**Enantioselective Synthesis of α-Stereogenic γ-Keto Esters via Formal Umpolung**

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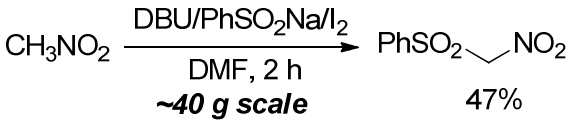
**General**

Unless otherwise mentioned, all the chemicals were purchased from commercial sources. Tetra-*n*-butylammonium Oxone (TBA-Oxone) was prepared according to a known procedure [1]. Silica gel chromatography was performed to isolate the products using 60-200 mesh silica gel (from silicycle) and using suitable solvent systems as eluent. $^{1}H$, $^{13}C$, and $^{19}F$ spectra were recorded on 400 MHz or 500 MHz Varian NMR spectrometers. $^{1}H$ NMR chemical shifts were determined relative to $CDCl_3$ as the internal standard (δ 7.26). $^{13}C$ NMR shifts were determined relative to $CDCl_3$ δ 77.16. $^{19}F$ NMR chemical shifts were determined relative to internal standard $CFCl_3$ at δ 0.00. Mass spectra were recorded on a high resolution mass spectrometer in the ESI mode. The high resolution X-ray crystal structures were derived from the Mo data, while the absolute configurations were determined with Cu data structures. Therefore, the high resolution absolute configurations were obtained from a Cu data set to refine the Mo data structures. The reliability of the absolute configurations thus obtained can be assessed by the Flack x parameter, which is between 0 and 1. X = 0 means the absolute configuration is correct, x = 1 means it is inverse, and x = 0.5 means the crystal is a racemic twin. The Hooft y parameter is similar to Flack x but is more sensitive for light atom structures. The Bayesian Statistics P2 value describes the likelihood that the absolute configuration is correct. P2 = 1 means 100% confidence.

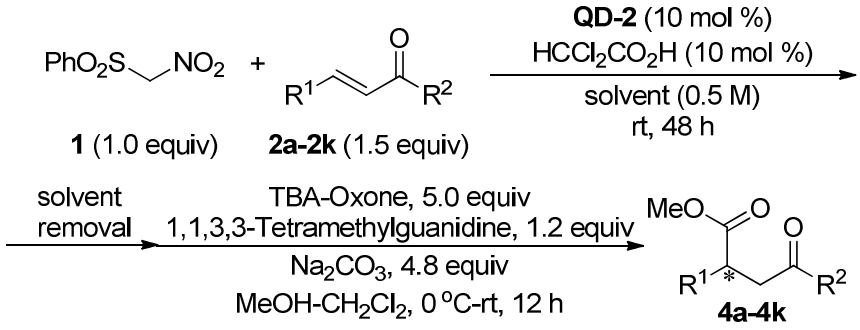
**Large-Scale preparation of nitro(phenylsulfonyl)methane (NSM, 1)**




*purified via extraction and recrystallization*

At 0 °C, to a solution of nitromethane (30.5 g, 0.5 mol) in DMF (600 mL) was added 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 76.1 g, 0.5 mol). The reaction mixture was stirred for 10 min. After the addition of sodium phenylsulfinate (68.9 g, 0.42 mol) and iodine (96.4 g, 0.38 mmol), the solution was allowed to warm to room temperature and stirred for another 1 h. Subsequently, saturated sodium sulfite aq. solution was added until the mixture turned to bright yellow. The pH was adjusted to 1 with concentrated HCl aq. solution. The aqueous layer was extracted three times with $CH_2Cl_2$. The combined $CH_2Cl_2$ solution was then extracted with NaOH aq. solution (2 M) four times [sodium nitro(phenylsulfonyl)methide dissolves in NaOH aq. solution rather than in $CH_2Cl_2$]. The combined aq. layers were further washed with $CH_2Cl_2$ twice and acidified to pH = 1 using HCl aq. solution with ice (ca. 2 M). A large amount of white precipitate (NSM) was observed. This aqueous mixture was then extracted with $CH_2Cl_2$ three times, and the combined organic layers were dried over $MgSO_4$ before the removal of the solvents under vacuum. The crude product was recrystallized in EtOH to afford white crystalline solid as NSM (39.6 g, 47%, based on sodium phenylsulfinate). $^1$H NMR (500 MHz, $CDCl_3$) δ 7.99-7.96 (m, 2H), 7.78-7.82 (m, 1H), 7.68-7.64 (m, 2H), 5.60 (s, 2H).

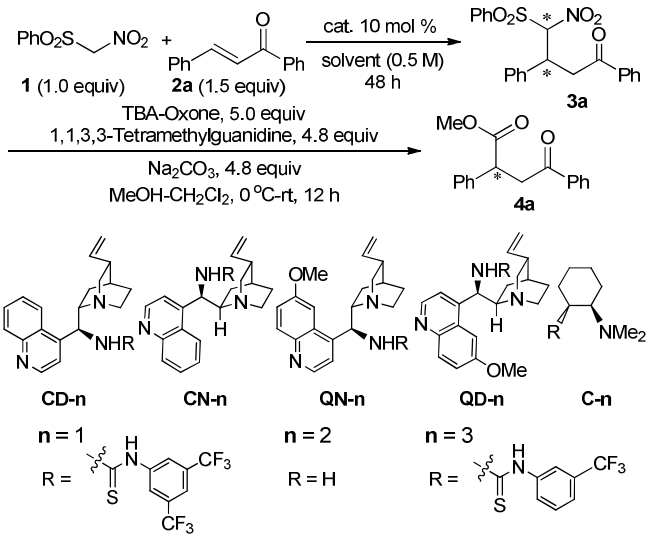
**Typical procedure for one-pot enantioselective synthesis of α-stereogenic γ-keto esters**




To a solution of enone (0.6 mmol, 1.5 equiv) in the indicated solvent (0.8 mL), **QD-2** (12.9 mg, 0.04 mmol, 10 mol %) and $CHCl_2CO_2H$ (5.2 mg, 0.04 mmol, 10 mol %) were added sequentially. NSM (80 mg, 0.4 mmol, 1.0 equiv) was added to the above mentioned solution. The reaction mixture was stirred for 48 h at room temperature before the removal of the reaction solvent under vacuum.

The chunk of the crude Michael adduct was then broken by a spatula and suspended in MeOH (4.4 mL) at 0 □C. To this suspension, 1,1,3,3-tetramethylguanidine (60 μL, 0.48 mmol, 1.2 equiv) was added dropwise. Upon the completion of the addition, a clear solution was formed, which was stirred at 0 $^oC$ for 10 min. TBA-Oxone (1.92 g, ca. 2 mmol active oxidizing agent $Bu_4NHSO_5$, ca. 5.0 equiv) was added in one load, followed by the addition of $Na_2CO_3$ (204 mg, 1.92 mmol, 4.8 equiv) and $CH_2Cl_2$ (6 mL). The reaction mixture was stirred vigorously for 12 h and the solvent was then removed under vacuum. The resulting mixture was passed through a short column (packed with ca. 50 mL silica gel) using $CH_2Cl_2$-Hexanes (1:1) as eluent to remove tetra-*n*-butyl ammonium salts and other inorganic salts. The organic solution was evaporated on a rotavap and the mixture was purified on column chromatography to afford the titled product.

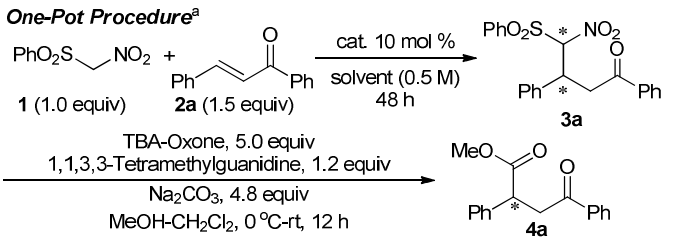
**Table S1**. Thiourea-Catalyzed Synthesis of α-Stereogenic γ-Keto Esters.




| Entry | Catalyst | Temp (°C) | Solvent | Yield (%) | *er* of **4a**[e] |
|---|---|---|---|---|---|
| 1 | **QN-1** | 20 °C | $CDCl_3$ | 6[a] | - |
| 2 | **C-1** | 20 °C | $CDCl_3$ | 5[a] | - |
| 3 | **QN-1** | 20 °C | Toluene | 5[a] | - |
| 4 | **C-1** | 20 °C | Toluene | 4[a] | - |
| 5 | **QN-1** | 20 °C | Toluene[c] | 90[b] | 93:7 |
| 6 | **C-1** | 20 °C | Toluene[c] | 94[b] | 86:14 |
| 7 | **QN-1** | 0 °C[d] | Toluene[c] | 0[a] | - |
| 8 | **QN-1** | -20 °C[d] | Toluene[c] | 0[a] | - |
| 9 | **QN-3** | 20 °C | Toluene[c] | 15[a] | - |
| 10 | **QD-1** | 20 °C | Toluene[c] | 99[b] | 10:90 |
| 11 | **CN-1** | 20 °C | Toluene[c] | 99[b] | 10:90 |
| 12 | **CD-1** | 20 °C | Toluene[c] | 98[b] | 86:14 |
| 13 | **QN-1** | 20 °C | $CH_2Cl_2$[c] | 87[b] | 89:11 |
| 14 | **QN-1** | 20 °C | Xylenes[c] | 95[b] | 94:6 |
| 15 | **QN-1** | -5 °C | $CH_2Cl_2$[c,d] | 0[a] | - |

[a] Conversions to **3a**, determined by $^1$H NMR; [b] isolated yields of **3a**; [c] performed with **1** at 2 M conc.; [d] performed without stirring; [e] measured by chiral HPLC.

**Table S2**. One-Pot Synthesis of α-Stereogenic γ-Keto Esters Catalyzed by Primary Amines.

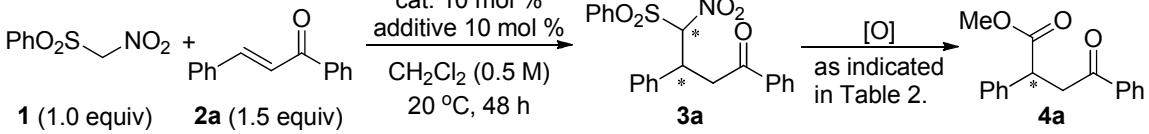



| Entry | Catalyst | Solvent | Yield (%) of **4a**[b] | *er* of **4a**[c] |
|---|---|---|---|---|
| 1 | **CD-2** | $CH_2Cl_2$ | 46 | 20:80 |
| 2 | **CN-2** | $CH_2Cl_2$ | 46 | 83:17 |
| 3 | **QN-2** | $CH_2Cl_2$ | 52 | 18:82 |
| 4 | **QD-2** | $CH_2Cl_2$ | 40 | 81:19 |
| 5 | **CN-2** | $CHCl_3$ | 55 | 83:17 |
| 6 | **CN-2** | MeOH | 61 | 63:37 |
| 7 | **CN-2** | EtOH | 57 | 81:19 |
| 8 | **CN-2** | $ClC_2H_4Cl$ | 53 | 81:19 |
| 9 | **CN-2** | THF | 54 | 60:40 |
| 10 | **CN-2** | $Et_2O$ | 53 | 52:48 |
| 11 | **CN-2** | *t*BuOMe | 65 | 58:42 |

[a]The oxidative methanolysis was performed after the completion of the Michael addition. Crude **3a** was subjected to the oxidation without purification; [b] isolated yields; [c] measured by chiral HPLC.

