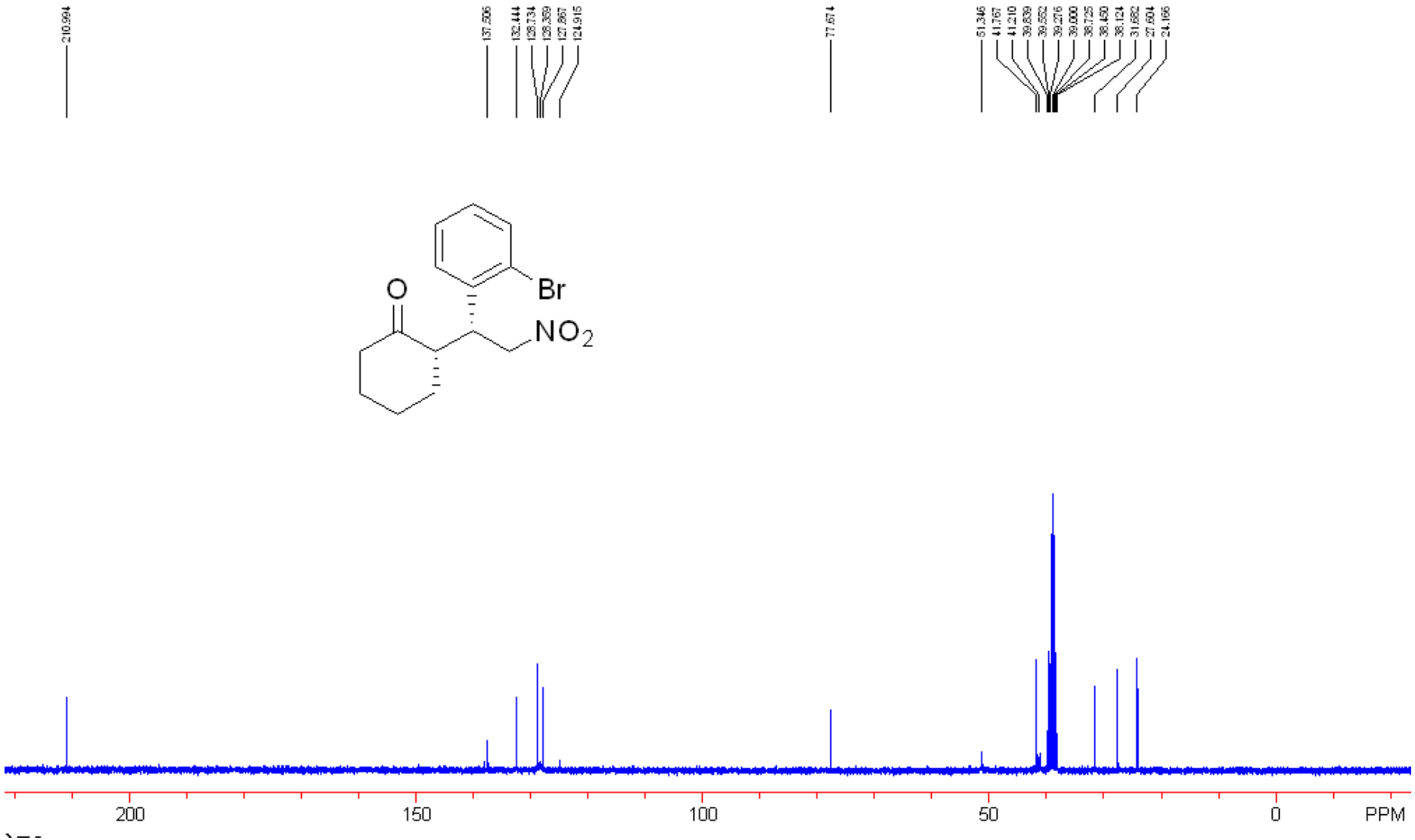
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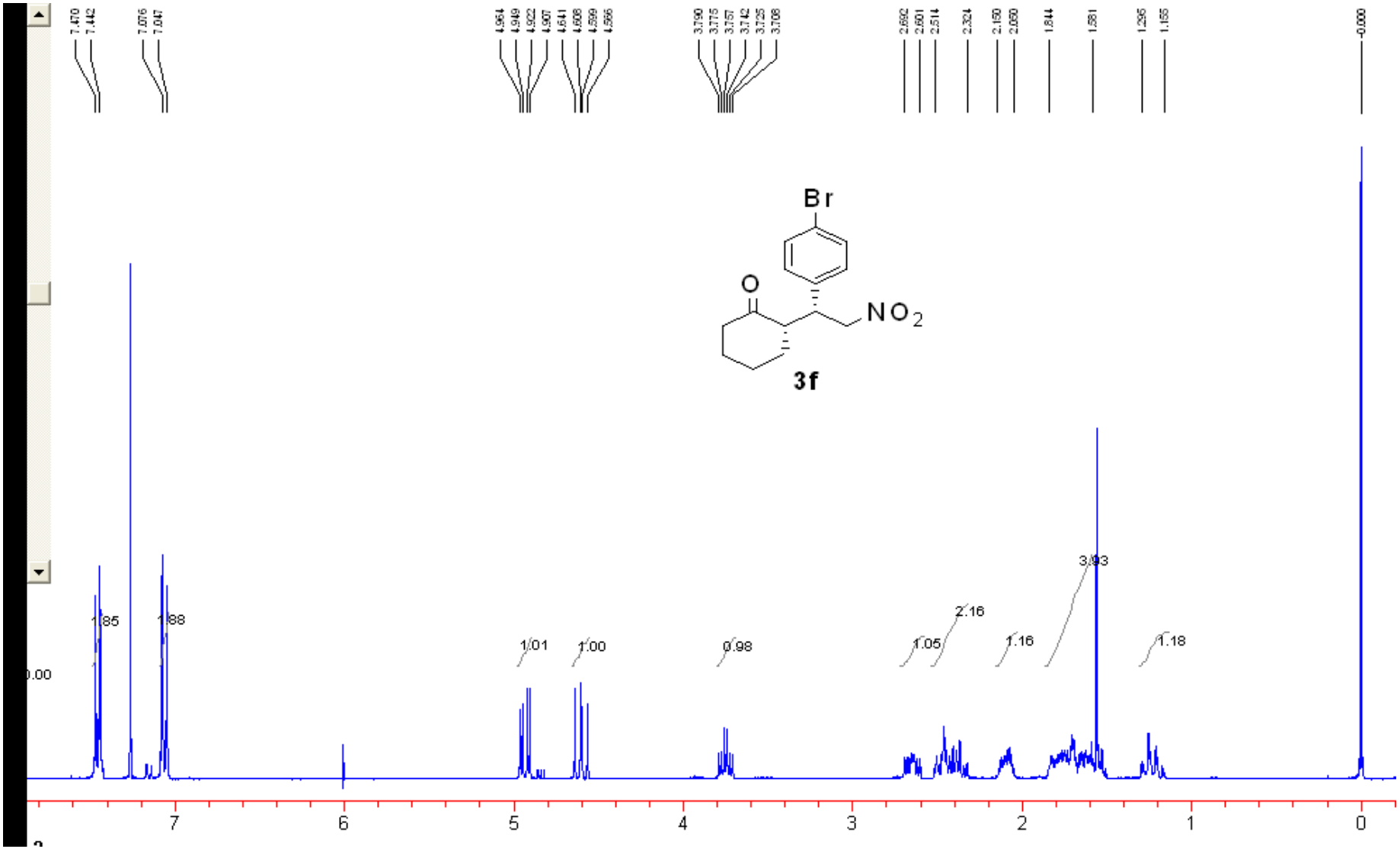
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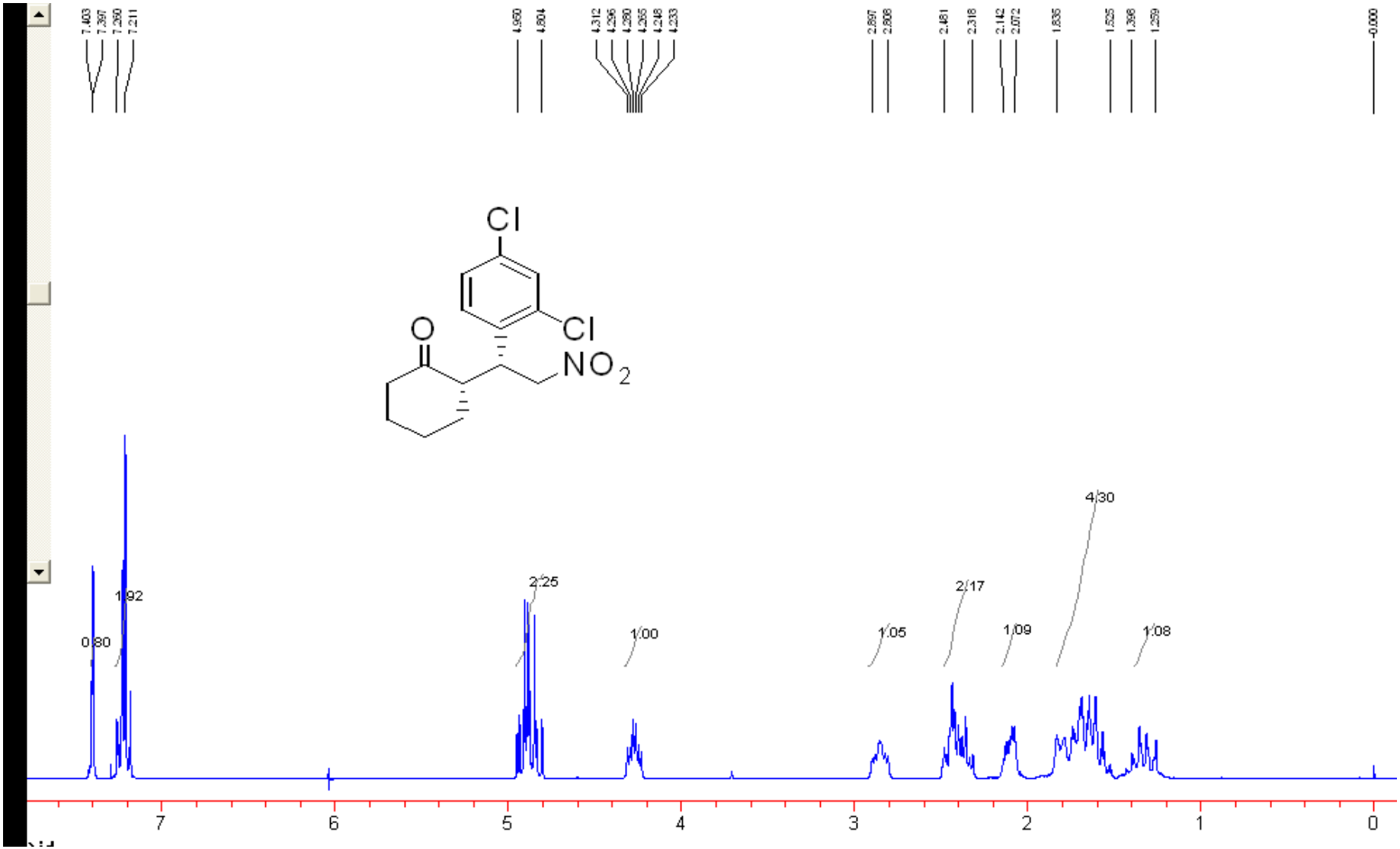
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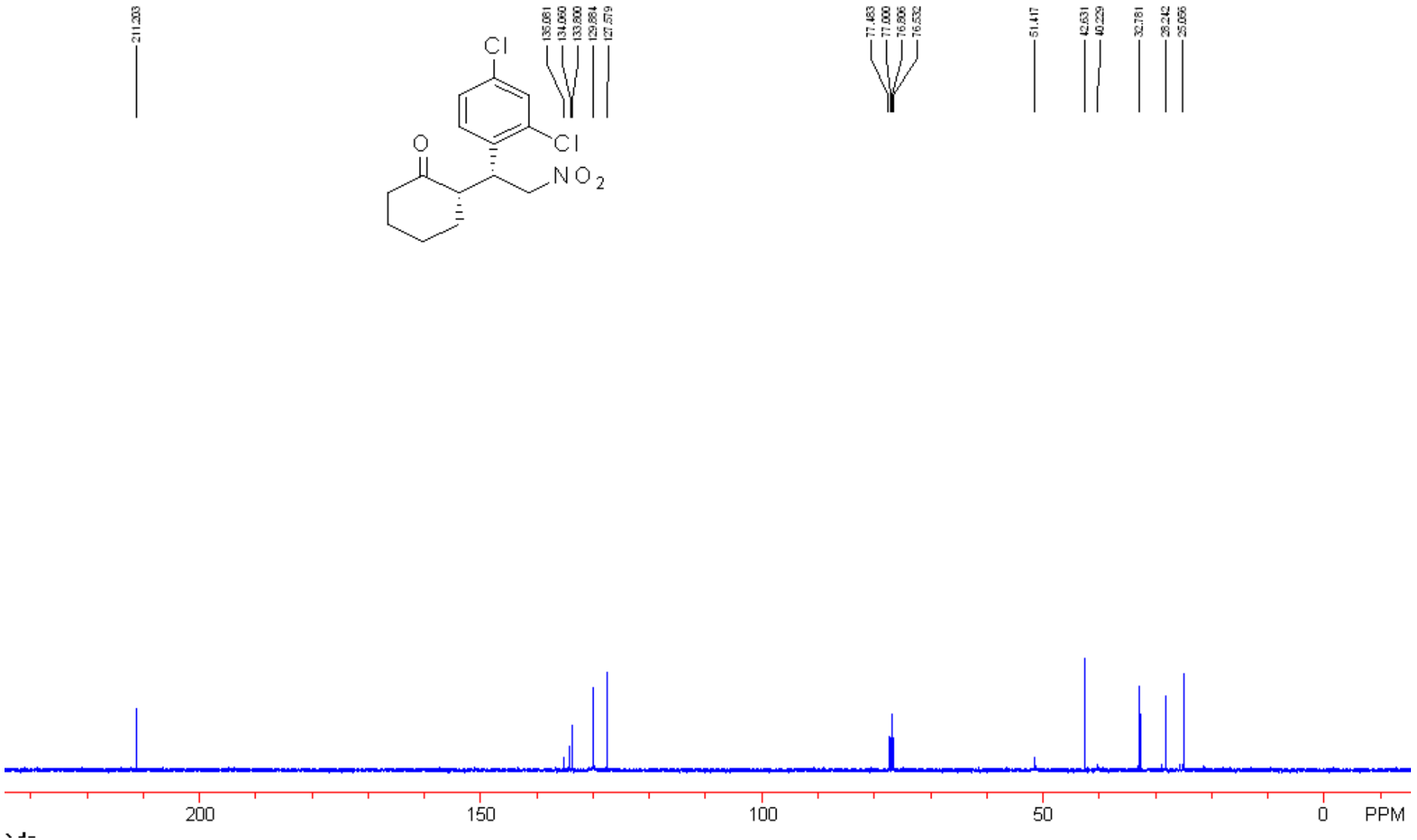
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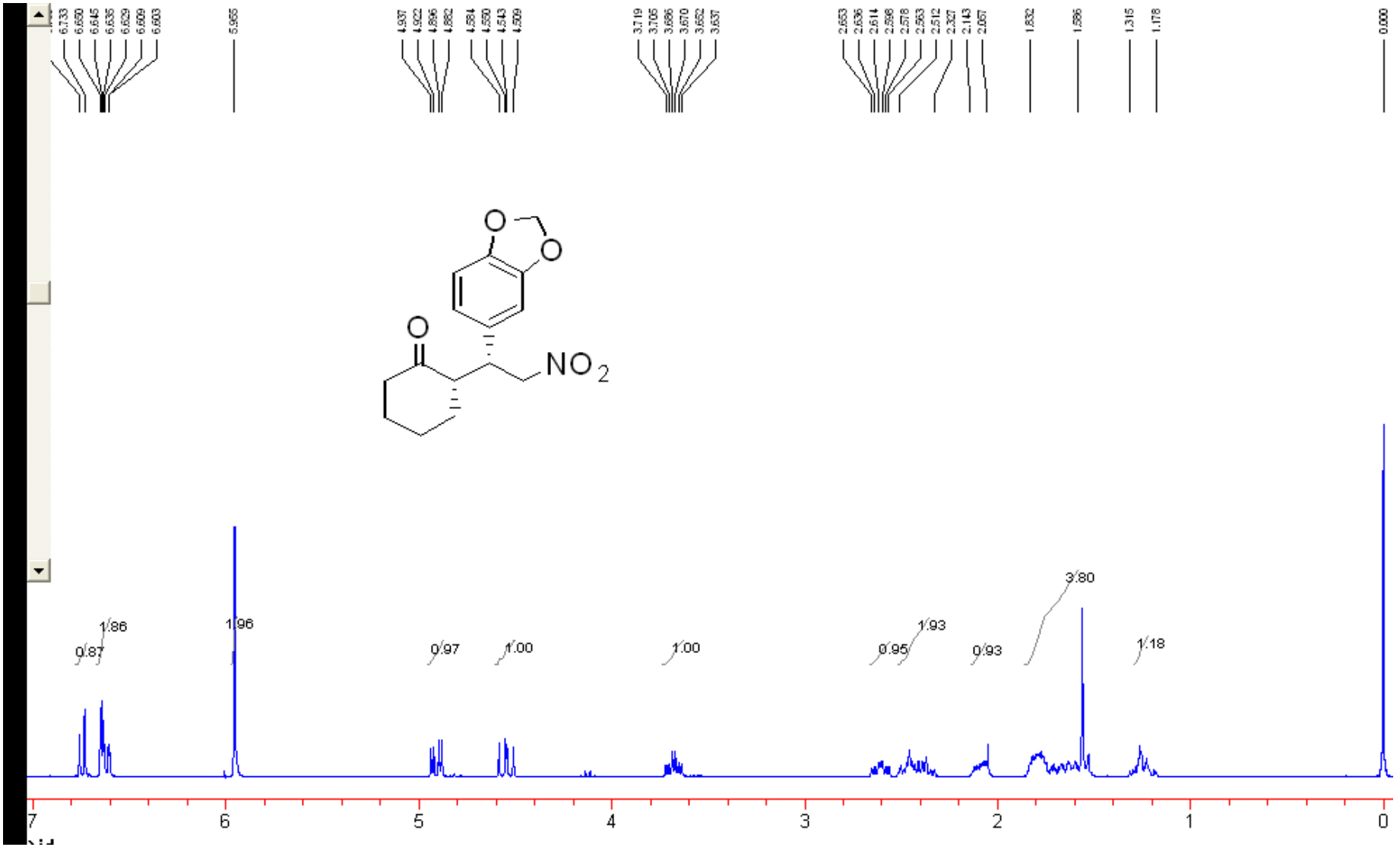
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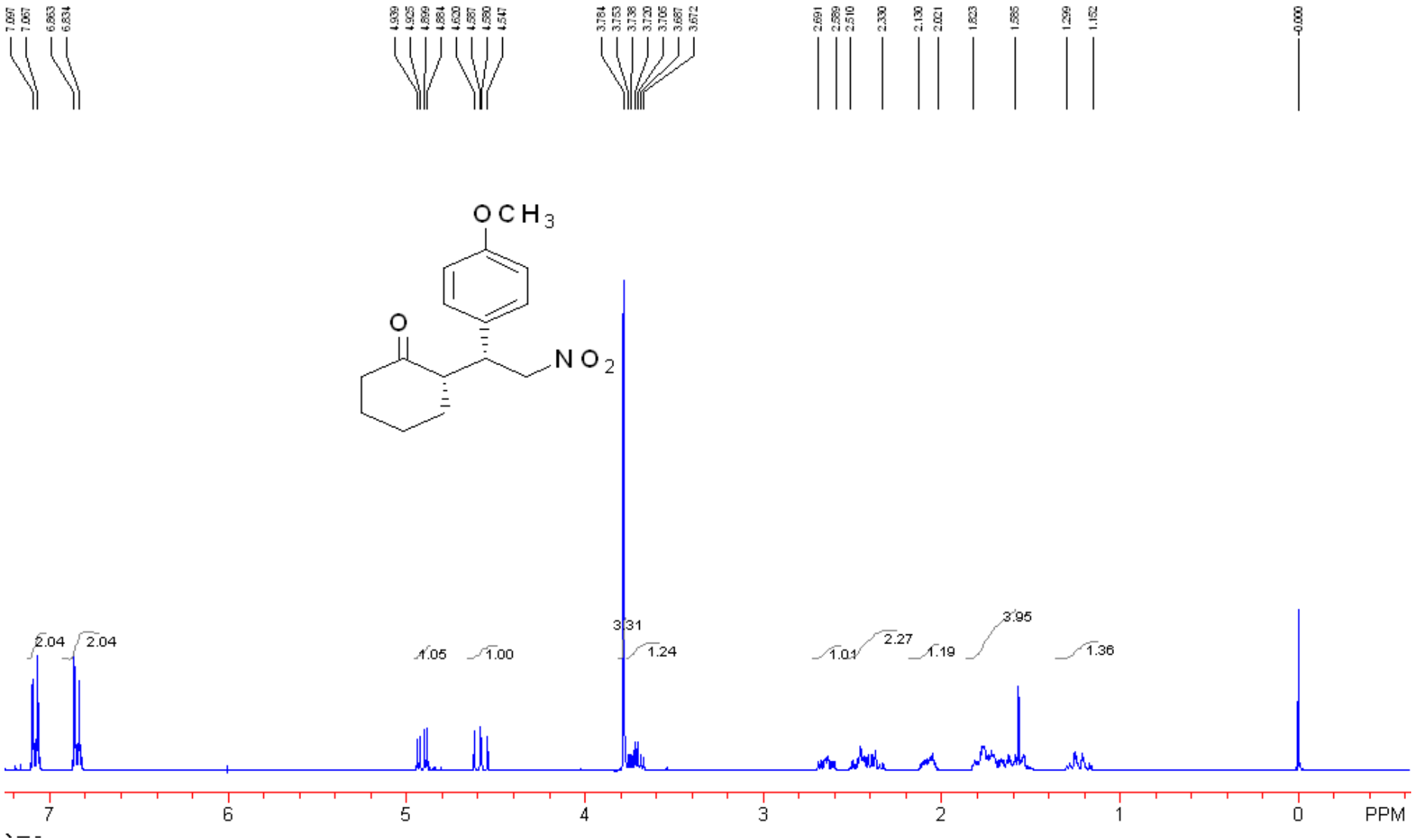