**Table S3.** Employment of Phenols as Brønsted Acid Additives.

***One-Pot Procedure***

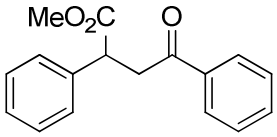



**1** (1.0 equiv) **2a** (1.5 equiv) **3a** **4a**

| entry | catalyst | additive | yield (%)[a] | er of **4a**[b] |
|---|---|---|---|---|
| 1 | **CN-2** | PhOH | 52 | 81:19 |
| 2 | **CN-2** | 4-F-PhOH | 65 | 82:18 |
| 3 | **CN-2** | 4-Cl-PhOH | 61 | 83:17 |
| 4 | **CN-2** | 4-Br-PhOH | 63 | 81:19 |
| 5 | **CN-2** | 4-*t*BuPhOH | 63 | 81:19 |
| 6 | **CN-2** | 4-MeOPhOH | 61 | 81:19 |
| 7 | **CN-2** | 2,4,6-Tri-*t*BuPhOH | 67 | 79:21 |
| 8 | **CN-2** | 2,4-DiPh-PhOH | 59 | 79:21 |
| 9 | **CN-2** | 3-$NO_2$PhOH | 39 | 81:19 |
| 10 | **CN-2** | 3,5-DiF-PhOH | 55 | 82:18 |
| 11 | **CN-2** | 3,5-DiMe-PhOH | 59 | 79:21 |
| 12 | **CN-2** | 2-$NO_2$PhOH | 58 | 83:17 |
| 13 | **CN-2** | 2-OH-PhOH | 59 | 81:19 |
| 14 | **CN-2** | PhOH (20 mol%) | 55 | 80:20 |
| 15 | **CN-2** | PhOH (30 mol%) | 51 | 80:20 |
| 16 | **CN-2** | $CF_3CH_2OH$ | 63 | 81:19 |
| 17 | **CN-2** | $(CF_3)_2CHOH$ | 58 | 79:21 |
| 18 | **CN-2** | $(CF_3)_3COH$ | 52 | 79:21 |

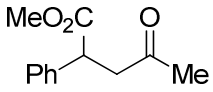
[a] Isolated yields of **4a**; [b] measured by chiral HPLC.

**Methyl 4-oxo-2,4-diphenylbutanoate (4a)**



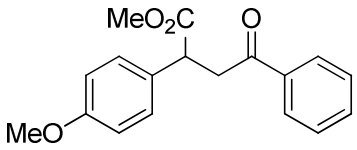

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (55 mg, 51%) was obtained with 96:4 *er*. $^1$H NMR (400 MHz, $CDCl_3$) δ 3.28 (dd, *J* = 18.1, 4.1 Hz, 1H), 3.70 (s, 3H), 3.96 (dd, *J* = 18.1, 10.3 Hz, 1H), 4.30 (dd, *J* = 10.3, 4.0 Hz, 1H), 7.28-7.36 (m, 5H), 7.45-7.47 (m, 2H), 7.55-7.58 (m, 1H), 7.97-7.98 (m, 2H).[2] HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 280 nm): $t_{R1}$ =14.3 min, $t_{R2}$ = 16.1 min.

**Methyl 4-oxo-2-phenylpentanoate (4b)**



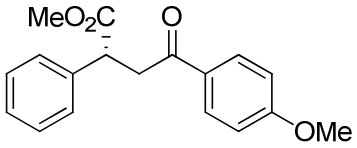

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (37 mg, 45%) was obtained with 73:27 *er*. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.34-7.30 (m, 2H), 7.29-7.24 (m, 3H), 4.11 (dd, *J* = 10.4, 4.2 Hz, 1H), 3.66 (s, 3H), 3.40 (dd, *J* = 18.0, 10.4 Hz, 1H), 2.72 (dd, *J* = 18.0, 4.3 Hz, 1H), 2.18 (s, 3H).[3] HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 93/7, flow 0.5 mL/min, detection at 254 nm): $t_{R1}$ = 27.8 min, $t_{R2}$ = 30.5 min.

**Methyl 2-(4-methoxyphenyl)-4-oxo-4-phenylbutanoate (4d)**



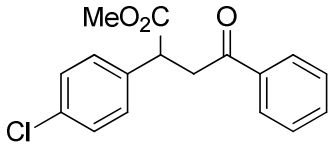
Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (63 mg, 53%) was obtained with 94:6 *er*. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.99-7.95 (m, 2H), 7.59-7.53 (m, 1H), 7.48-7.42 (m, 2H), 7.30-7.25 (m, 2H), 4.25 (dd, *J* = 10.2, 4.2 Hz, 1H), 3.91 (dd, *J* = 18.0, 10.2 Hz, 1H), 3.80 (s, 3H), 3.69 (s, 2H), 3.26 (dd, *J* = 18.0, 4.2 Hz, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 197.9, 174.3, 159.2, 136.6, 133.4, 130.5, 129.0, 128.7, 128.2, 114.4, 55.4, 52.5, 45.6, 43.0. MS (EI): 298, 238, 207, 161, 133, 105, 91, 77, 51. HRMS: Calcd for $C_{18}H_{19}O_4$ ($MH^+$): 299.1278; Found: 299.1276. HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 18.5 min, $t_{R2}$ = 25.6 min.

**(*R*)-Methyl 4-(4-methoxyphenyl)-4-oxo-2-phenylbutanoate (4e)**



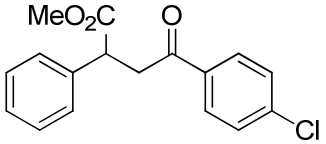

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (66 mg, 55%) was obtained with with 97:3 *er*. The single crystal of **4e** was obtained via crystallization from acetonitrile. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.97-7.92 (m, 2H), 7.38-7.32 (m, 4H), 7.31–7.26 (m, 1H), 6.96 – 6.88 (m, 2H), 4.29 (dd, *J* = 10.3, 4.1 Hz, 1H), 3.90 (dd, *J* = 17.8, 10.3 Hz, 1H), 3.86 (s, 1H), 3.69 (s, 1H), 3.23 (dd, *J* = 10.3, 4.1 Hz, 1H).[2] HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 40.2 min, $t_{R2}$ = 48.4 min.

**Methyl 2-(4-chlorophenyl)-4-oxo-4-phenylbutanoate (4f)**



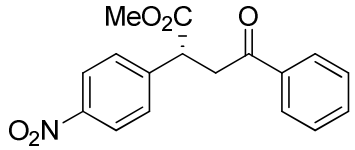

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (67 mg, 55%) was obtained with 96:4 *er*. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2H), 7.57 (m, 1H), 7.49-7.43 (m, 2H), 7.33-7.28 (m, 4H), 4.28 (dd, *J* = 9.9, 4.5 Hz, 1H), 3.91 (dd, *J* = 18.0, 9.9 Hz, 1H), 3.70 (s, 3H), 3.27 (dd, *J* = 18.0, 4.5 Hz, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 197.4, 173.7, 137.0, 136.4, 133.7, 133.6, 129.4, 129.2, 128.8, 128.2, 52.6, 45.9, 42.7. MS (EI): 272, 270, 209, 207, 167, 165, 105, 77, 50. HRMS: Calcd for $C_{17}H_{15}ClNaO_3$ ($MNa^+$): 325.0605; Found: 325.0602. HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 220 nm): $t_{R1}$ = 11.9 min, $t_{R2}$ = 15.7 min.

**Methyl 4-(4-chlorophenyl)-4-oxo-2-phenylbutanoate (4g)**



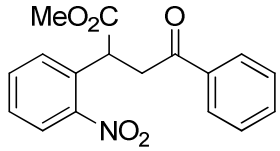

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (80 mg, 66%) was obtained with 94:6 *er*. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.95- 7.86 (m, 2H), 7.47- 7.39 (m, 2H), 7.38-7.27 (m, 5H), 4.29 (dd, *J* = 10.3, 4.0 Hz, 1H), 3.92 (dd, *J* = 18.0, 10.3 Hz, 1H), 3.70 (s, 3H), 3.22 (dd, *J* = 10.3, 4.0 Hz, 1H).[2] HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 20.1 min, $t_{R2}$ = 24.1 min.

**(*R*)-Methyl 2-(4-nitrophenyl)-4-oxo-4-phenylbutanoate (4h)**




Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (71 mg, 57%) was obtained with 94:6 *er*. The single crystal of **4h** was obtained via crystallization from ethanol-water solution. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 8.24-8.16 (m, 2H), 7.99-7.92 (m, 2H), 7.62-7.56 (m, 1H), 7.56-7.51 (m, 2H), 7.50-7.44 (m, 2H), 4.44 (dd, *J* = 9.3, 5.0 Hz, 1H), 3.95 (dd, *J* = 18.0, 9.3 Hz, 1H), 3.72 (s, 3H), 4.44 (dd, *J* = 18.0, 5.0 Hz, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 196.8, 172.8, 147.6, 145.8, 136.2, 133.8, 129.1, 128.9, 128.2, 124.2, 52.9, 46.4, 42.3. MS (EI): 313, 281, 207, 105, 77, 51. HRMS: Calcd for $C_{17}H_{16}NO_5$ ($MH^+$): 314.1023; Found: 314.1021. HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 50.3 min, $t_{R2}$ = 58.7 min.

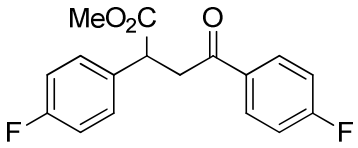
**Methyl 2-(2-nitrophenyl)-4-oxo-4-phenylbutanoate (4i)**




Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (81 mg, 65%) was obtained with 97:3 *er*. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 8.02-7.94 (m, 3H), 7.62 -7.53 (m, 3H), 7.49- 7.42 (m, 3H), 4.91 (dd, *J* = 7.9, 5.1 Hz, 1H), 4.03 (dd, *J* = 18.1, 7.9 Hz, 1H), 3.69 (s, 3H), 3.47 (dd, *J* = 18.1, 5.1 Hz, 1H).[4] HPLC

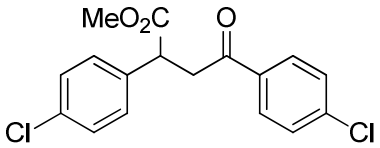
(DAICEL CHIRALCEL OD-H, hexane/2-propanol = 92/8, flow 0.5 mL/min, detection at 254 nm): $t_{R1}$ = 38.8 min, $t_{R2}$ = 40.8 min.

**Methyl 2,4-bis(4-fluorophenyl)-4-oxobutanoate (4j)**



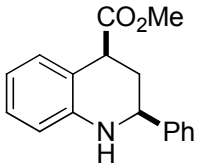

Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (73 mg, 60%) was obtained with 94:6 *er*. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 3.23 (dd, *J* = 17.9, 4.4 Hz, 1H), 3.71 (s, 3H), 3.89 (dd, *J* = 17.9, 10.0 Hz, 1H), 4.39 (dd, *J* = 10.0, 4.3 Hz, 1H), 7.04 (m, 2H), 7.14 (m, 2H), 7.33 (m, 2H), 8.01 (m, 2H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 42.8, 45.7, 52.6, 115.9 (d, *J* = 22.0 Hz, 1C), 116.0 (d, *J* = 21.4 Hz, 1C), 129.6 (d, *J* = 8.1 Hz, 1C), 130.9 (d, *J* = 9.4 Hz, 1C), 132.9 (d, *J* = 3.1 Hz, 1C), 134.1 (d, *J* = 3.4 Hz, 1C), 162.4 (d, *J* = 246.5 Hz, 1C), 166.1 (d, *J* = 225.3 Hz, 1C), 173.8, 196.0. $^{19}$F NMR (470 MHz, $CDCl_3$) δ -105.1 (m, 1F), -115.3 (m, 1F). MS (EI): 304, 272, 149, 123, 95, 75. HRMS: Calcd for $C_{17}H_{14}F2NaO_3$ ($MNa^+$): 327.0803; Found: 327.0799. HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 14.4 min, $t_{R2}$ = 23.9 min.