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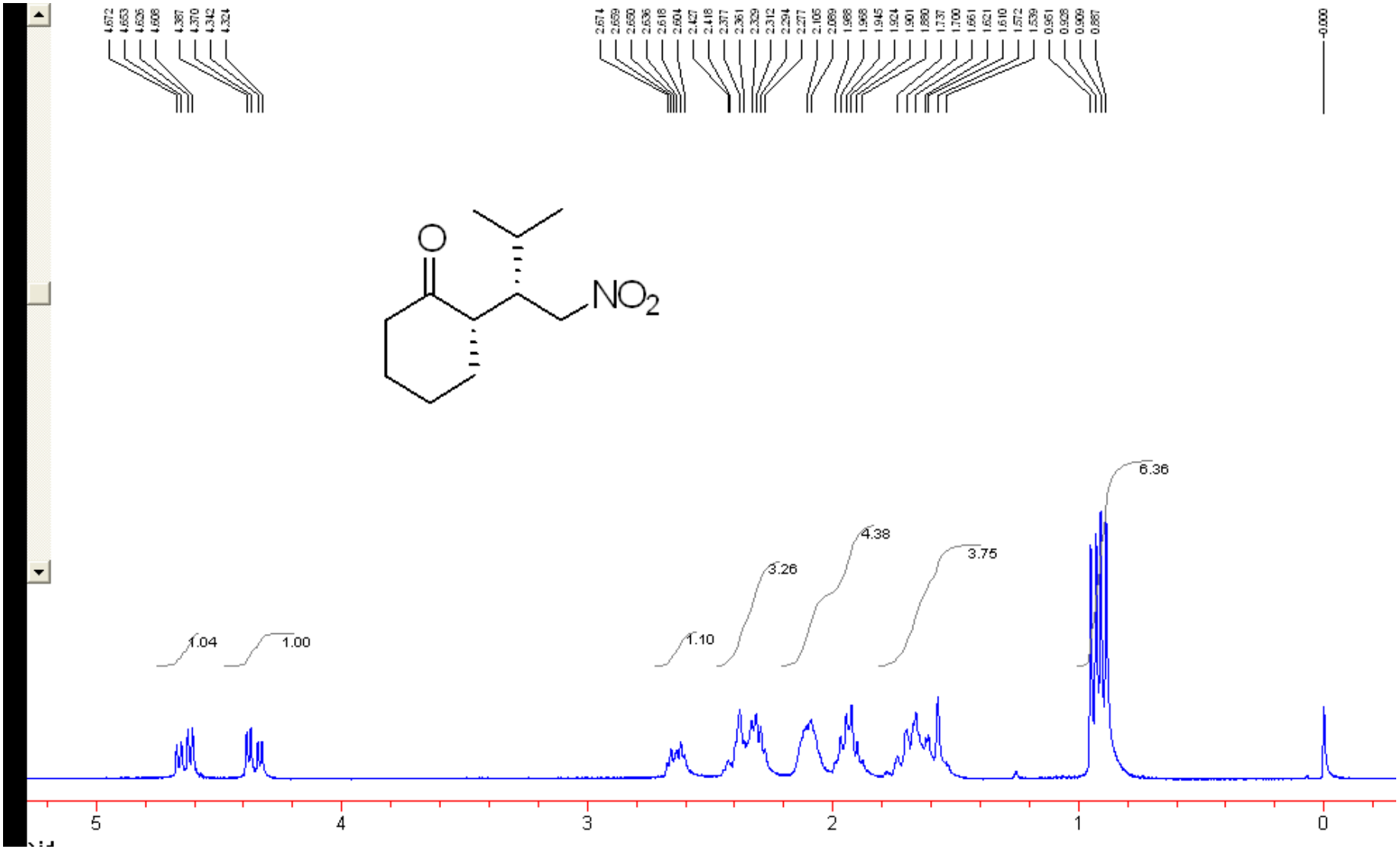
7
6
5
4
3
2
1
0
PPM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4.626
4.608
4.387
4.370
4.342
4.324
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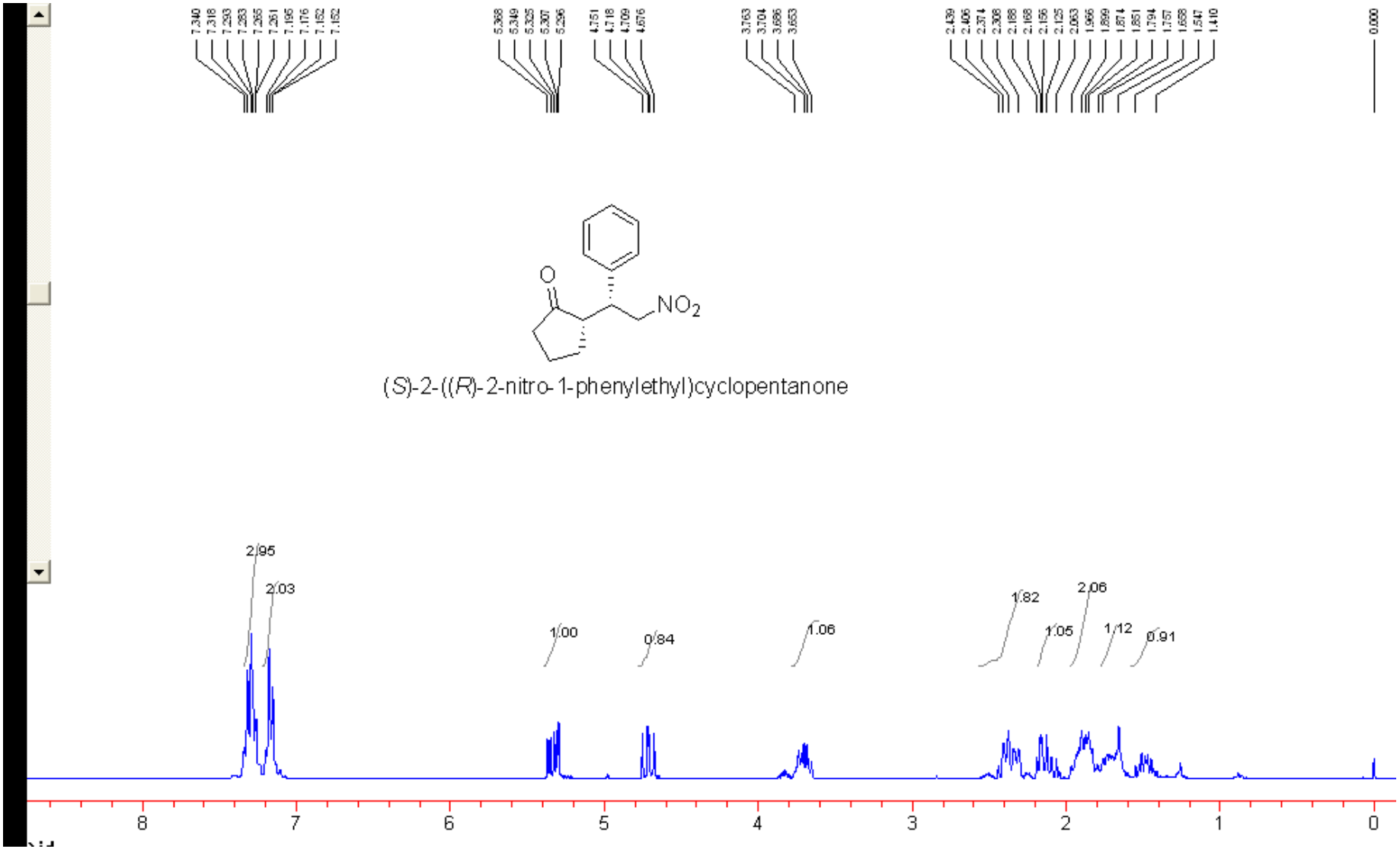
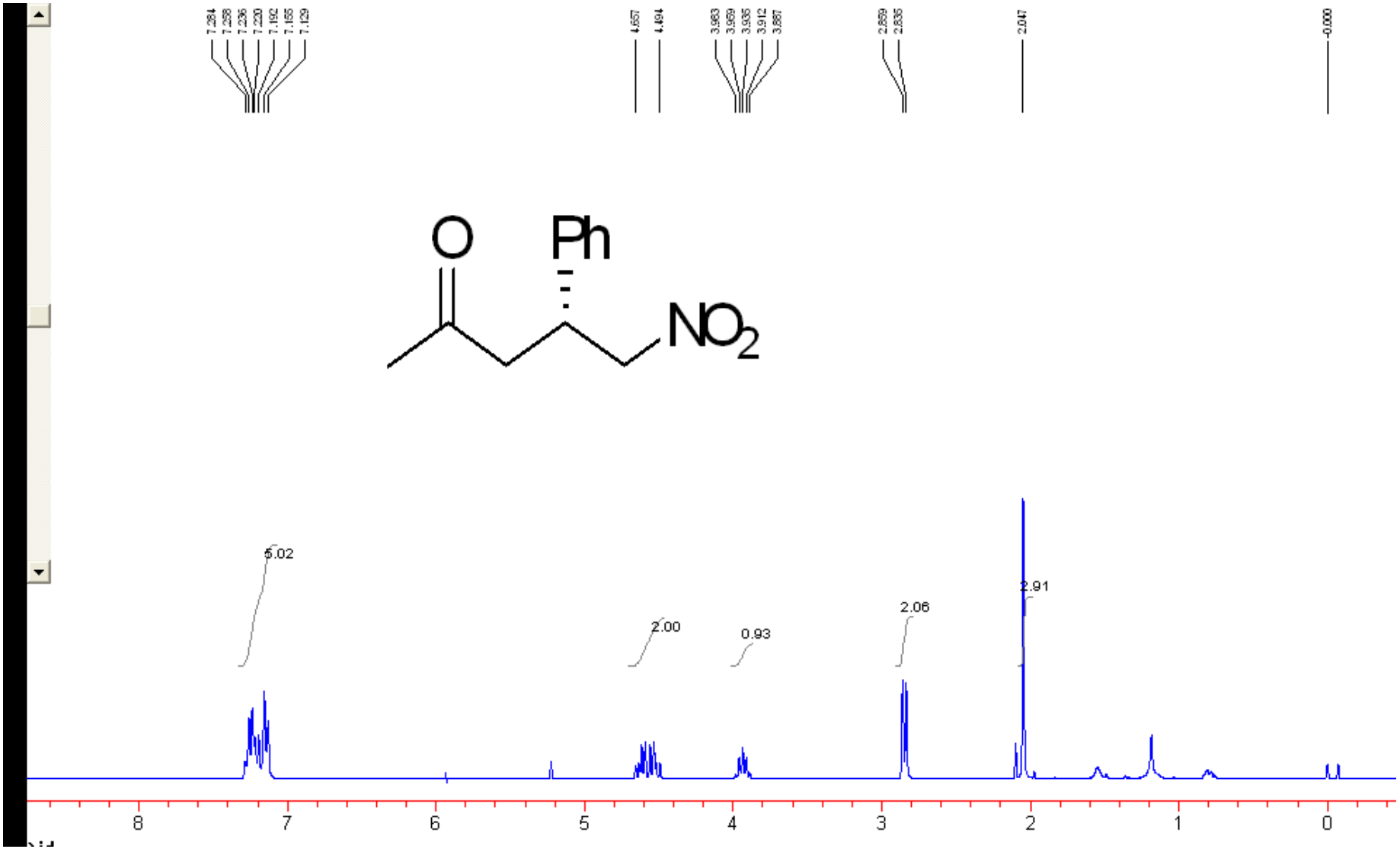
2.659
2.650
2.636
2.618
2.604
2.427
2.418
2.377
2.361
2.329
2.312
2.294
2.277
2.105
2.089
1.988
1.968
1.945
1.924
1.901
1.880
1.737
1.700
1.661
1.621
1.610
1.572
1.539
0.951
0.928
0.909
0.887
-0.000
O
NO2
1.04
1.00
1.10
3.26
4.38
3.75
6.36
5
4
3
2
1
0