**Methyl 2,4-bis(4-chlorophenyl)-4-oxobutanoate (4k)**

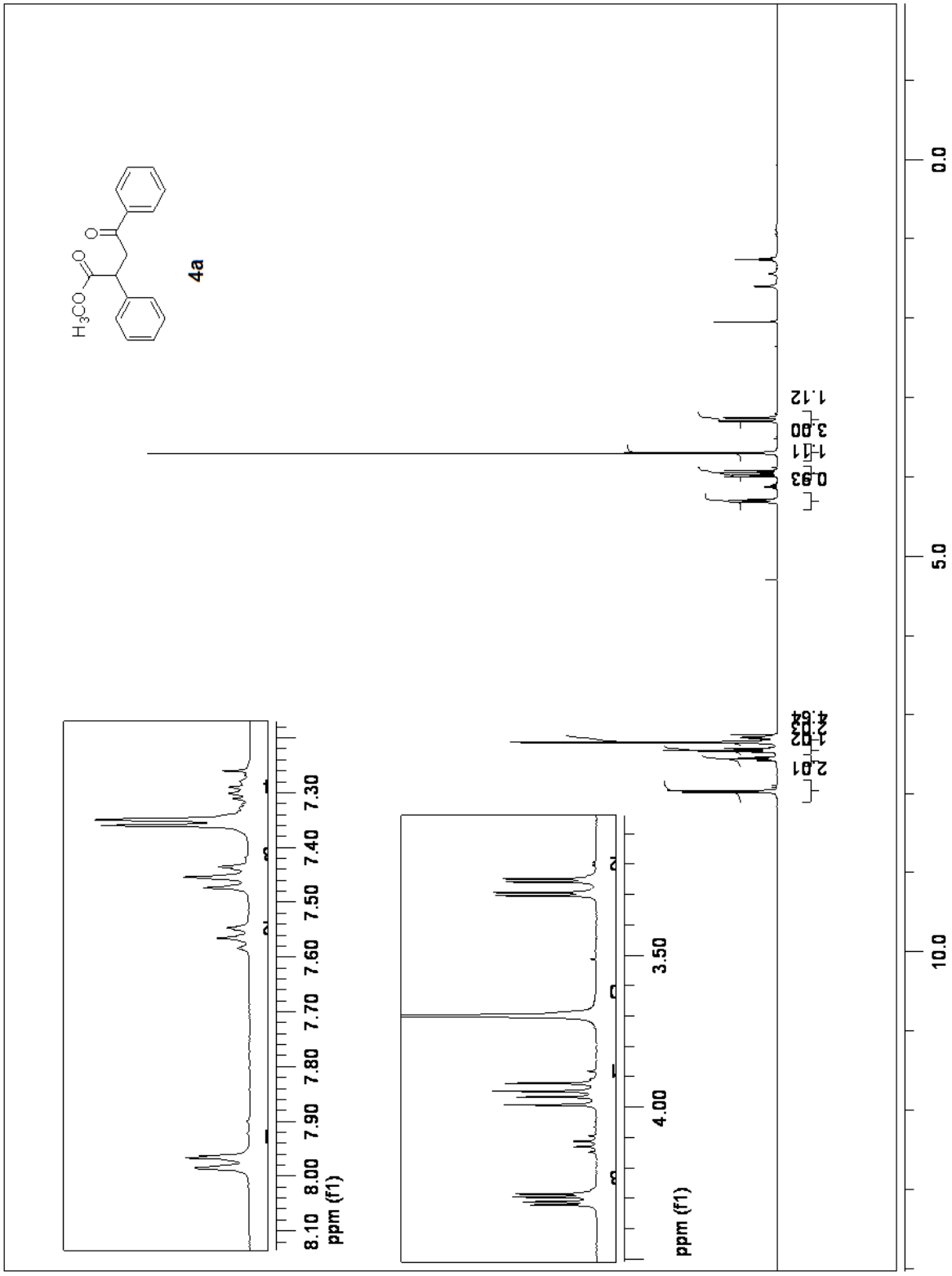


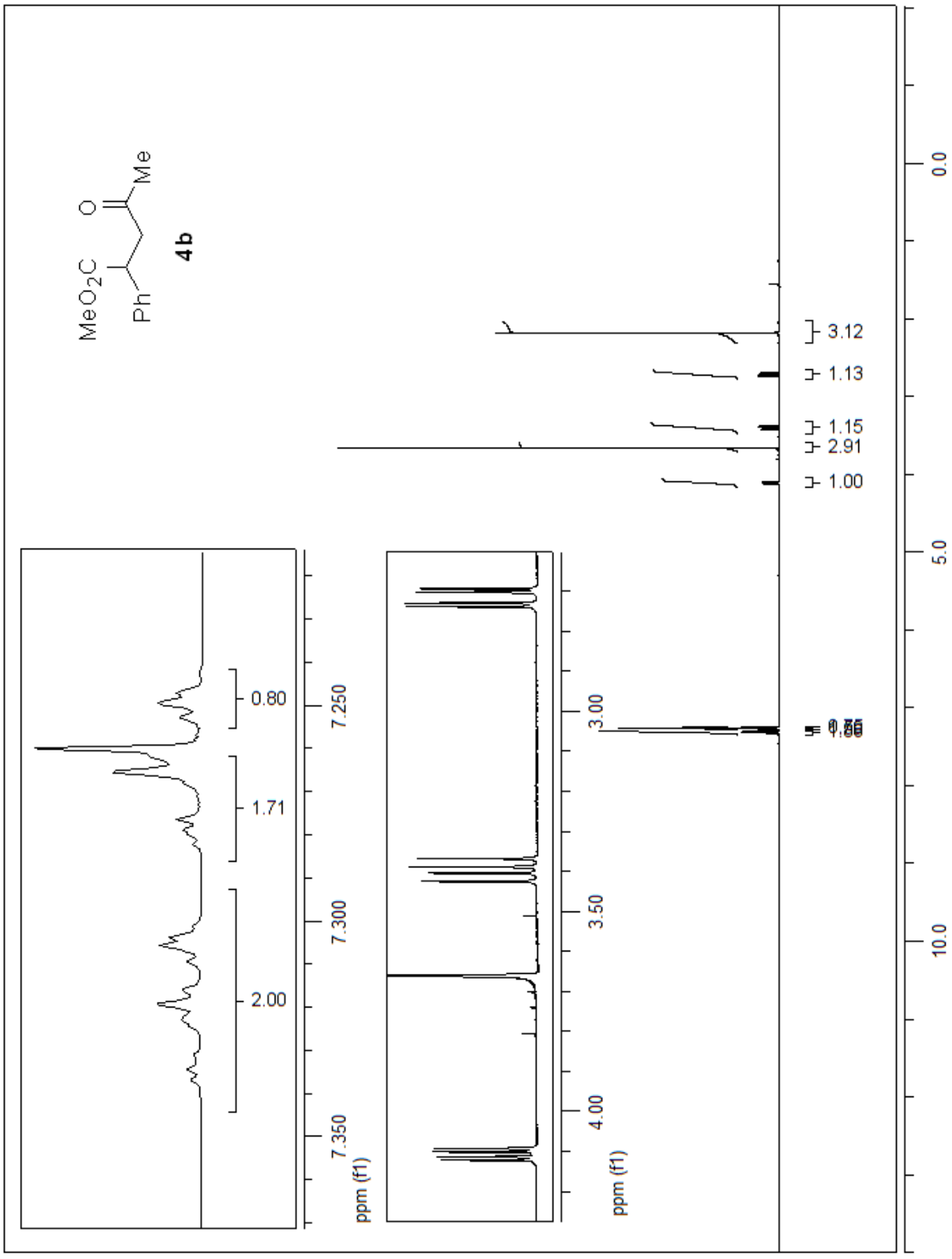
Prepared according to the typical procedure on a 0.4 mmol scale (based on NSM). A colorless oil (65 mg, 48%) was obtained with 93:7 *er*. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.92-7.88 (m, 2H), 7.45- 7.41 (m, 2H), 7.34- 7.26 (m, 4H), 4.27 (dd, *J* = 9.9, 4.4 Hz, 1H), 3.87 (dd, *J* = 18.0, 9.9 Hz, 1H), 3.69 (s, 3H), 3.21 (dd, *J* = 18.0, 4.4 Hz, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$) δ 200.0, 196.2, 173.6, 140.1, 136.8, 134.7, 133.8, 129.6, 129.4, 129.3, 129.2, 52.7, 45.8, 42.6. MS (EI): 306, 304, 241, 167, 165, 141, 139, 113, 111, 77, 51. HRMS: Calcd for $C_{17}H_{14}Cl_2NaO_3$ ($MNa^+$): 359.0212; Found: 259.0213. HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 98/2, flow 1.0 mL/min, detection at 254 nm): $t_{R1}$ = 17.2 min, $t_{R2}$ = 20.8 min.

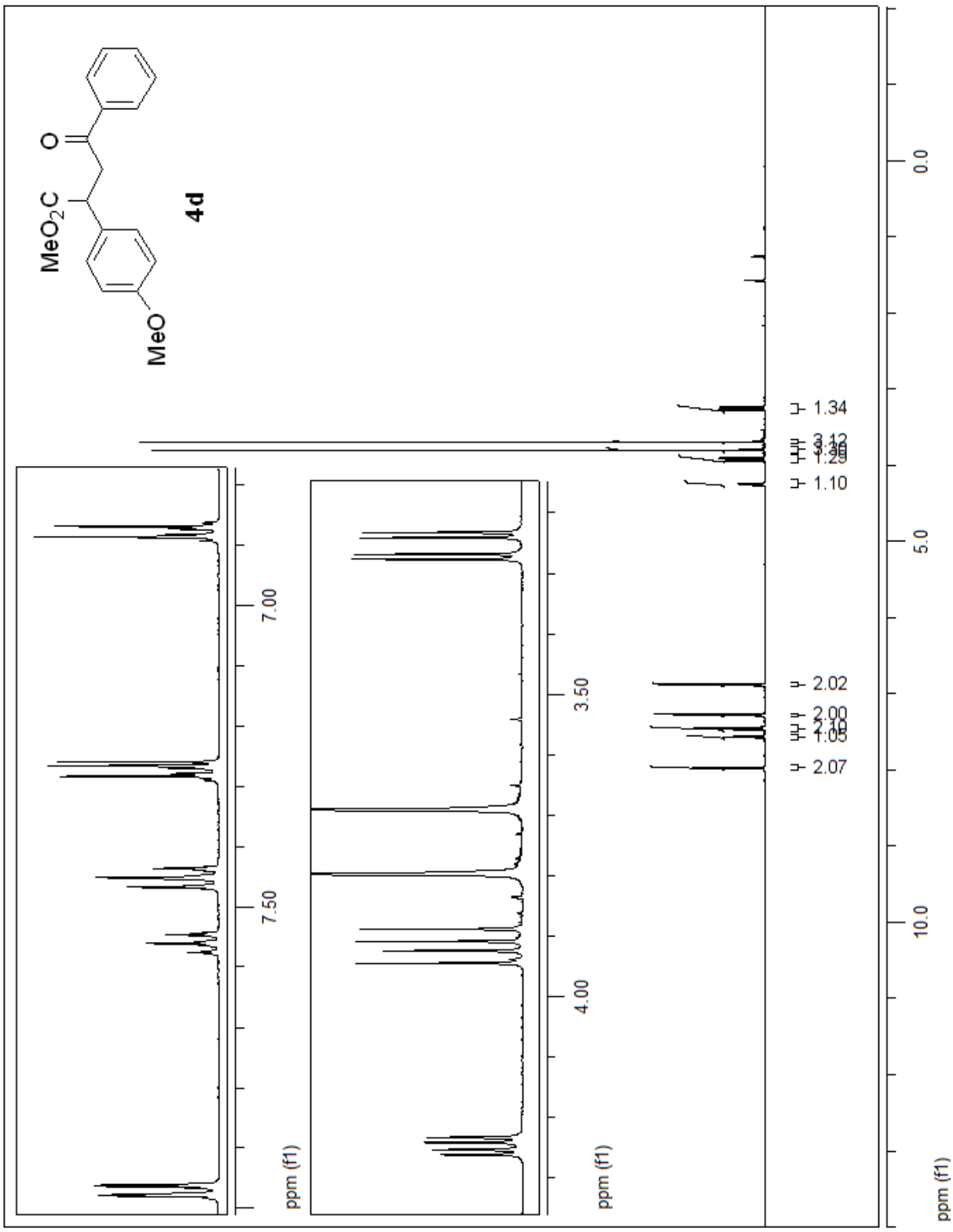
**_cis_-Methyl 2-phenyl-1,2,3,4-tetrahydroquinoline-4-carboxylate (5)**

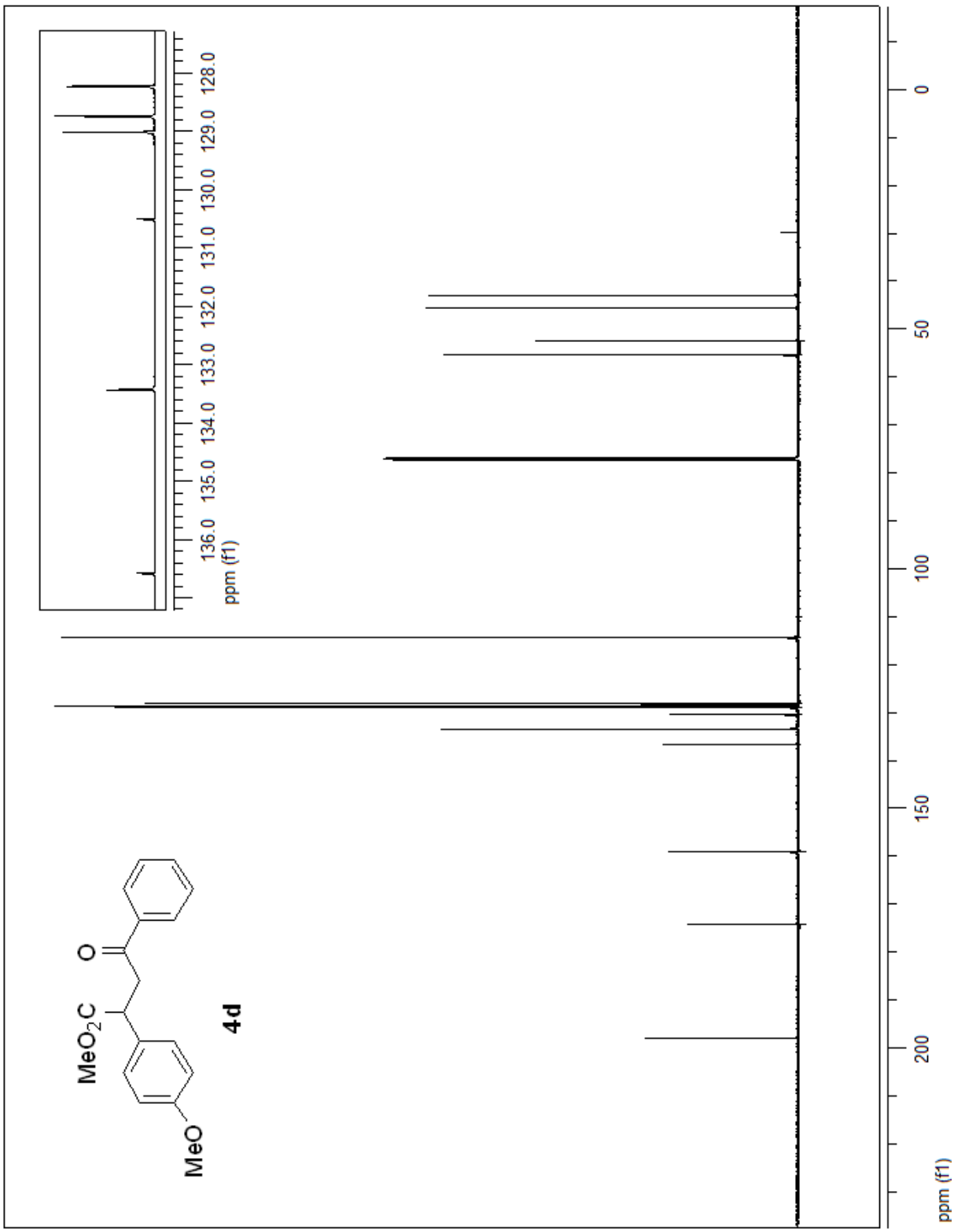


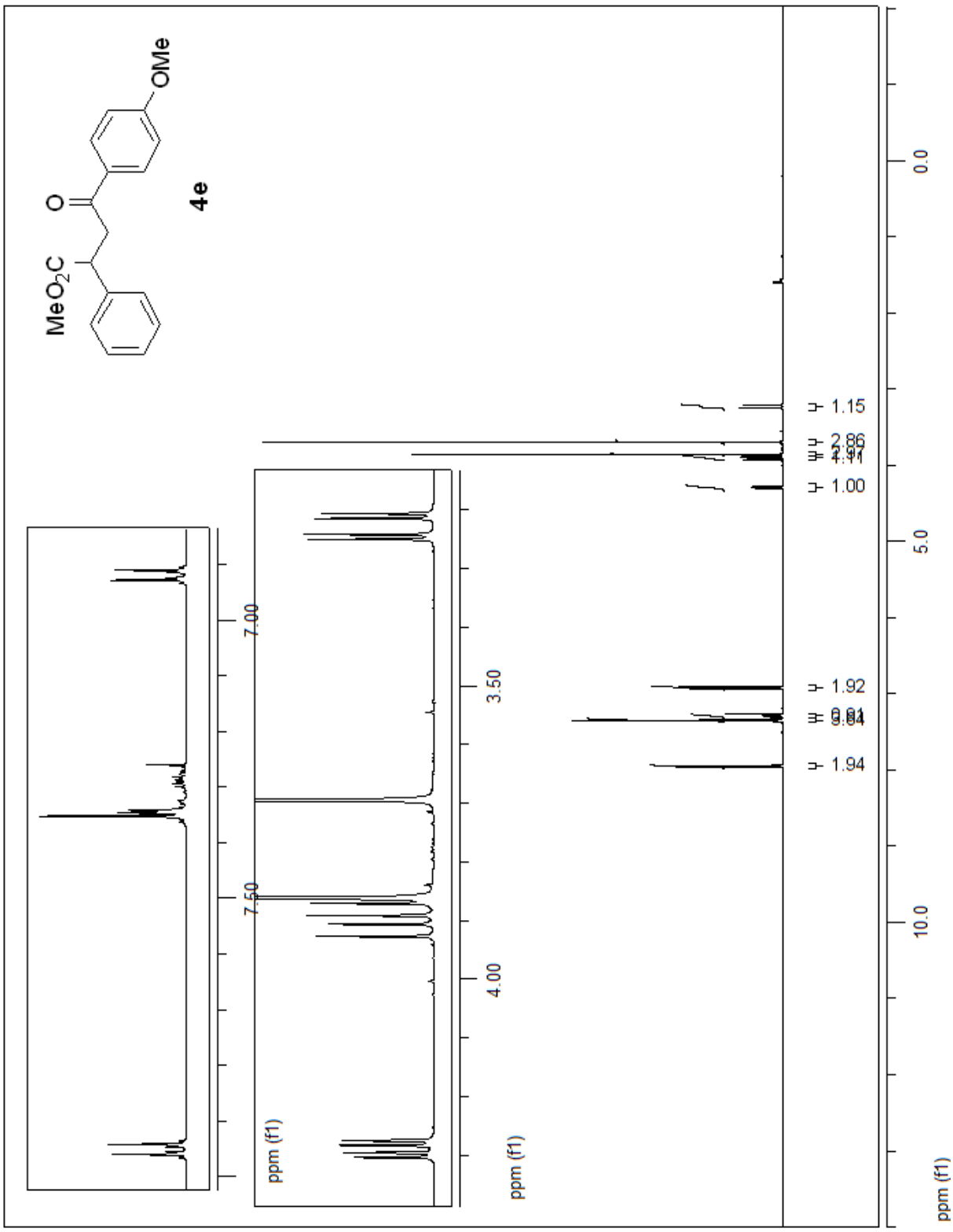

Prepared according to a known procedure[4] with **4i** on a 0.25 mmol scale. To avoid undesired demethylcarboxylation reaction, the reaction time was shortened to 1 h. A light yellow oil (54 mg, 81%) was obtained with 97:3 *er*. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.45-7.43 (m, 2H), 7.42-7.35 (m, 2H), 7.32 (ddt, *J* = 8.0, 7.1, 3.5 Hz, 1H), 7.12-7.02 (m, 2H), 6.74-6.68 (m, 1H), 6.58 (d, *J* = 8.0 Hz, 1H), 4.43 (dd, *J* = 10.7, 3.1 Hz, 1H), 4.12 (dd, *J* = 11.4, 6.0 Hz, 1H), 4.00 (br, 1H), 3.72 (s, 3H), 2.45-2.27 (m, 2H). HPLC (DAICEL CHIRALCEL OD-H, hexane/2-propanol = 90/10, flow 0.5 mL/min, detection at 254 nm): $t_{R1}$ = 32.0 min, $t_{R2}$ = 35.3 min.

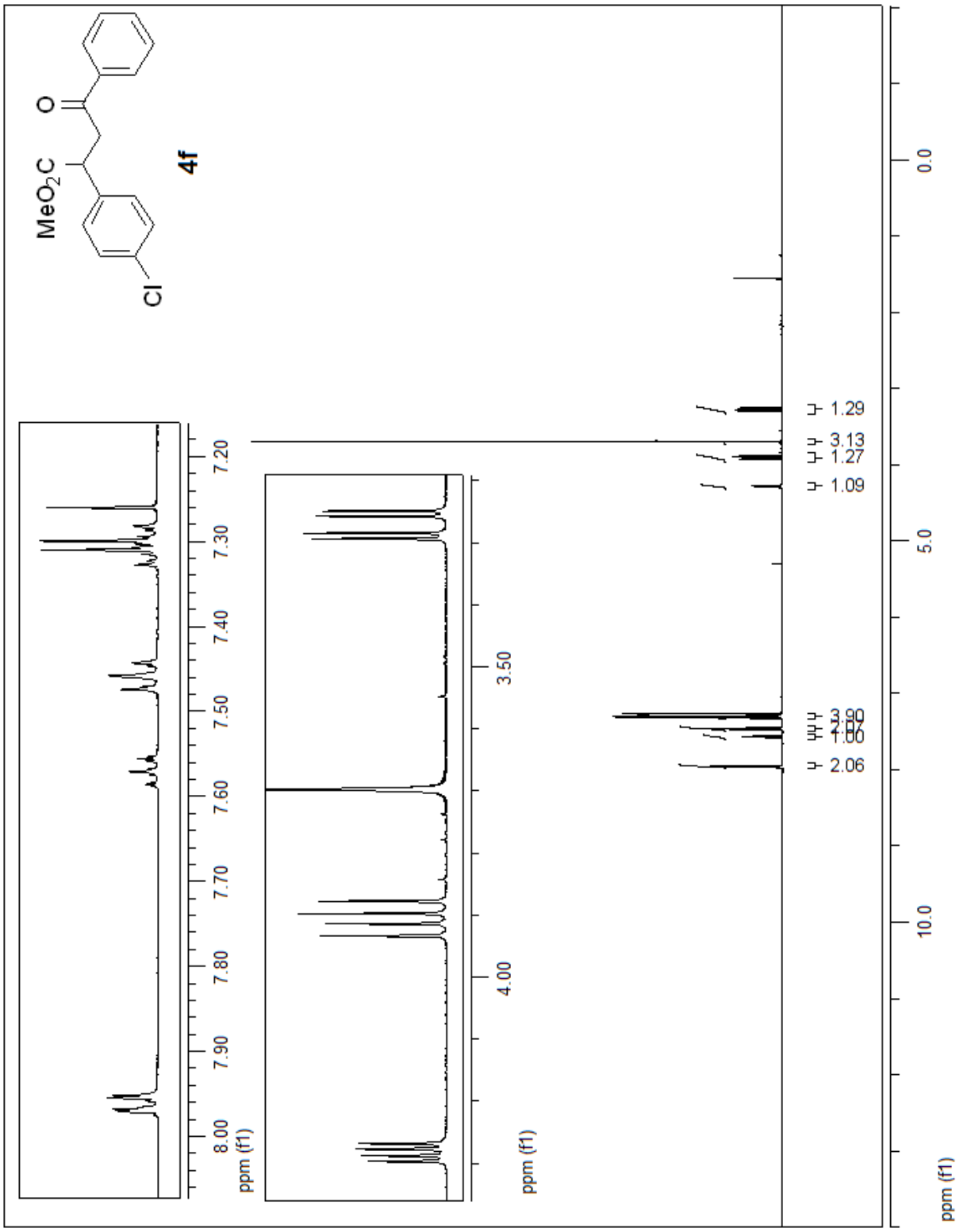

4a
H3CO
O
O
2.01
4.22
4.63
1.12
3.00
1.11
0.93
10.0
5.0
0.0
8.10 8.00 7.90 7.80 7.70 7.60 7.50 7.40 7.30
ppm (f1)
4.00
3.50
ppm (f1)