(*S*)-2-((*R*)-2-nitro-1-phenylethyl)cyclopentanone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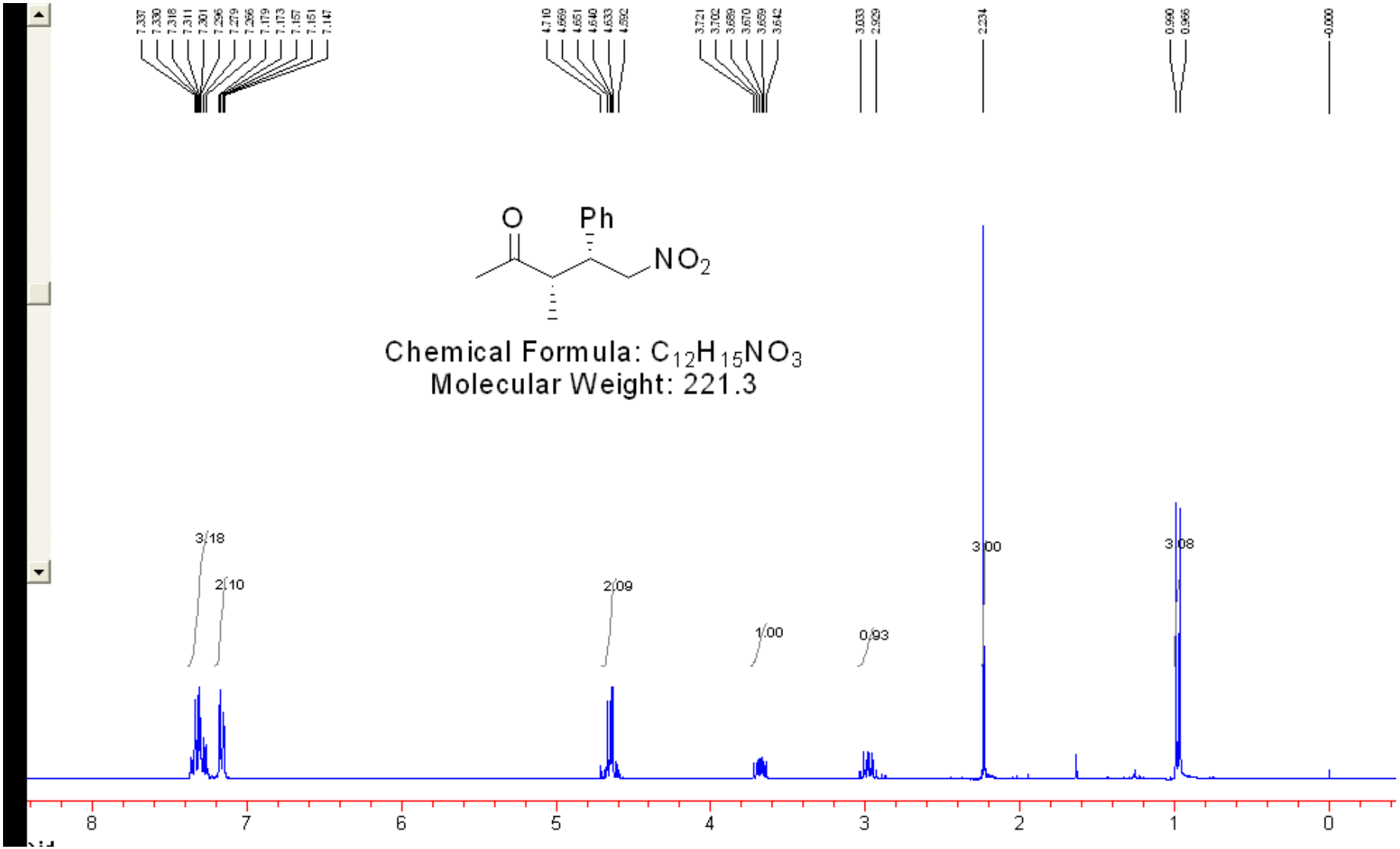
7.220
7.192
7.155
7.129
4.657
4.494
3.963
3.959
3.935
3.912
3.887
2.859
2.835
2.047
0.000
O
Ph
NO2
5.02
2.00
0.93
2.06
2.91
8
7
6
5
4
3
2
1
0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7.296
7.279
7.266
7.179
7.173
7.157
7.151
7.147
4.710
4.669
4.651
4.640
4.633
4.592
3.721
3.702
3.689
3.670
3.659
3.642
3.033
2.929
2.234
0.990
0.966
-0.000
O
Ph
NO2
Chemical Formula: C12H15NO3
Molecular Weight: 221.3
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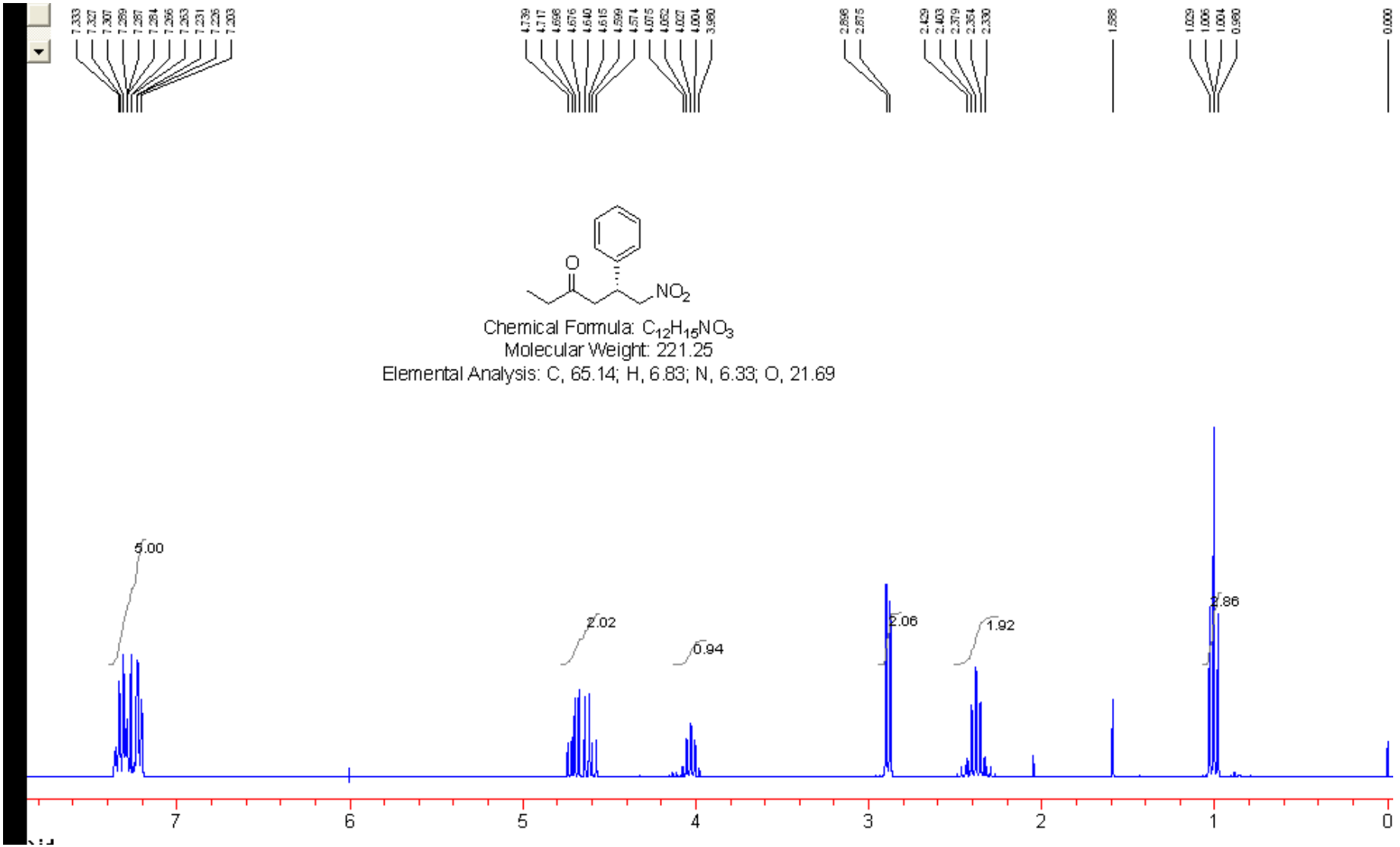
2.10
2.09
1.00
0.93
3.00
3.08
8
7
6
5
4
3
2
1
0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7.284
7.266
7.263
7.231
7.226
7.203
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4.717
4.698
4.676
4.640
4.615
4.599
4.574
4.075
4.052
4.027
4.004
3.990
2.896
2.875
2.429
2.403
2.379
2.354
2.330
1.588
1.029
1.006
1.004
0.990
0.000
O
NO2
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Molecular Weight: 221.25
Elemental Analysis: C, 65.14; H, 6.83; N, 6.33; O, 21.69
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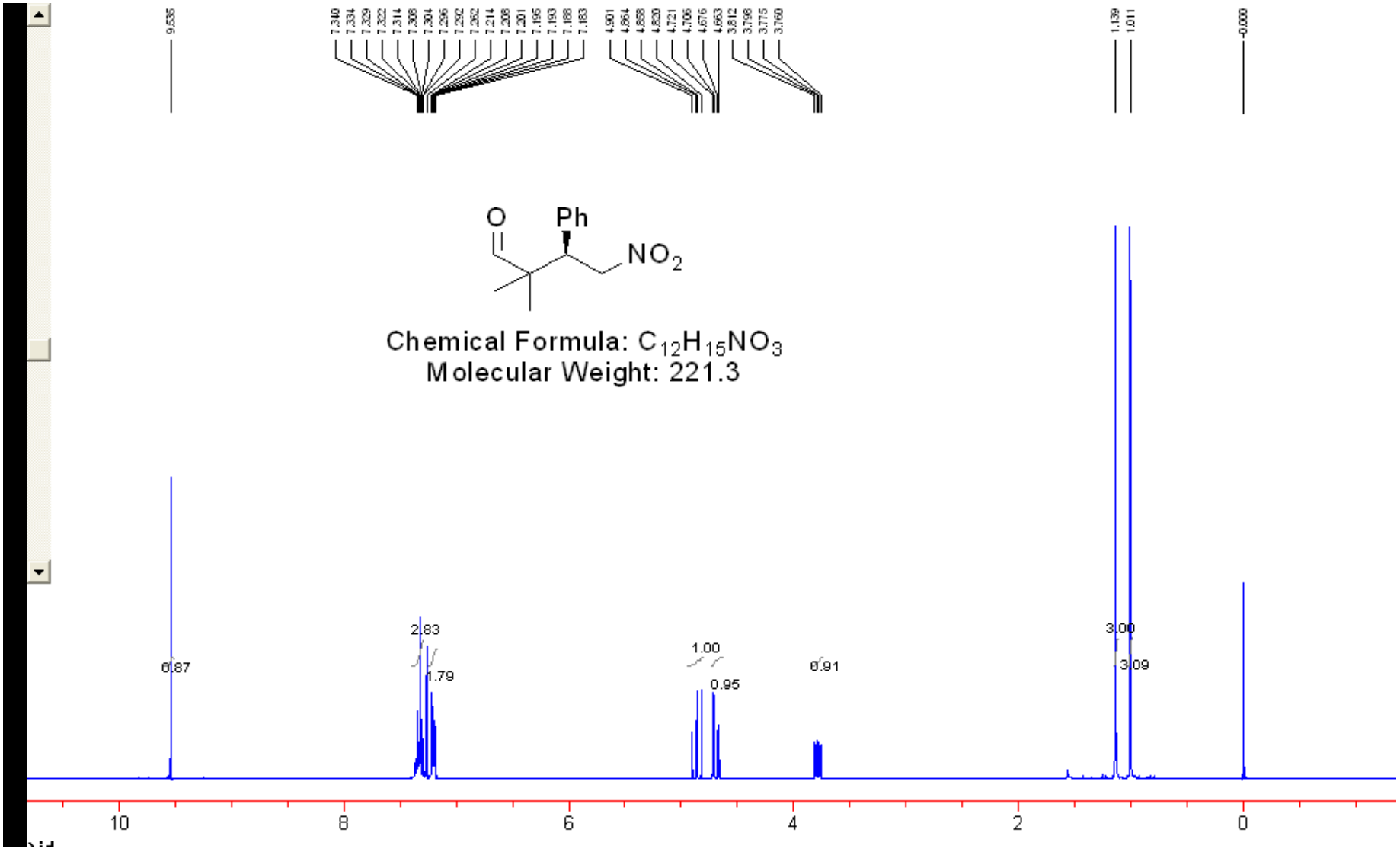
3
2
1
0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7.304
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7.292
7.282
7.214
7.208
7.201
7.195
7.193
7.188
7.183
4.901