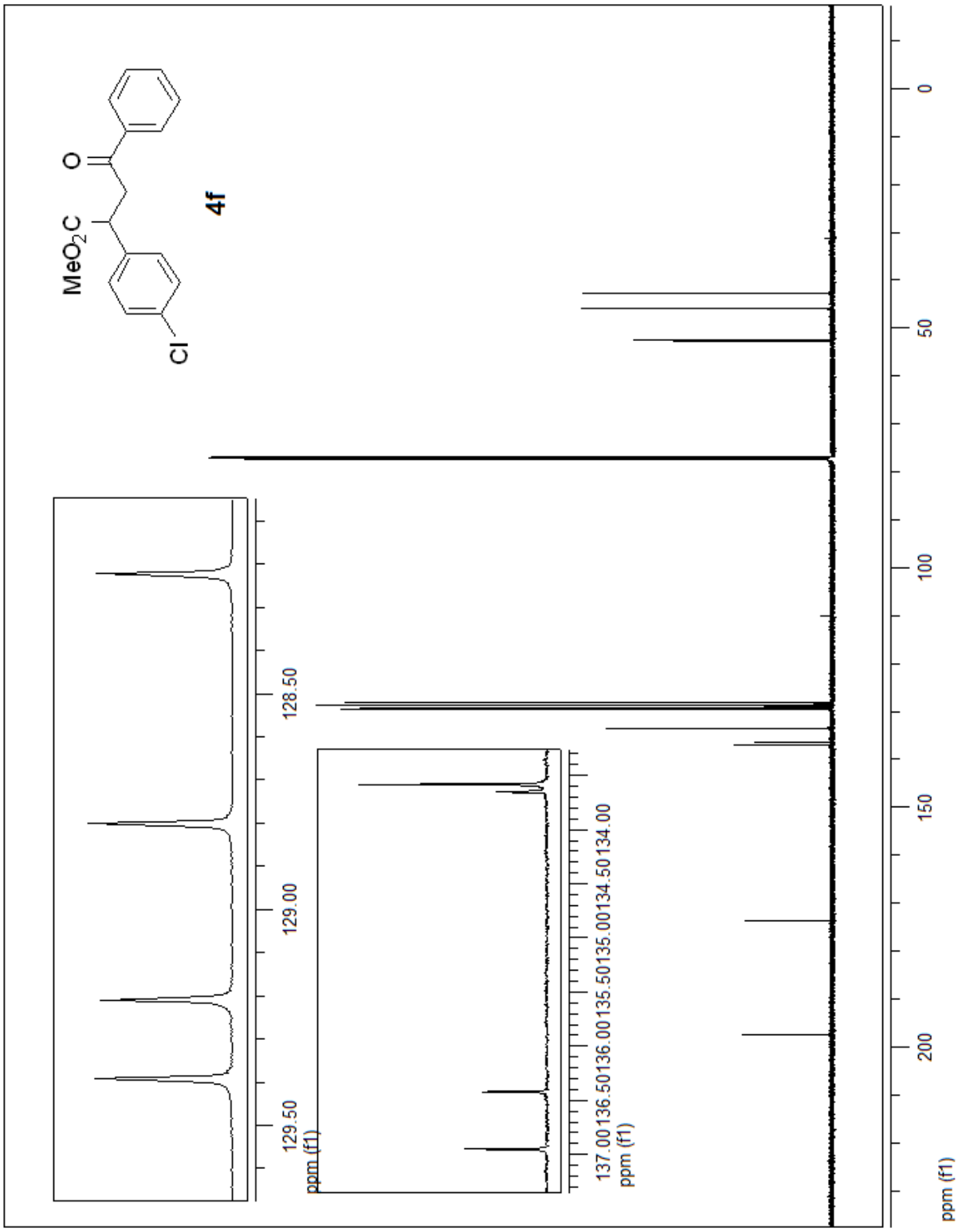

MeO2C
O
Me
Ph
4b
3.12
1.13
1.15
2.91
1.00
0.80
1.71
2.00
7.350
7.300
7.250
ppm (f1)
4.00
3.50
3.00
ppm (f1)
10.0
5.0
0.0

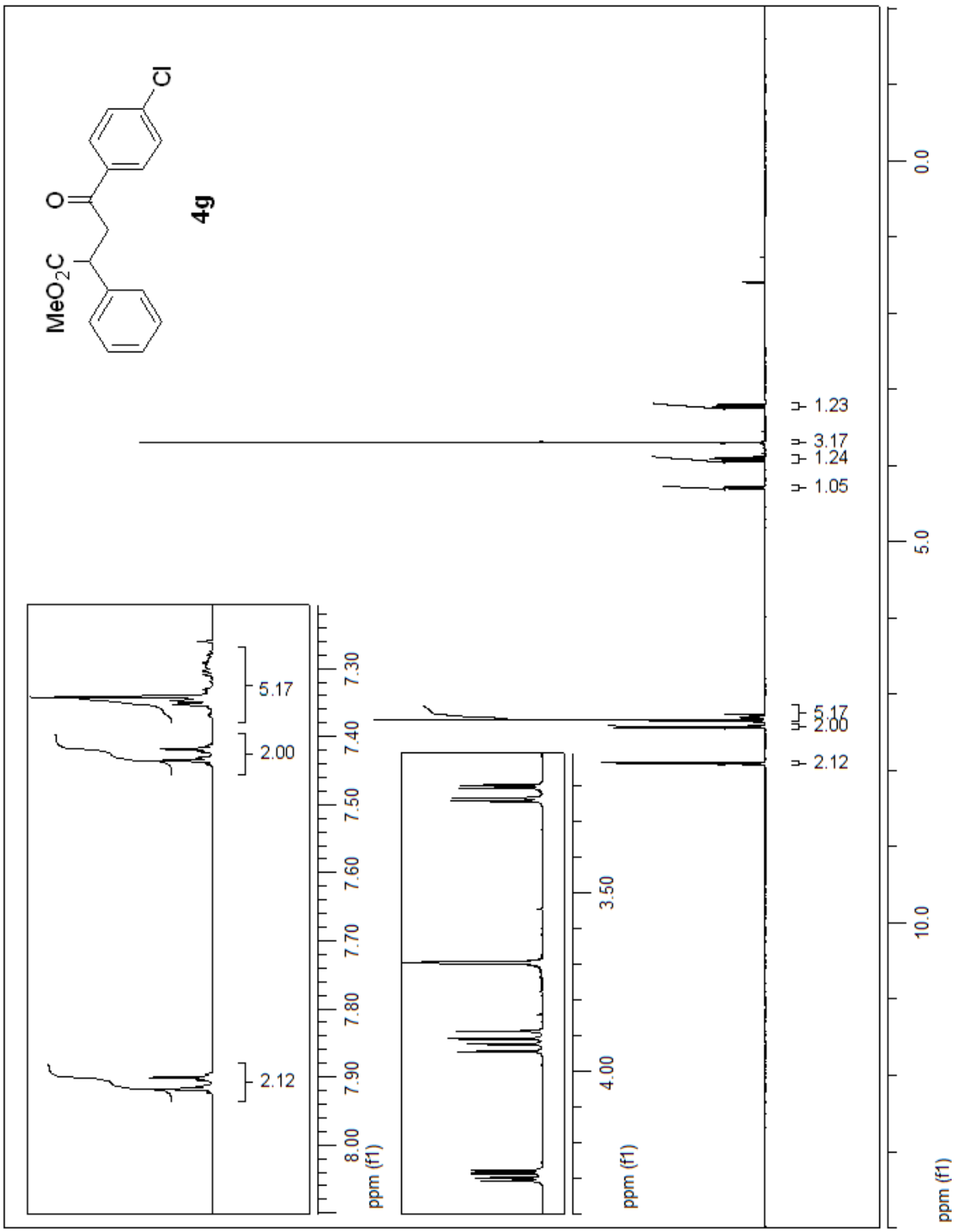

MeO
MeO2C
O
4d
1.34
3.12
1.10
2.02
2.00
1.05
2.07
7.50
7.00
4.00
3.50
5.0
0.0
10.0
ppm (f1)

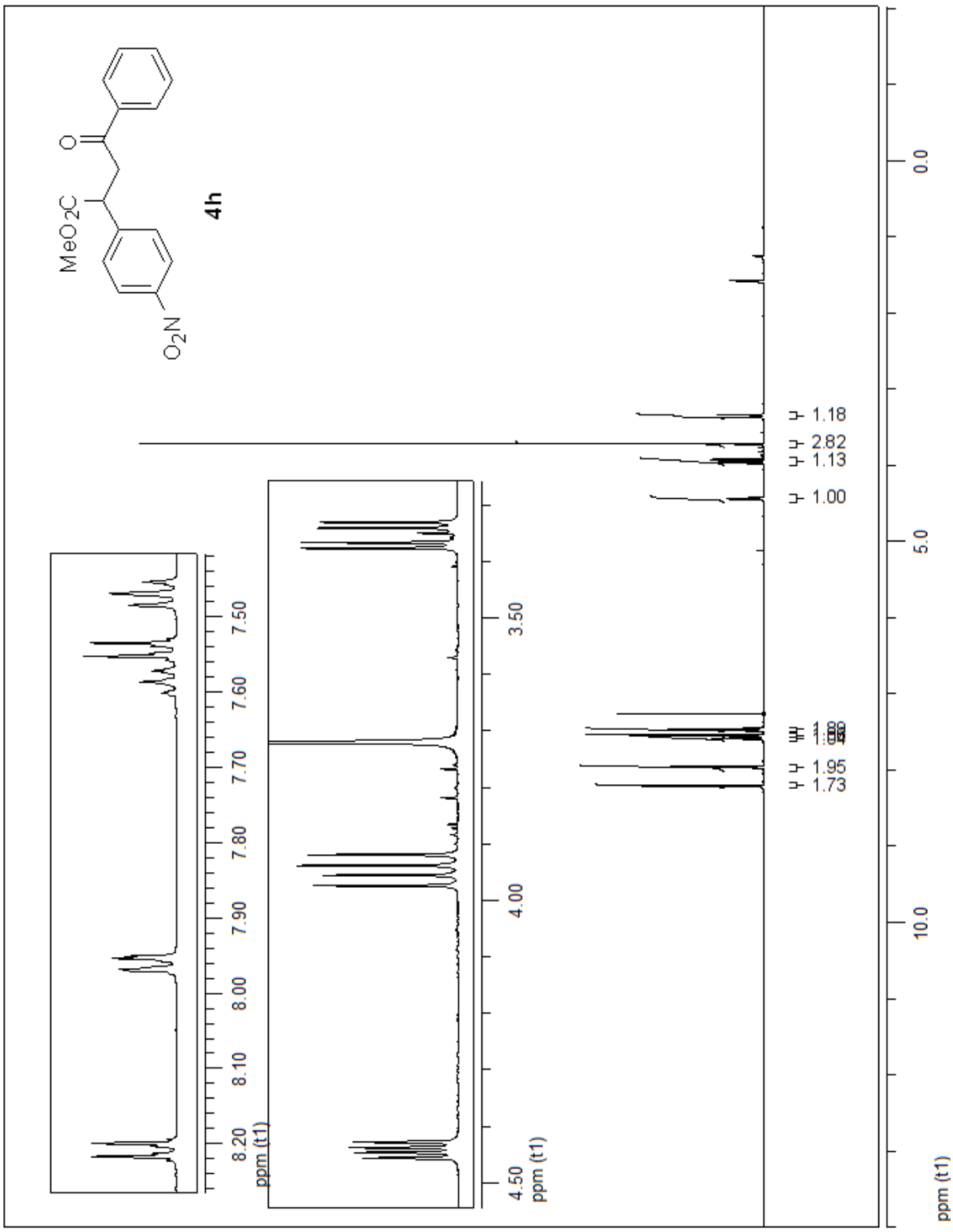

MeO
MeO2C
O
4d
136.0 135.0 134.0 133.0 132.0 131.0 130.0 129.0 128.0
ppm (f1)
200 150 100 50 0
ppm (f1)