4.864
4.858
4.820
4.721
4.706
4.676
4.663
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3.798
3.775
3.760
1.139
1.011
-0.000
O
Ph
NO2
Chemical Formula: C12H15NO3
Molecular Weight: 221.3
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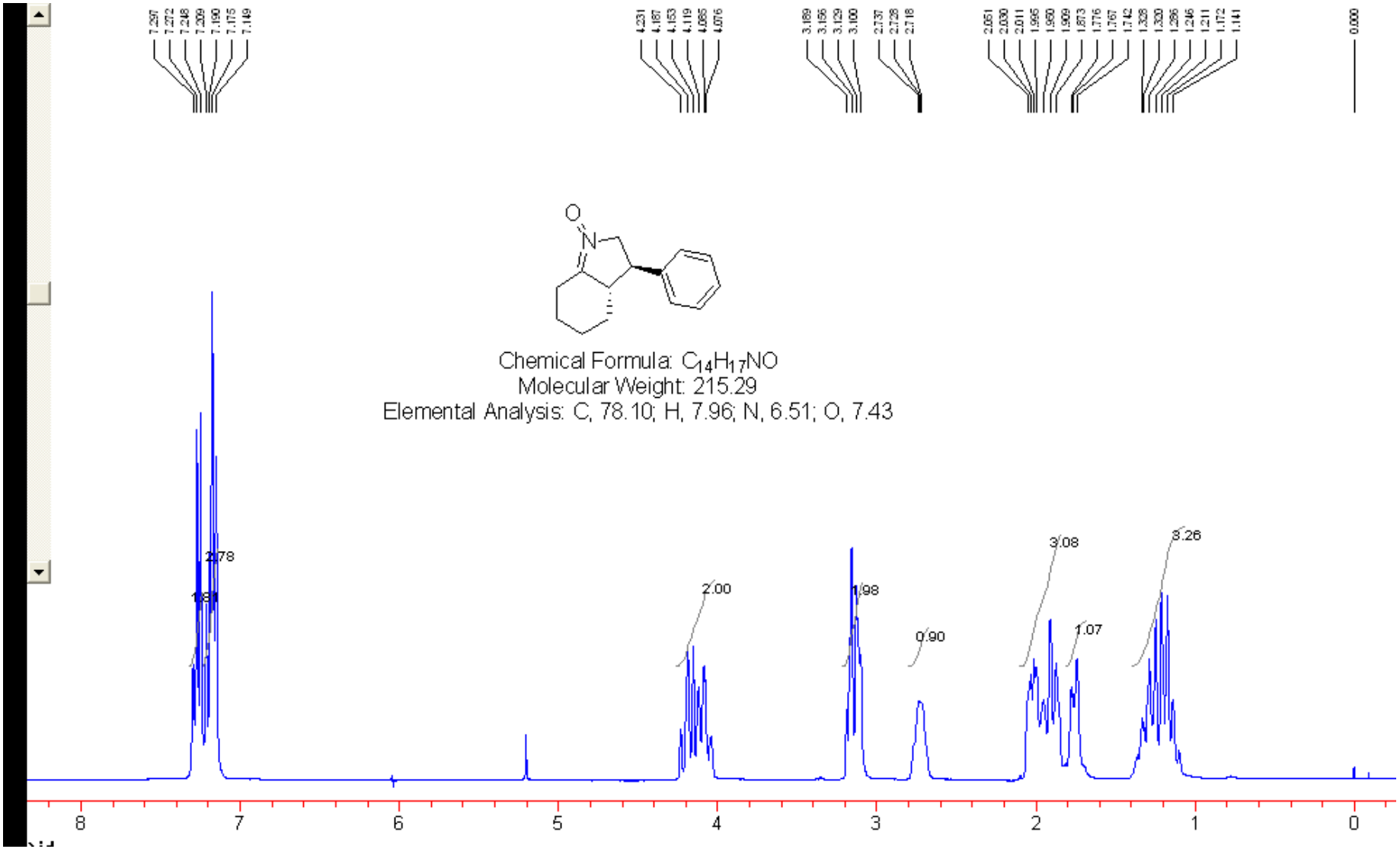
3.09
10
8
6
4
2
0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7.190
7.175
7.149
4.231
4.161
4.153
4.119
4.085
4.076
3.189
3.156
3.129
3.100
2.737
2.728
2.718
2.051
2.030
2.011
1.995
1.950
1.909
1.873
1.776
1.767
1.742
1.328
1.300
1.296
1.246
1.211
1.172
1.141
0.000
O
N
Chemical Formula: C14H17NO
Molecular Weight: 215.29
Elemental Analysis: C, 78.10; H, 7.96; N, 6.51; O, 7.43
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2.00
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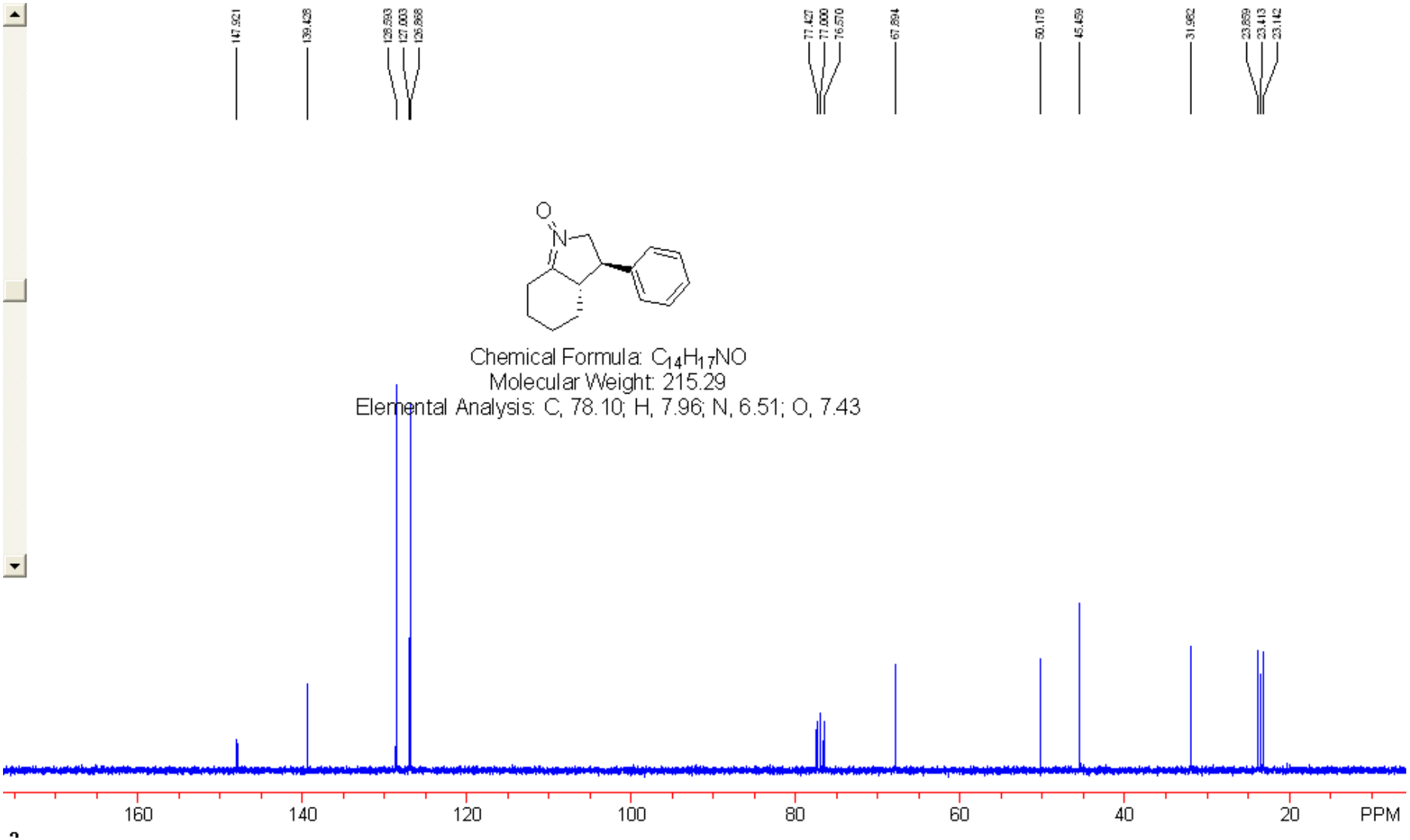
0.90
3.08
1.07
3.26
8
7
6
5
4
3
2
1
0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128.593
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77.000
76.570
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50.178
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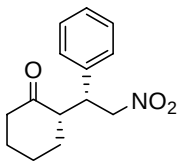
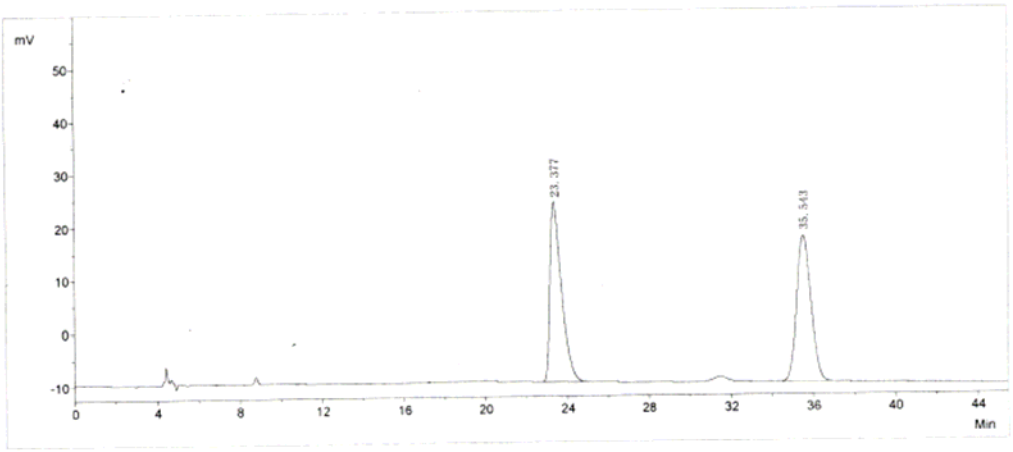
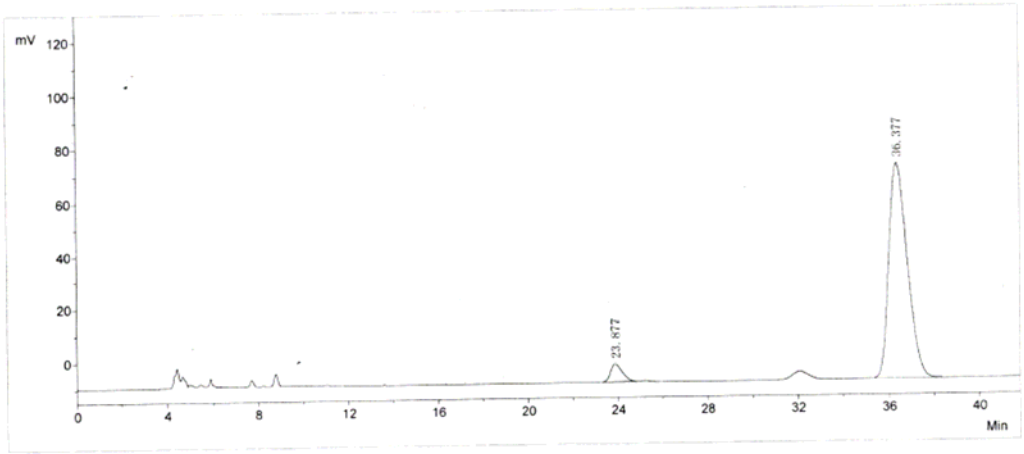
31.962
23.859
23.413
23.142
O
N
Chemical Formula: C14H17NO
Molecular Weight: 215.29
Elemental Analysis: C, 78.10; H, 7.96; N, 6.51; O, 7.43
160
140
120
100
80
60
40
20
PPM



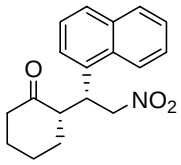

**5a**



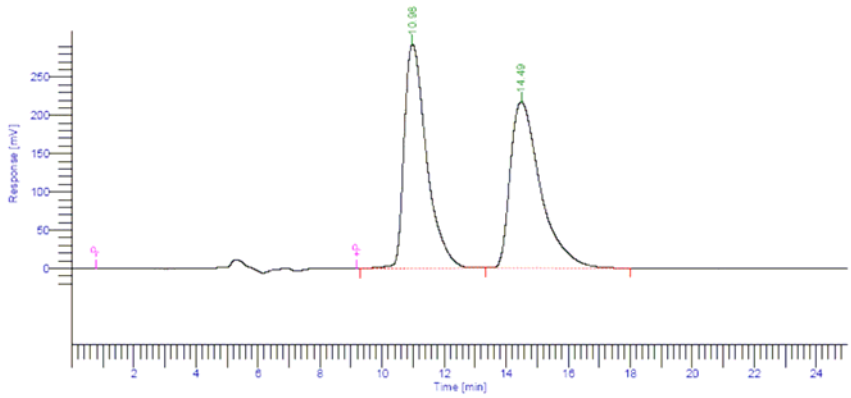

| No. | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|
| 1 | 23.377 | 33661.7 | 1321860.2 | 49.9768 |
| 2 | 35.543 | 27397.3 | 1323087.5 | 50.0232 |
| Total | | 61059.0 | 2644947.7 | 100.0000 |




| No. | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|
| 1 | 23.877 | 6585.1 | 238170.5 | 4.9749 |
| 2 | 36.377 | 80529.7 | 4549228.7 | 95.0251 |
| Total | | 87114.8 | 4787399.2 | 100.0000 |



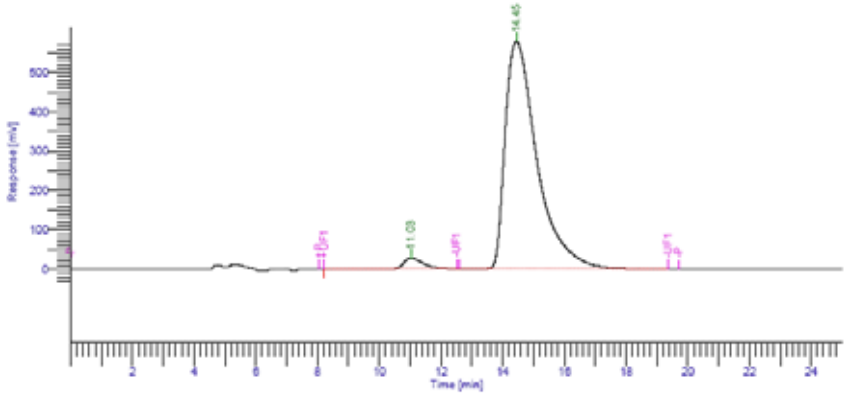

**5b**




ccl

ccl-5-80a-race

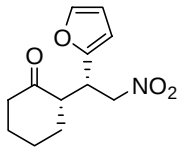
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 10.98 | 1.4704e+07 | 2.933e+05 | 49.93 | 49.93 |
| 2 | 14.49 | 1.4745e+07 | 2.177e+05 | 50.07 | 50.07 |
| | | 2.9449e+07 | 5.111e+05 | 100.00 | 100.00 |



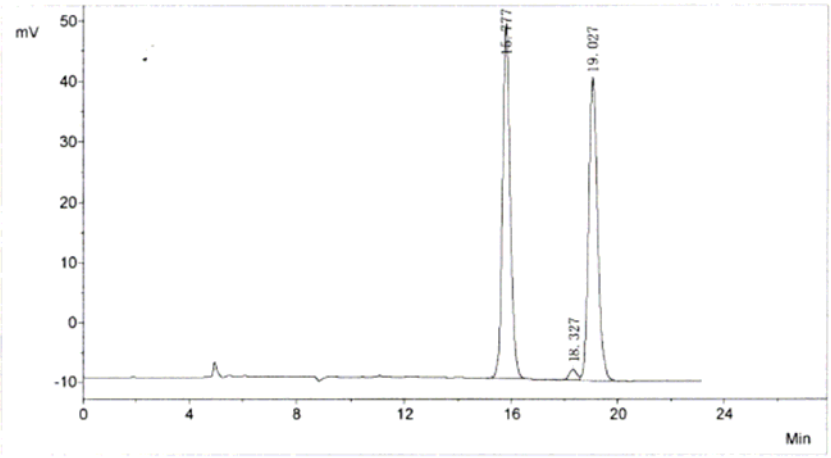

ccl

ccl-5-80a-biao

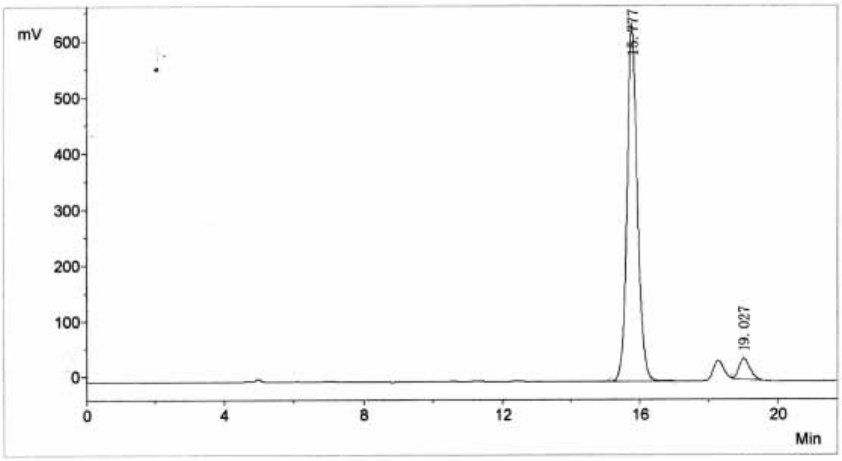
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 11.03 | 1.1321e+06 | 2.658e+04 | 2.67 | 2.67 |
| 2 | 14.45 | 4.1249e+07 | 5.770e+05 | 97.33 | 97.33 |
| | | 4.2381e+07 | 6.036e+05 | 100.00 | 100.00 |