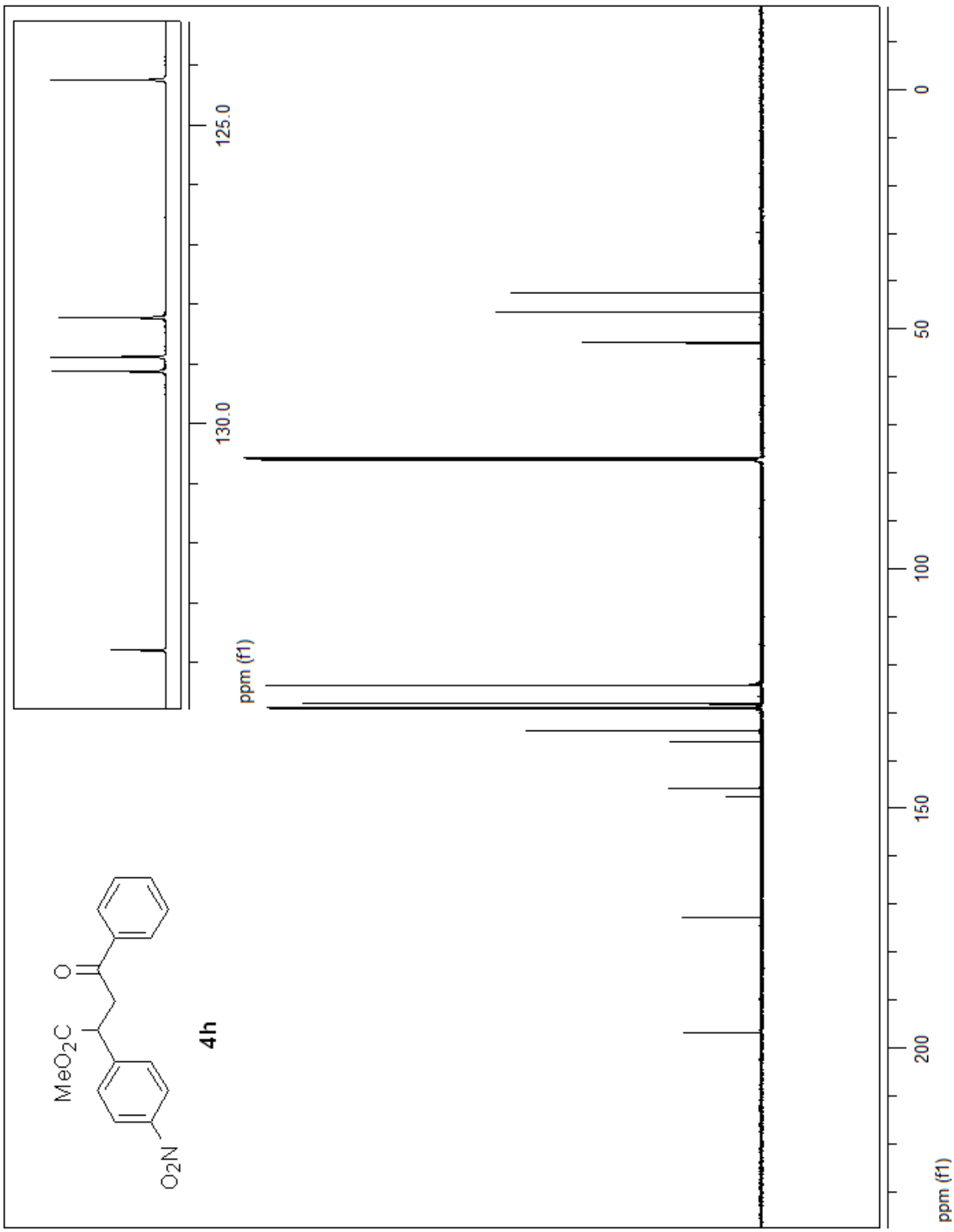

MeO2C
O
OMe
4e
1.15
2.86
1.00
1.92
1.94
7.00
7.50
3.50
4.00
5.0
10.0
0.0
ppm (f1)

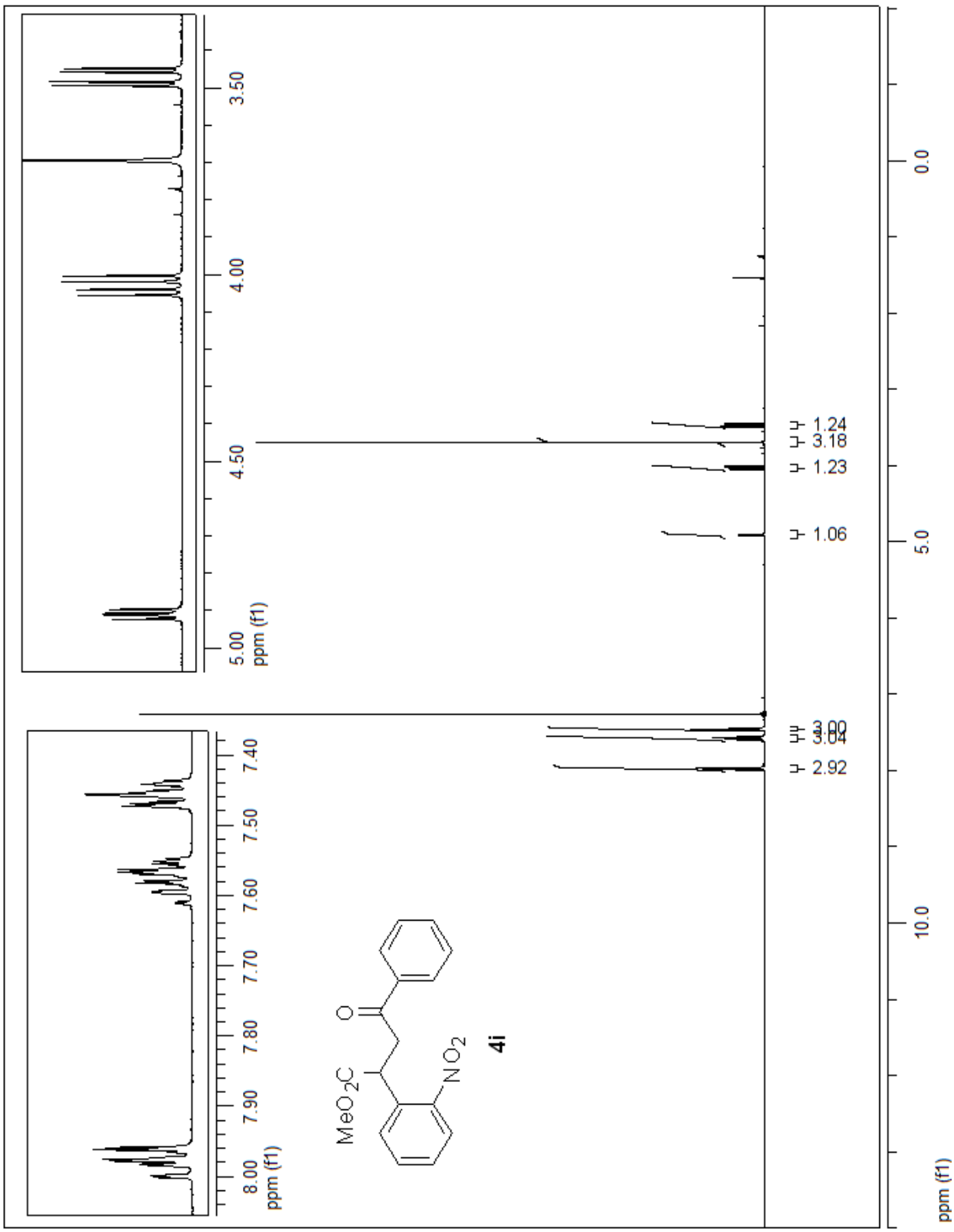

MeO2C
O
Cl
4f
8.00
7.90
7.80
7.70
7.60
7.50
7.40
7.30
7.20
ppm (f1)
4.00
3.50
ppm (f1)
1.29
3.13
1.27
1.09
3.90
1.07
1.00
2.06
10.0
5.0
0.0
ppm (f1)

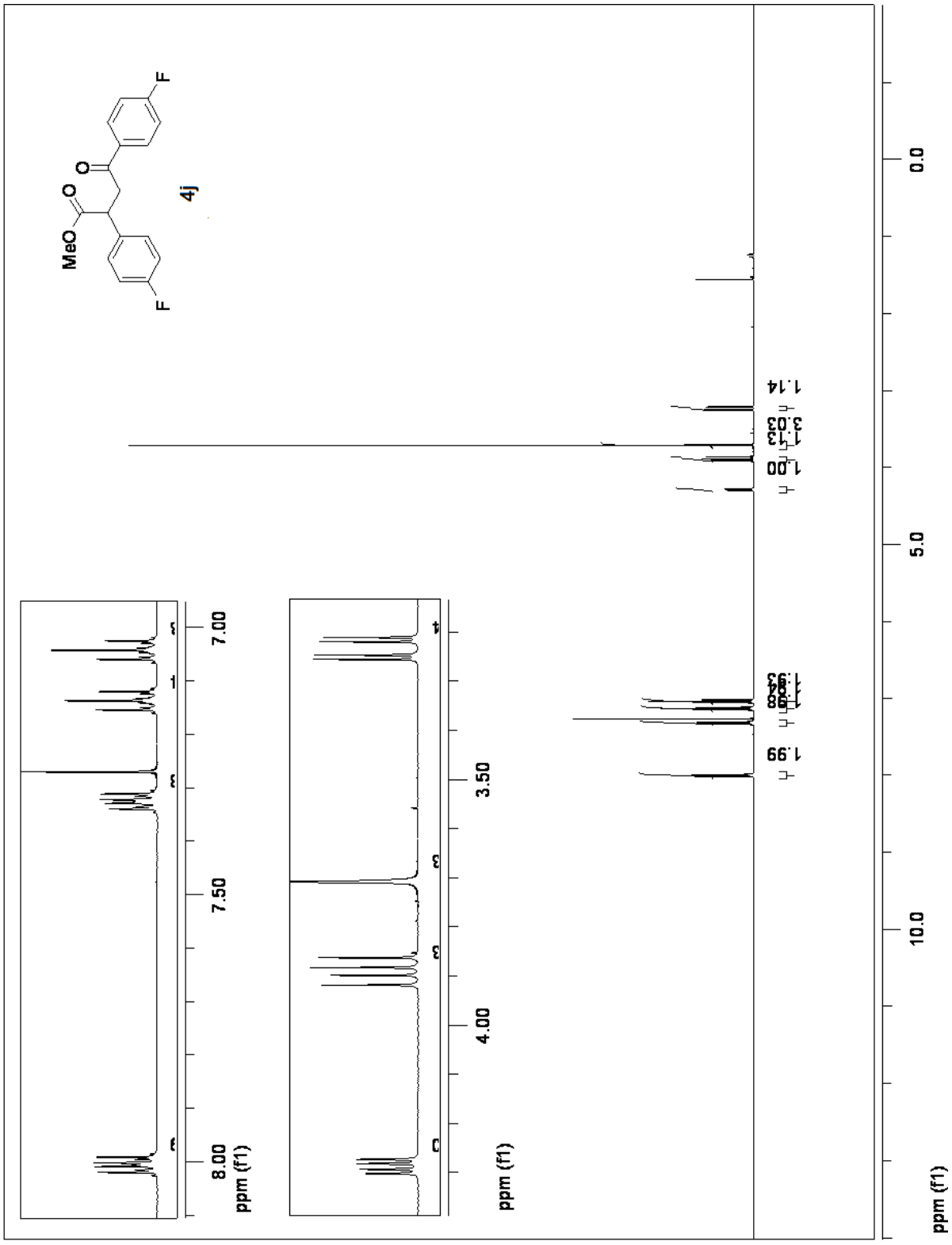

MeO2C
O
Cl
4f
129.50
129.00
128.50
ppm (f1)
137.00
136.50
136.00
135.50
135.00
134.50
134.00
ppm (f1)
200
150
100
50
0
ppm (f1)