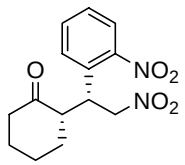

**5c**



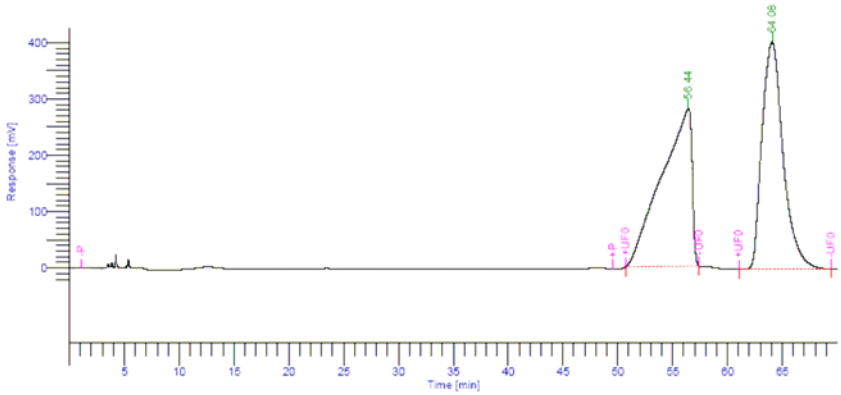

| No. | PeakNo | ID.Name | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 15.777 | 58877.2 | 1190714.3 | 49.4746 |
| 2 | 2 | Unknown | 18.327 | 1675.7 | 33798.2 | 1.4043 |
| 3 | 3 | Unknown | 19.027 | 49934.2 | 1182204.8 | 49.1210 |
| Total | | | | 110487.1 | 2406717.3 | 100.0000 |




| No. | PeakNo | ID.Name | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 15.777 | 633515.0 | 13270053.8 | 94.6858 |
| 2 | 2 | Unknown | 19.027 | 35710.4 | 744778.0 | 5.3142 |
| Total | | | | 669225.4 | 14014831.8 | 100.0000 |



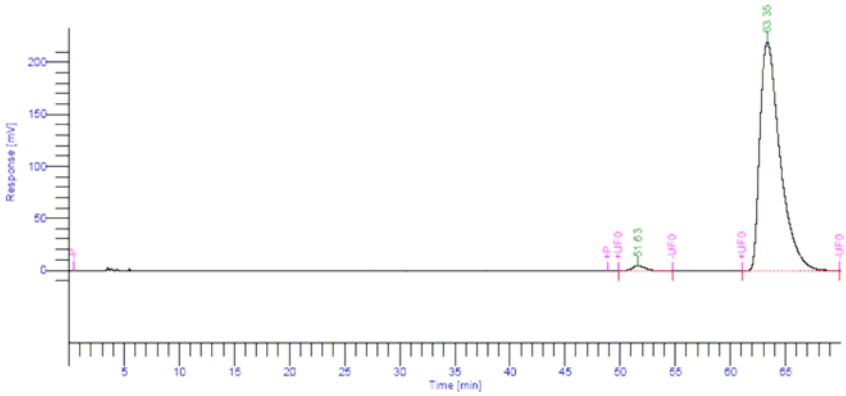

**5d**




ccl

ccl-5-79b-race

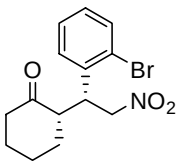
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 56.44 | 5.3760e+07 | 2.800e+05 | 49.92 | 49.92 |
| 2 | 64.08 | 5.3934e+07 | 4.032e+05 | 50.08 | 50.08 |
| | | 1.0769e+08 | 6.833e+05 | 100.00 | 100.00 |



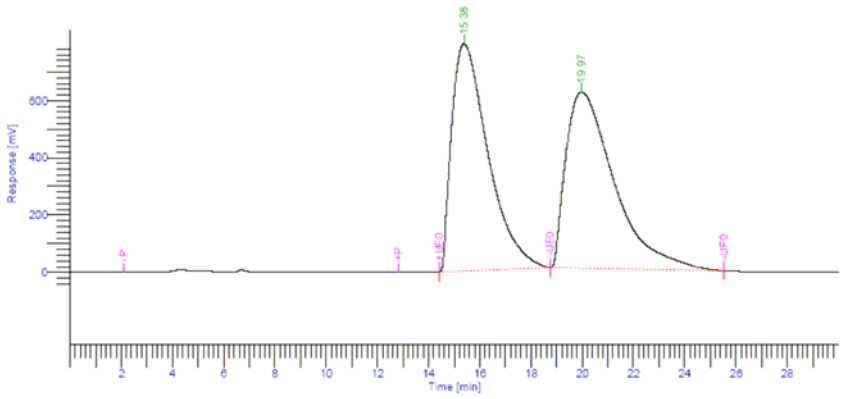

ccl

ccl-5-79b-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 51.63 | 442379.7519 | 4974.9066 | 1.55 | 1.55 |
| 2 | 63.35 | 2.8145e+07 | 2.206e+05 | 98.45 | 98.45 |
| | | 2.8587e+07 | 2.256e+05 | 100.00 | 100.00 |



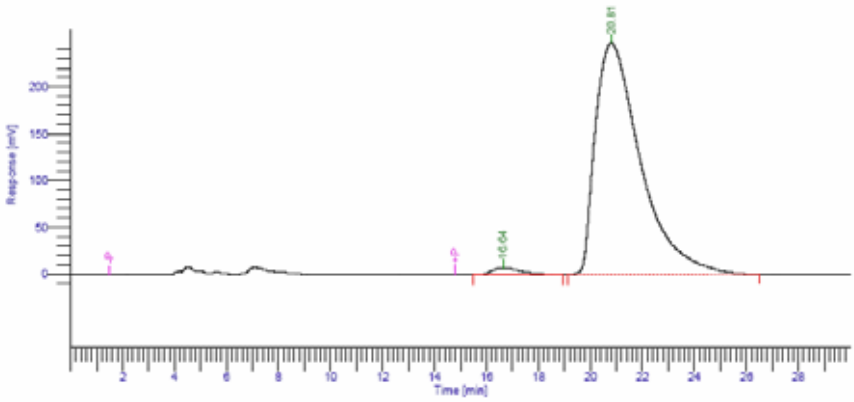

**5e**




ccl

ccl-5-83a-race

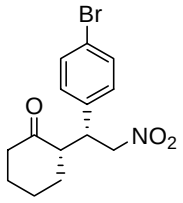
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 15.38 | 7.8169e+07 | 7.943e+05 | 49.24 | 49.24 |
| 2 | 19.97 | 8.0587e+07 | 6.157e+05 | 50.76 | 50.76 |
| | | 1.5876e+08 | 1.410e+06 | 100.00 | 100.00 |



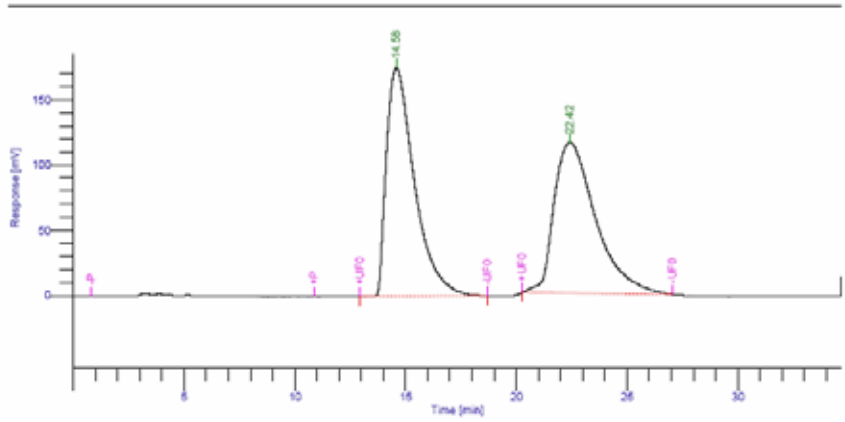

ccl

ccl-5-83a-race

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 16.64 | 544086.9176 | 6896.8825 | 1.78 | 1.78 |
| 2 | 20.81 | 2.9964e+07 | 2.471e+05 | 98.22 | 98.22 |
| | | 3.0508e+07 | 2.540e+05 | 100.00 | 100.00 |



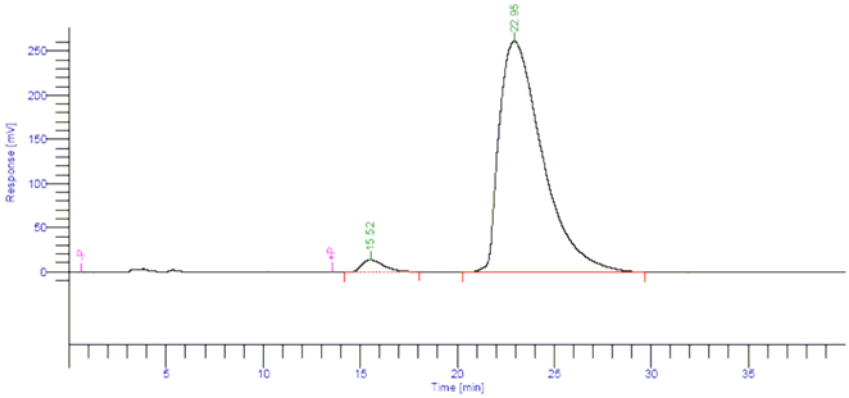

**5f**




ccl

ccl-5-80b-race

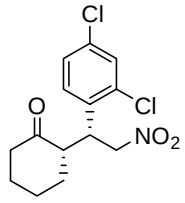
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 14.58 | 1.5361e+07 | 1.750e+05 | 50.53 | 50.53 |
| 2 | 22.42 | 1.5036e+07 | 1.152e+05 | 49.47 | 49.47 |
| | | 3.0397e+07 | 2.902e+05 | 100.00 | 100.00 |



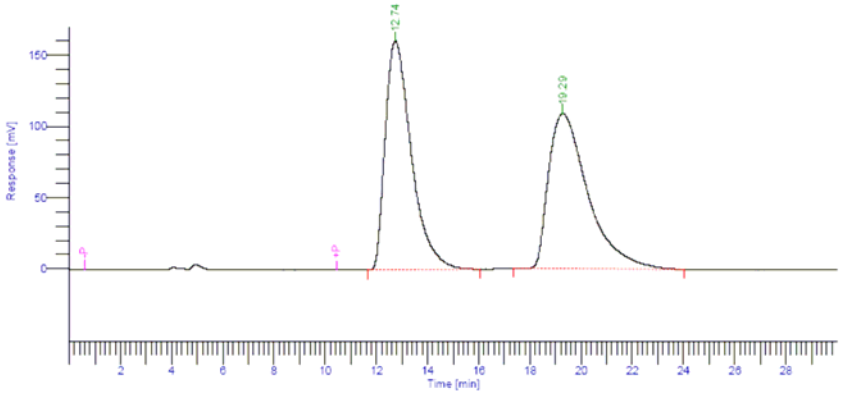

ccl

ccl-5-80b-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 15.52 | 1.1077e+06 | 1.366e+04 | 2.67 | 2.67 |
| 2 | 22.95 | 4.0407e+07 | 2.614e+05 | 97.33 | 97.33 |
| | | 4.1515e+07 | 2.751e+05 | 100.00 | 100.00 |



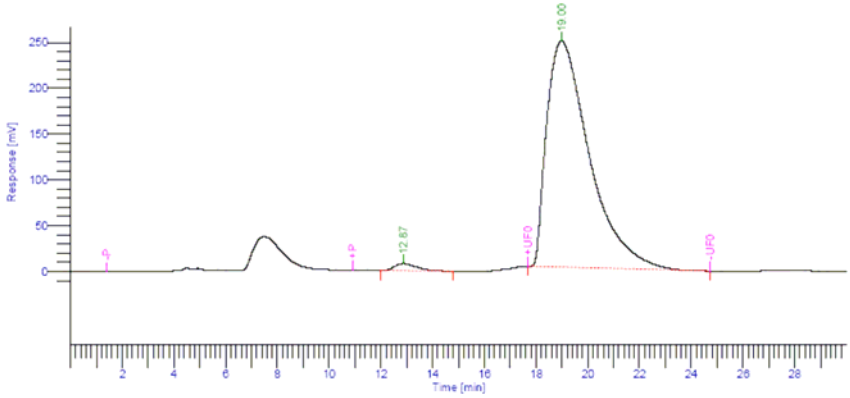

**5g**




ccl

ccl-5-84-race

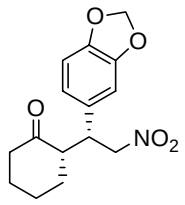
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 12.74 | 1.1415e+07 | 1.608e+05 | 49.60 | 49.60 |
| 2 | 19.29 | 1.1601e+07 | 1.088e+05 | 50.40 | 50.40 |
| | | 2.3017e+07 | 2.696e+05 | 100.00 | 100.00 |



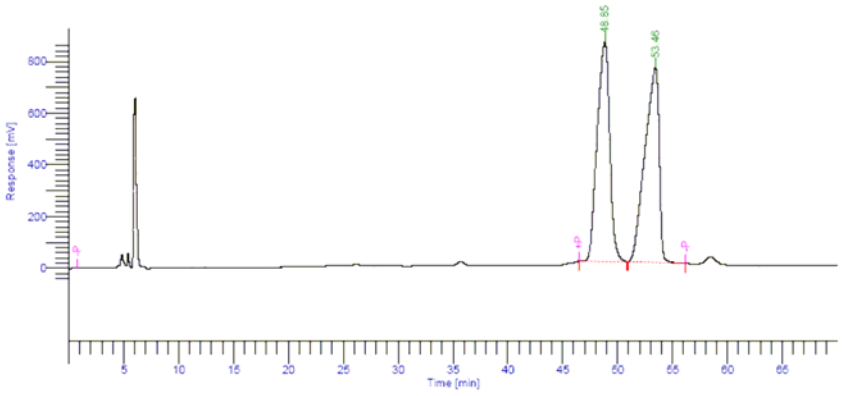

ccl

ccl-5-84-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 12.87 | 467172.0400 | 7798.8437 | 1.63 | 1.63 |
| 2 | 19.00 | 2.8256e+07 | 2.468e+05 | 98.37 | 98.37 |
| | | 2.8723e+07 | 2.546e+05 | 100.00 | 100.00 |