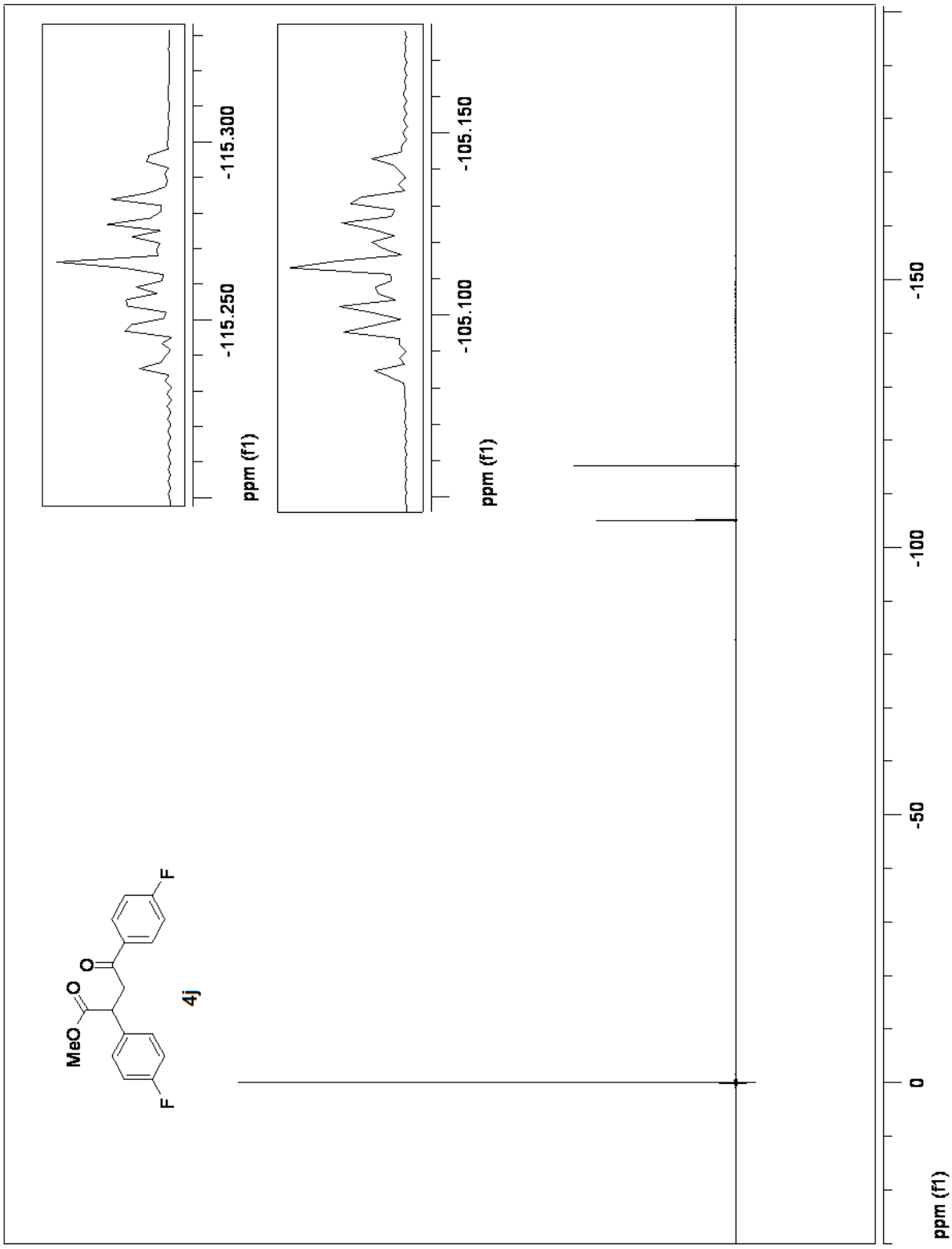

Cl
O
MeO2C
4g
1.23
3.17
1.24
1.05
5.17
2.00
2.12
ppm (f1)

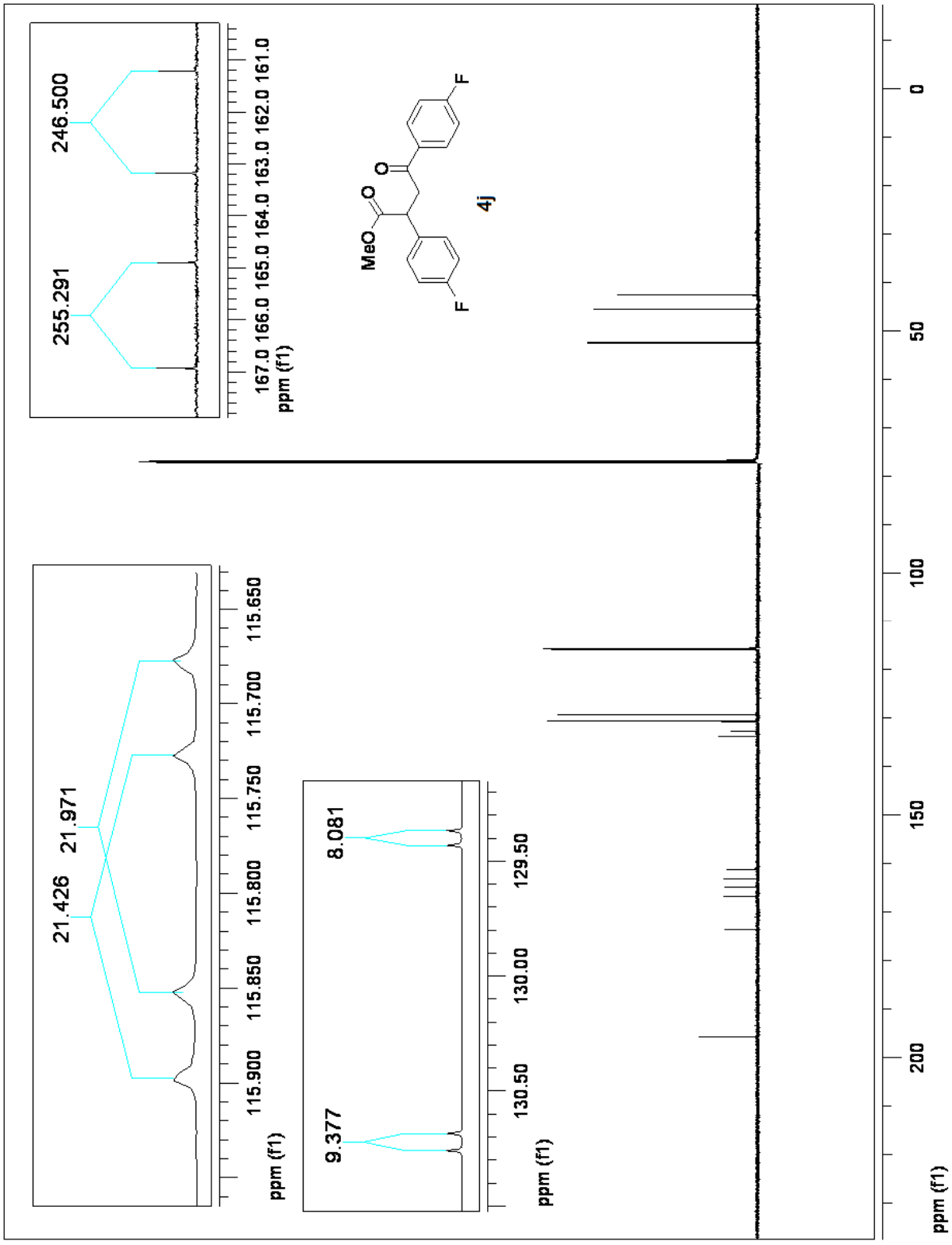

4h
MeO2C
O
O2N
1.18
2.82
1.13
1.00
1.95
1.73
8.20
8.10
8.00
7.90
7.80
7.70
7.60
7.50
4.50
4.00
3.50
10.0
5.0
0.0
ppm (t1)

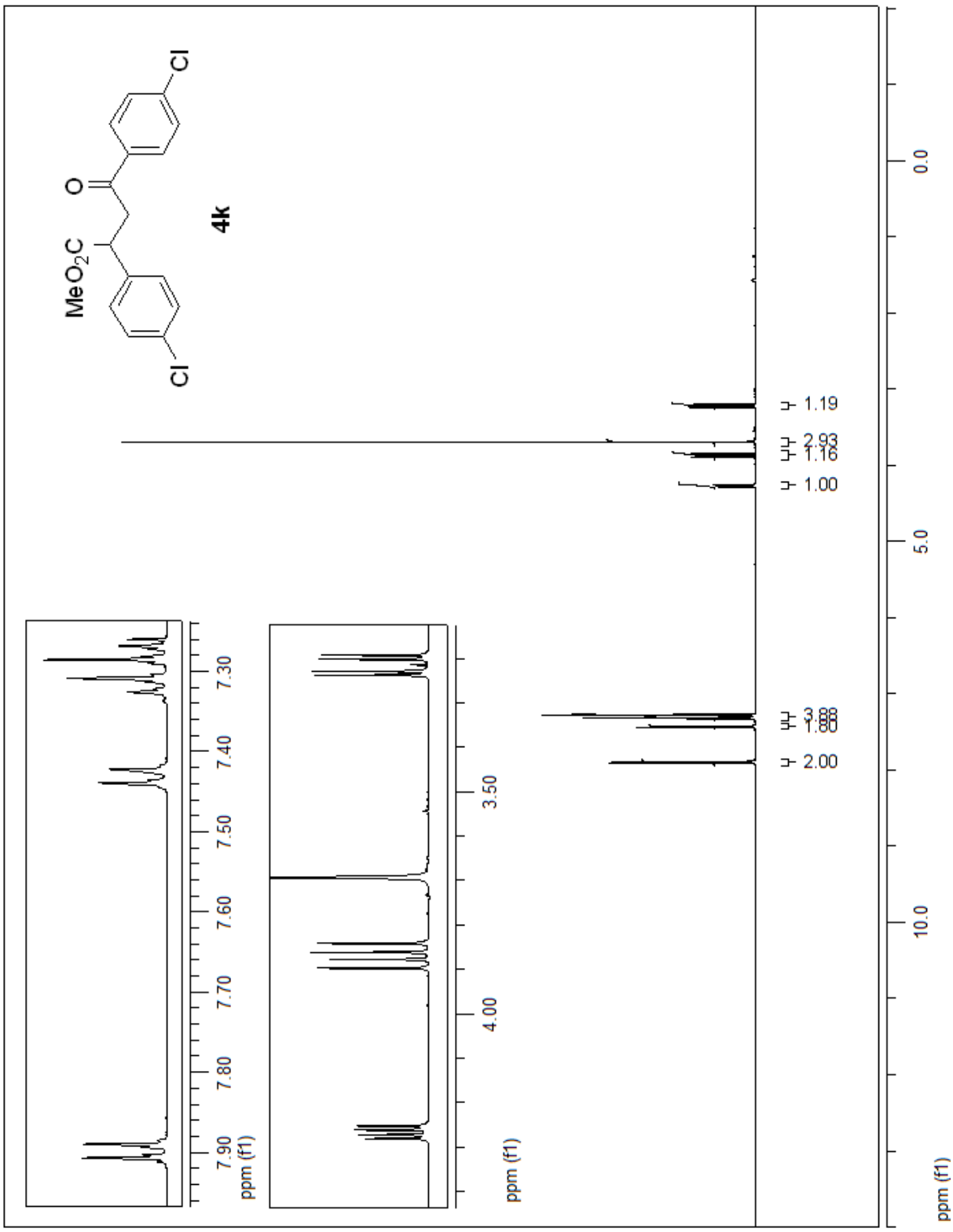

MeO2C
O
O2N
4h
125.0
130.0
ppm (f1)
0
50
100
150
200
ppm (f1)

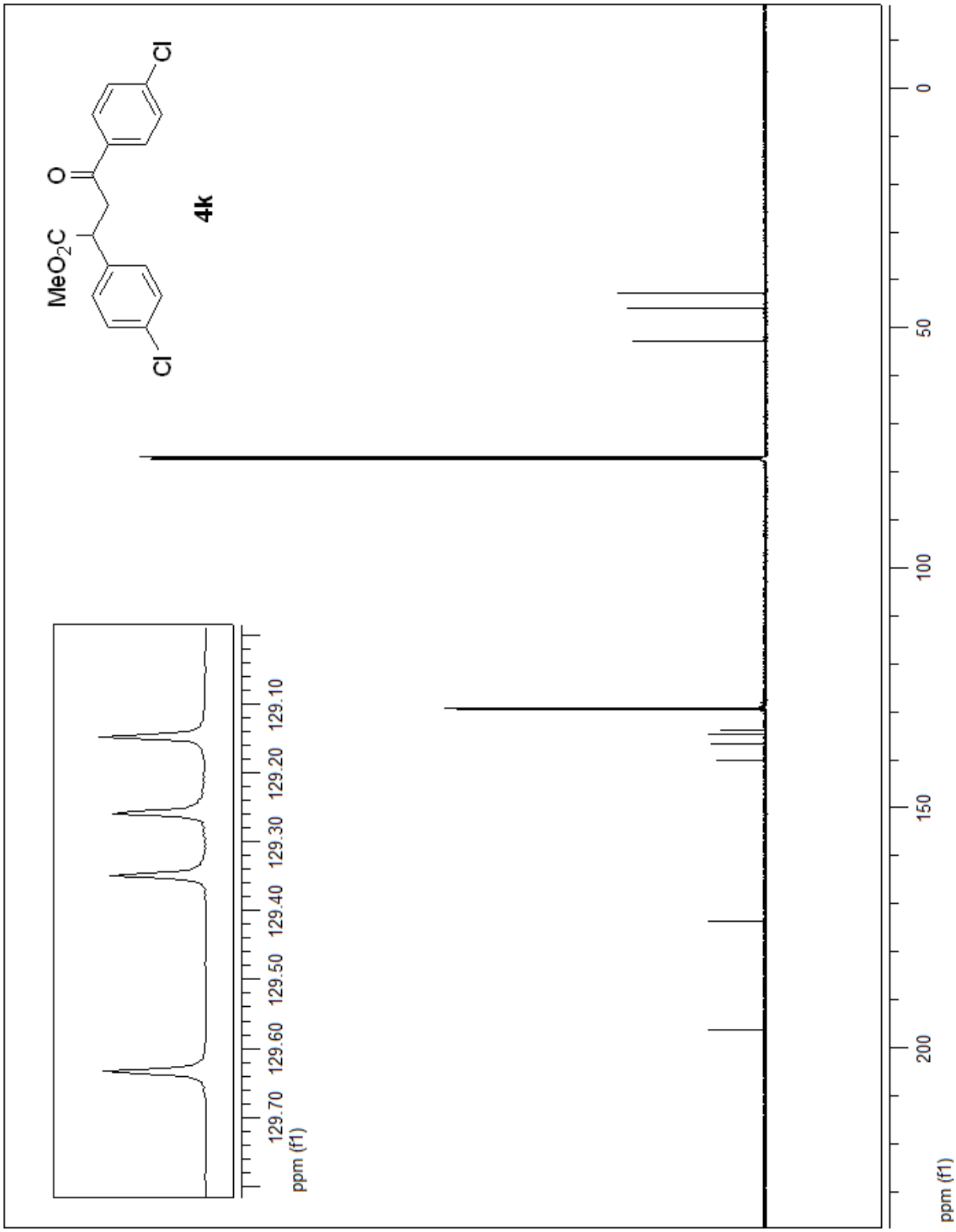

8.00
7.90
7.80
7.70
7.60
7.50
7.40
ppm (f1)
5.00
4.50
4.00
3.50
ppm (f1)
MeO2C
O
NO2
4i
1.24
3.18
1.23
1.06
3.00
3.04
2.92
10.0
5.0
0.0
ppm (f1)