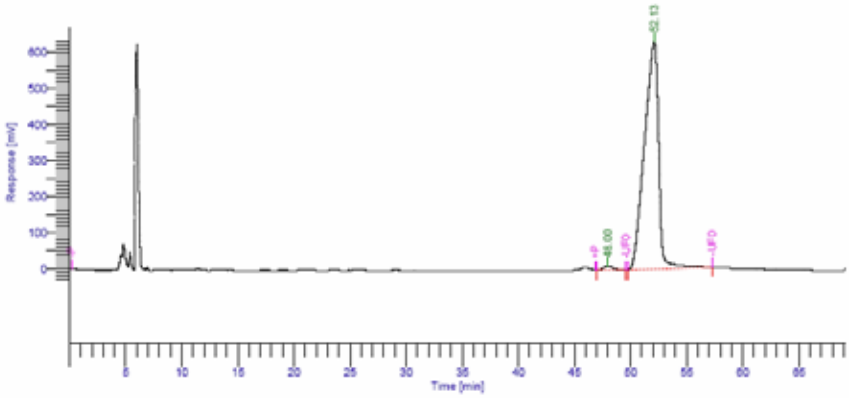
**5h**




ccl

ccl-5-81-biao

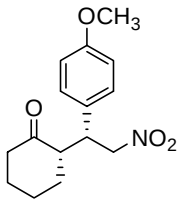
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 48.85 | 6.7568e+07 | 8.484e+05 | 49.81 | 49.81 |
| 2 | 53.46 | 6.8093e+07 | 7.550e+05 | 50.19 | 50.19 |
| | | 1.3566e+08 | 1.603e+06 | 100.00 | 100.00 |



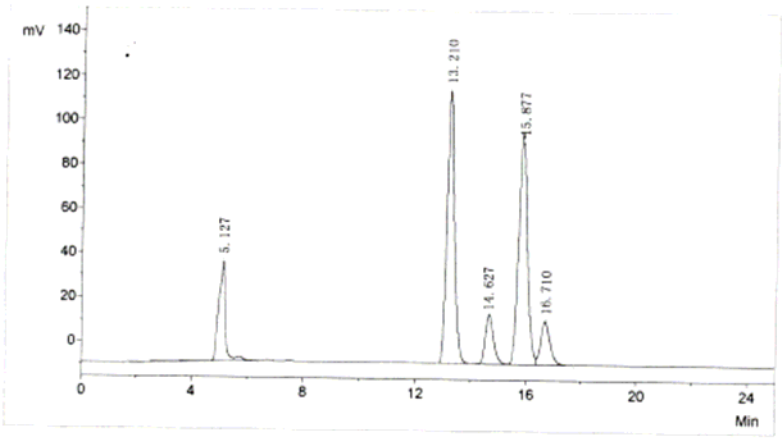

ccl

ccl-5-81-biao

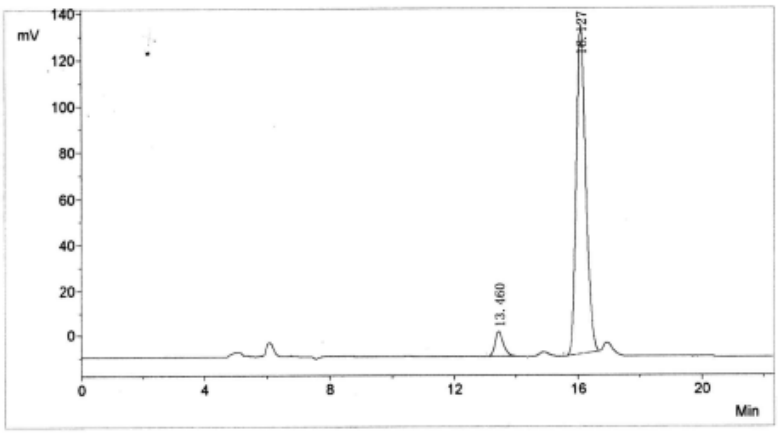
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 48.00 | 676737.0806 | 1.041e+04 | 1.20 | 1.20 |
| 2 | 52.13 | 5.5547e+07 | 6.305e+05 | 98.80 | 98.80 |
| | | 5.6224e+07 | 6.409e+05 | 100.00 | 100.00 |



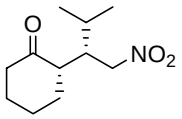

**5i**



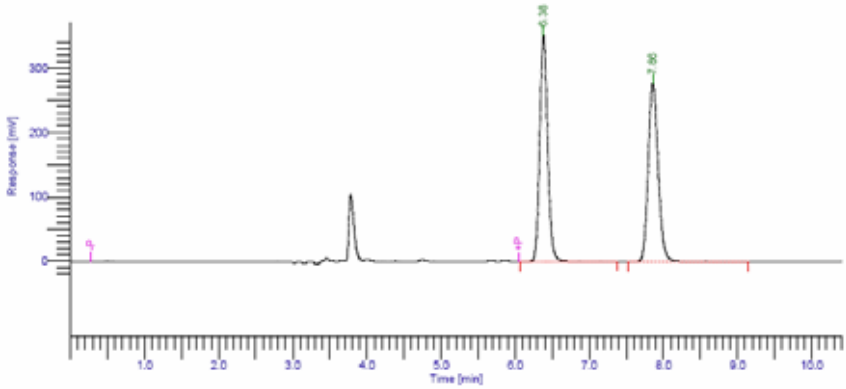

| No. | PeakNo | ID. Name | R. Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 5.127 | 45058.4 | 742648.2 | 11.9475 |
| 2 | 2 | Unknown | 13.210 | 123005.5 | 2298414.4 | 36.9762 |
| 3 | 3 | Unknown | 14.627 | 21073.9 | 450657.8 | 7.2500 |
| 4 | 4 | Unknown | 15.877 | 100093.5 | 2277580.0 | 36.6410 |
| 5 | 5 | Unknown | 16.710 | 19288.3 | 446636.9 | 7.1854 |
| Total | | | | 308519.6 | 6215937.3 | 100.0000 |




| No. | PeakNo | ID. Name | R. Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 13.460 | 10298.3 | 197598.8 | 6.0314 |
| 2 | 2 | Unknown | 16.127 | 141002.5 | 3078544.1 | 93.9686 |
| Total | | | | 151300.8 | 3276142.9 | 100.0000 |



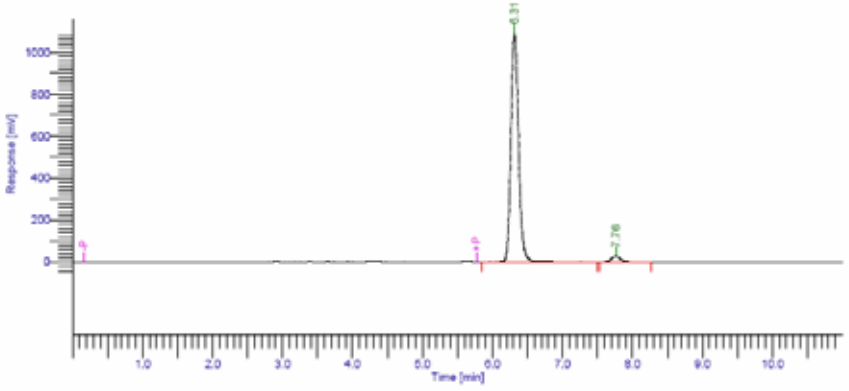

**5j**




ccl

ccl-5-90b-race

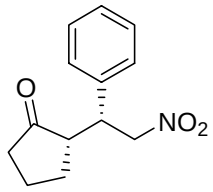
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 6.38 | 2.6235e+06 | 3.511e+05 | 50.06 | 50.06 |
| 2 | 7.86 | 2.6169e+06 | 2.775e+05 | 49.94 | 49.94 |
| | | 5.2404e+06 | 6.286e+05 | 100.00 | 100.00 |



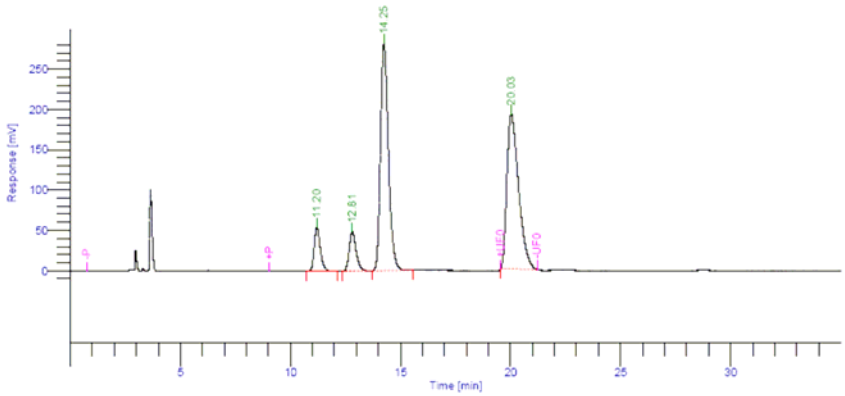

ccl

ccl-5-90b-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 6.31 | 8.3790e+06 | 1.089e+06 | 97.00 | 97.00 |
| 2 | 7.76 | 259396.4899 | 2.919e+04 | 3.00 | 3.00 |
| | | 8.6384e+06 | 1.118e+06 | 100.00 | 100.00 |



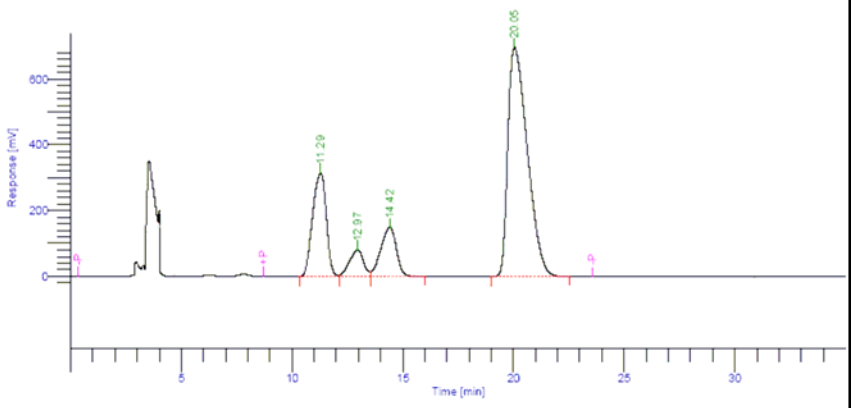

**5k**




ccl

ccl-6-7a-race-biao

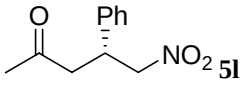
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 11.20 | 1.0576e+06 | 5.511e+04 | 6.52 | 6.52 |
| 2 | 12.81 | 1.0600e+06 | 4.879e+04 | 6.54 | 6.54 |
| 3 | 14.25 | 7.0449e+06 | 2.822e+05 | 43.44 | 43.44 |
| 4 | 20.03 | 7.0564e+06 | 1.918e+05 | 43.51 | 43.51 |
| | | 1.6219e+07 | 5.779e+05 | 100.00 | 100.00 |



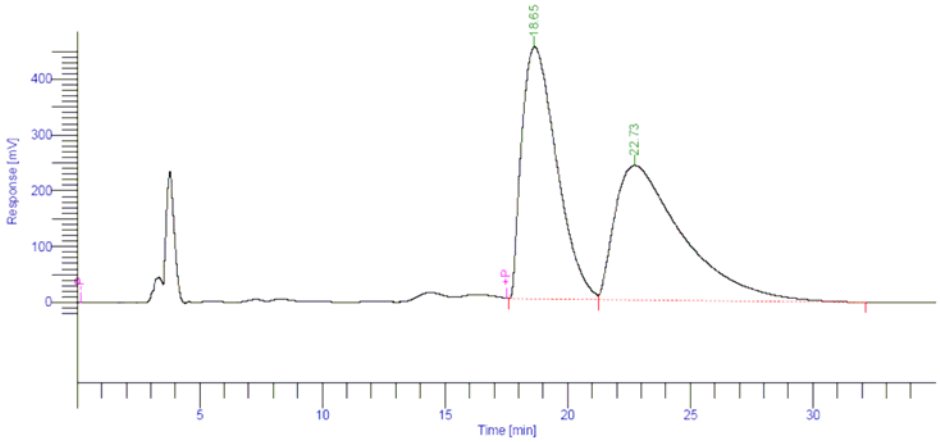

ccl

ccl-6-7a-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 11.29 | 1.3526e+07 | 3.149e+05 | 20.23 | 20.23 |
| 2 | 12.97 | 3.5609e+06 | 8.061e+04 | 5.33 | 5.33 |
| 3 | 14.42 | 7.2435e+06 | 1.493e+05 | 10.84 | 10.84 |
| 4 | 20.05 | 4.2519e+07 | 6.973e+05 | 63.60 | 63.60 |
| | | 6.6849e+07 | 1.242e+06 | 100.00 | 100.00 |



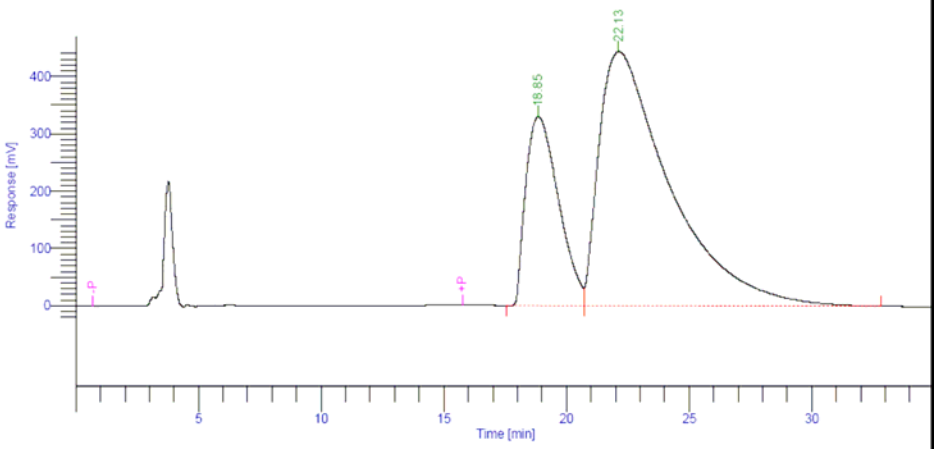

**5l**




ccl

ccl-5-86-race-biao

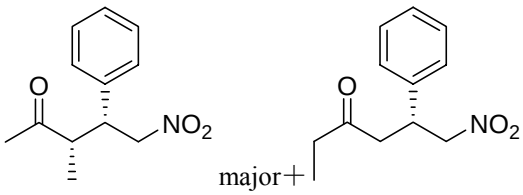
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 18.65 | 4.4634e+07 | 4.515e+05 | 49.06 | 49.06 |
| 2 | 22.73 | 4.6349e+07 | 2.411e+05 | 50.94 | 50.94 |
| | | 9.0983e+07 | 6.926e+05 | 100.00 | 100.00 |



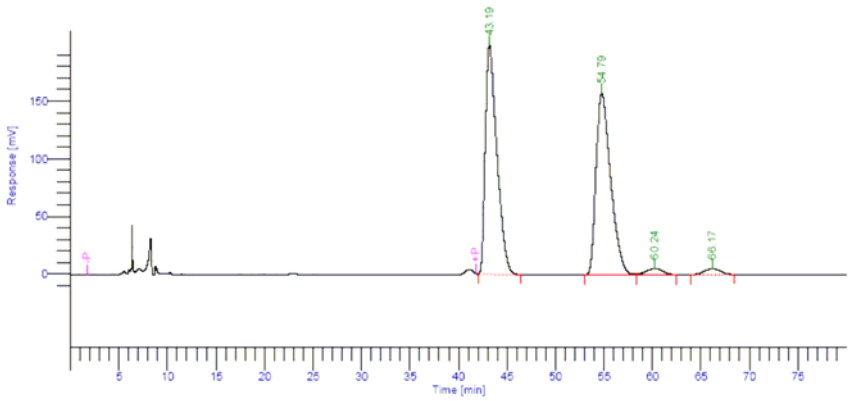

ccl

ccl-5-86-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 18.85 | 3.0622e+07 | 3.302e+05 | 25.66 | 25.66 |
| 2 | 22.13 | 8.8702e+07 | 4.434e+05 | 74.34 | 74.34 |
| | | 1.1932e+08 | 7.735e+05 | 100.00 | 100.00 |



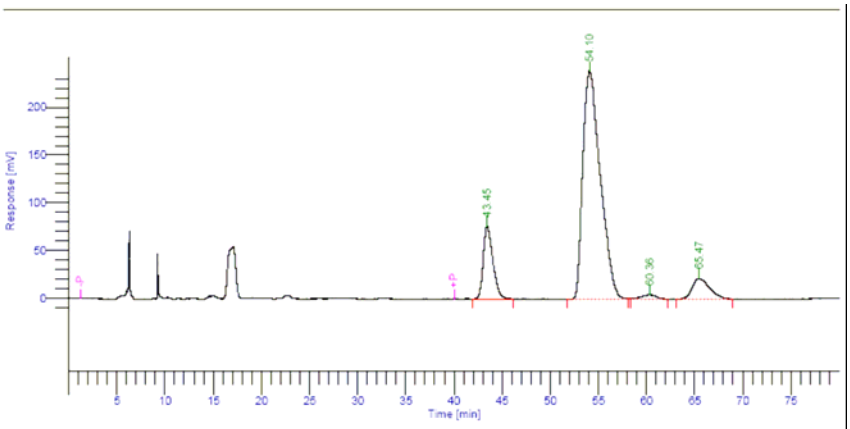

**5m**




ccl

ccl-6-84b-race-biao-qiandianxiao-houdianda

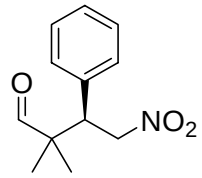
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 43.19 | 1.6459e+07 | 1.993e+05 | 48.02 | 48.02 |
| 2 | 54.79 | 1.6553e+07 | 1.576e+05 | 48.30 | 48.30 |
| 3 | 60.24 | 635720.9397 | 5566.9825 | 1.85 | 1.85 |
| 4 | 66.17 | 624451.5423 | 5387.0975 | 1.82 | 1.82 |
| | | 3.4271e+07 | 3.679e+05 | 100.00 | 100.00 |



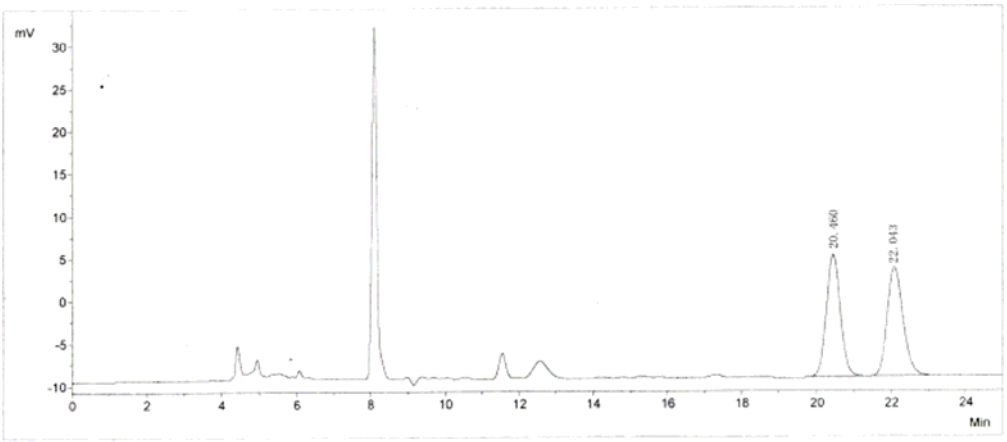

ccl

ccl-6-84b-biao-qiandianxiao-houdianda

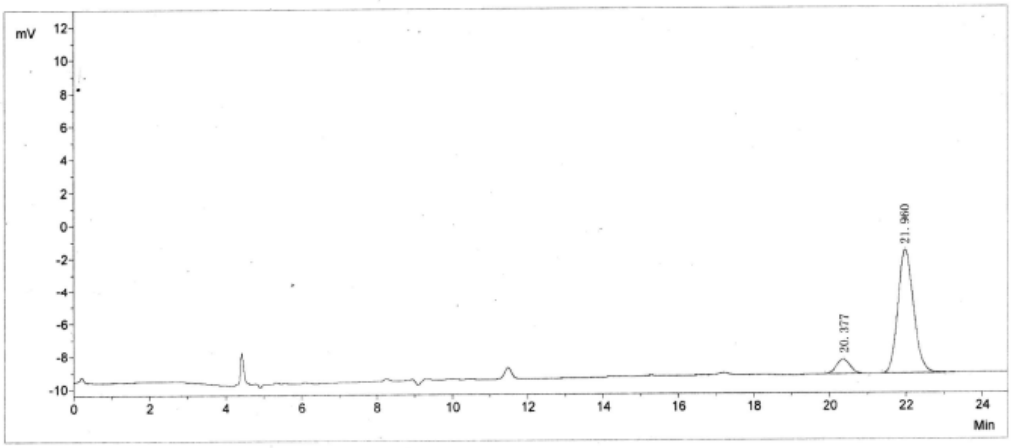
| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 43.45 | 5.2952e+06 | 7.642e+04 | 13.70 | 13.70 |
| 2 | 54.10 | 3.0168e+07 | 2.392e+05 | 78.04 | 78.04 |
| 3 | 60.36 | 459152.1056 | 4211.3901 | 1.19 | 1.19 |
| 4 | 65.47 | 2.7349e+06 | 2.181e+04 | 7.07 | 7.07 |
| | | 3.8657e+07 | 3.417e+05 | 100.00 | 100.00 |



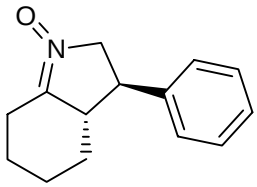

**5n**



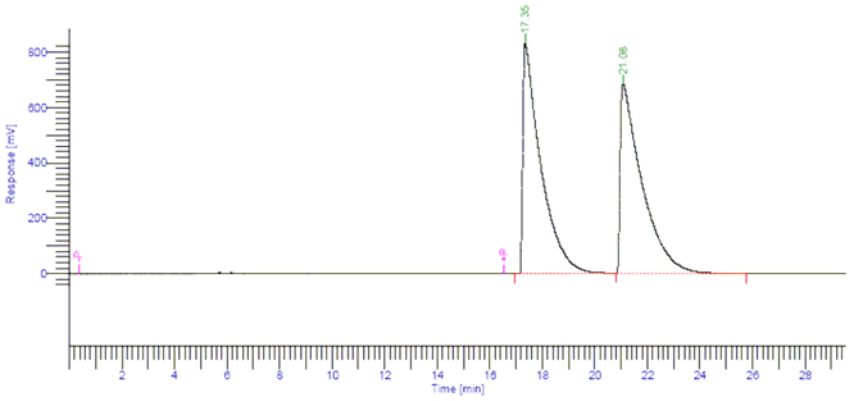

| No. | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|
| 1 | 20.460 | 14223.0 | 371065.2 | 49.9135 |
| 2 | 22.043 | 12401.0 | 372350.6 | 50.0865 |
| Total | | 26624.0 | 743415.8 | 100.0000 |




| No. | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|
| 1 | 20.377 | 871.4 | 21817.1 | 9.1964 |
| 2 | 21.960 | 7529.3 | 215418.2 | 90.8036 |
| Total | | 8400.7 | 237235.3 | 100.0000 |



**6**

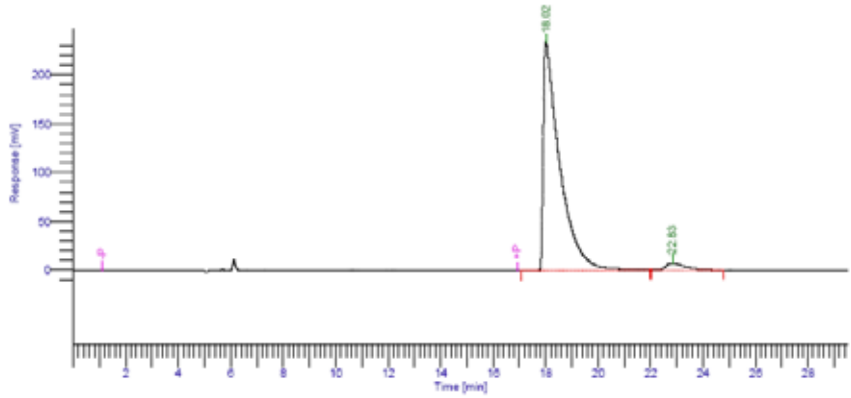



ccl

ccl-6-29-race-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 17.35 | 3.9126e+07 | 8.340e+05 | 50.08 | 50.08 |
| 2 | 21.08 | 3.9007e+07 | 6.841e+05 | 49.92 | 49.92 |
| | | 7.8133e+07 | 1.518e+06 | 100.00 | 100.00 |

Result File :
Sequence File : D:\HPLC\ccl\xmd-1.seq




ccl

ccl-6-30-biao

| Peak # | Time [min] | Area [uV*sec] | Height [uV] | Area [%] | Norm. Area [%] |
|---|---|---|---|---|---|
| 1 | 18.02 | 1.0250e+07 | 2.342e+05 | 96.06 | 96.06 |
| 2 | 22.83 | 419961.8226 | 7652.7734 | 3.94 | 3.94 |
| | | 1.0670e+07 | 2.418e+05 | 100.00 | 100.00 |