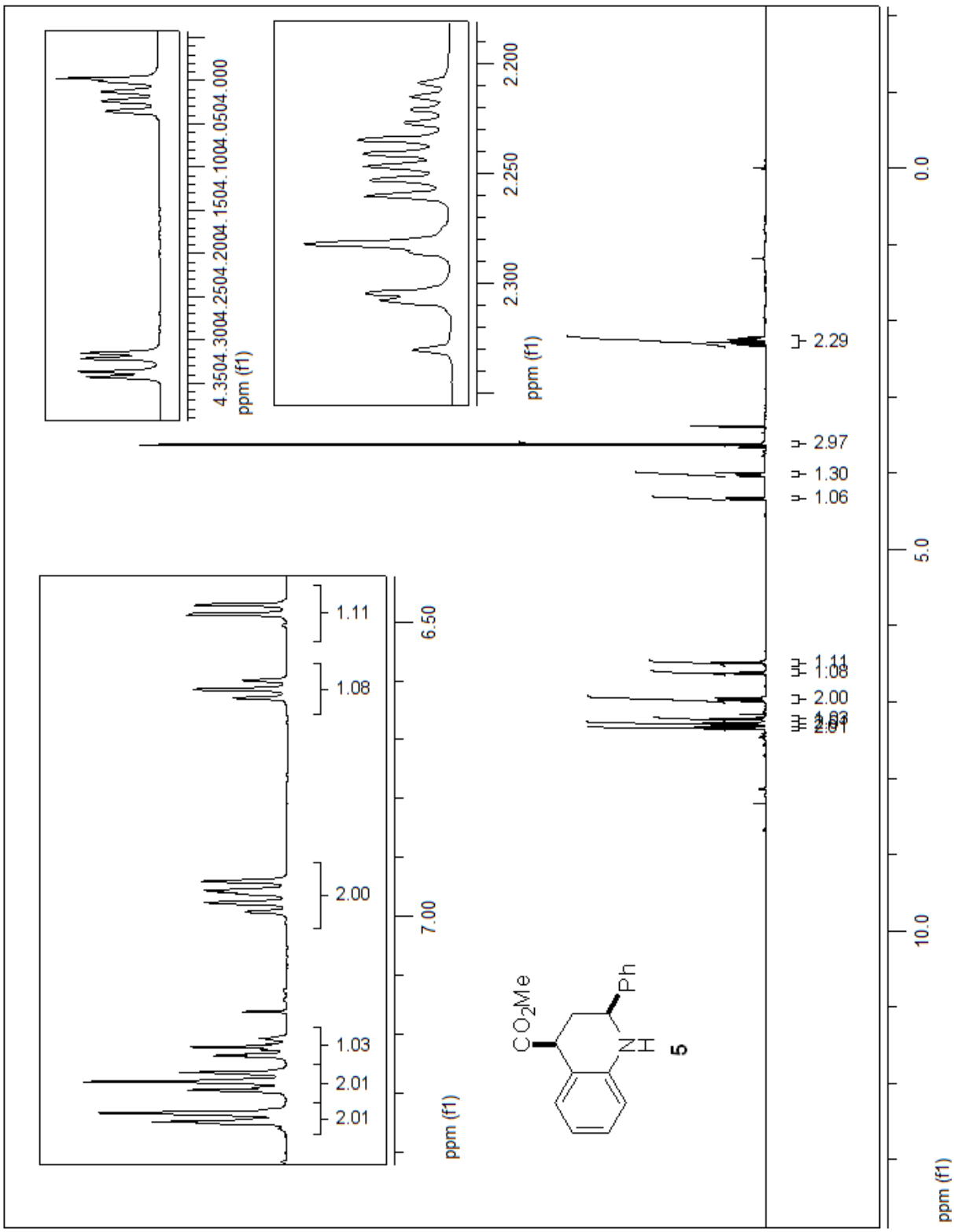

F
O
O
MeO
F
4j
1.14
3.03
1.13
1.00
1.99
8.00
7.50
7.00
4.00
3.50
ppm (f1)
10.0
5.0
0.0

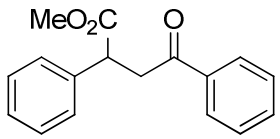

MeO
O
O
F
F
4j
-115.300
-115.250
ppm (f1)
-105.150
-105.100
ppm (f1)
0
-50
-100
-150
ppm (f1)


246.500
255.291
167.0 166.0 165.0 164.0 163.0 162.0 161.0
ppm (f1)
MeO
O
O
F
F
4j
21.426
21.971
115.900 115.850 115.800 115.750 115.700 115.650
ppm (f1)
8.081
9.377
130.50 130.00 129.50
ppm (f1)
200 150 100 50 0
ppm (f1)

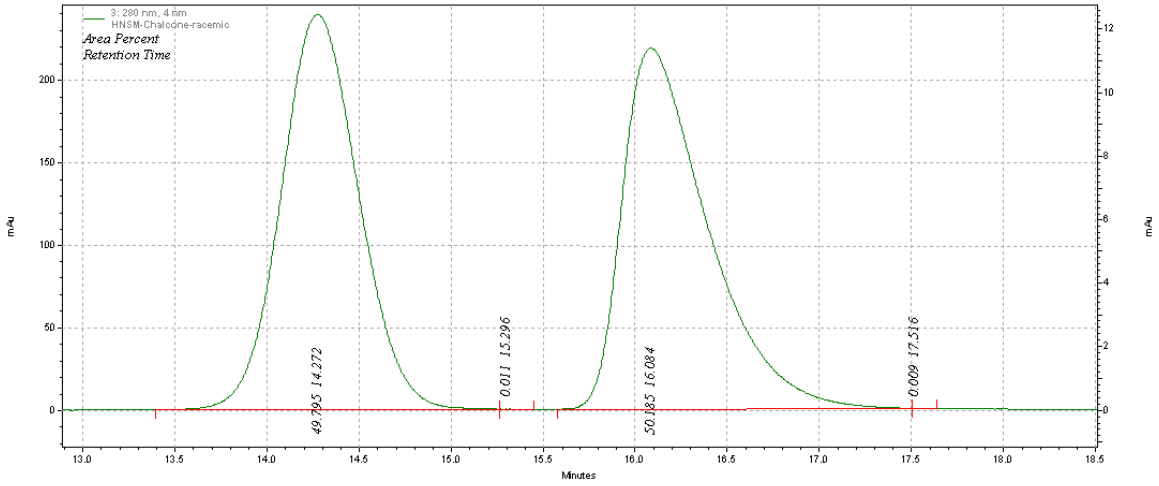

Cl
O
MeO2C
Cl
4k
1.19
2.93
1.16
1.00
3.88
1.80
2.00
7.90
7.80
7.70
7.60
7.50
7.40
7.30
4.00
3.50
10.0
5.0
0.0
ppm (f1)

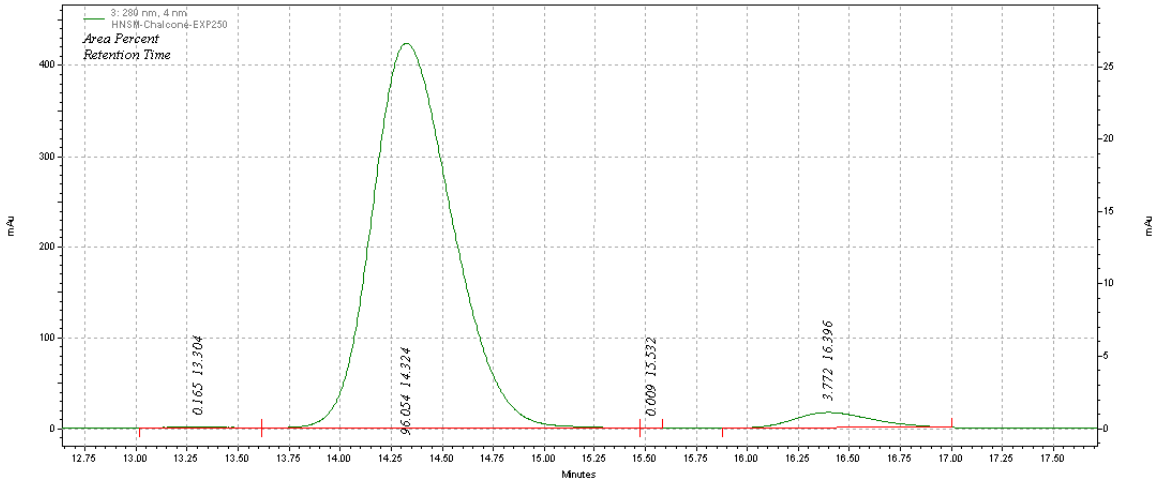

Cl
O
MeO2C
Cl
4k
129.70 129.60 129.50 129.40 129.30 129.20 129.10
ppm (f1)
200 150 100 50 0
ppm (f1)

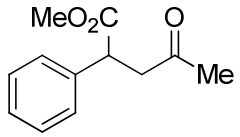

2.29
2.97
1.30
1.06
1.11
1.08
2.00
1.03
2.01
2.01
ppm (f1)
CO2Me
Ph
N
H
5


MeO2C
O

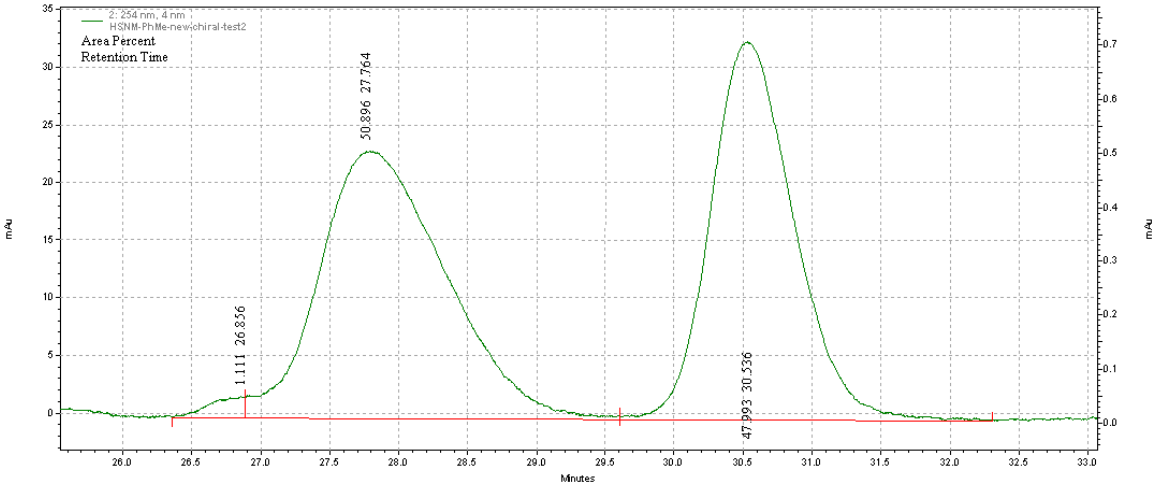
**4a**

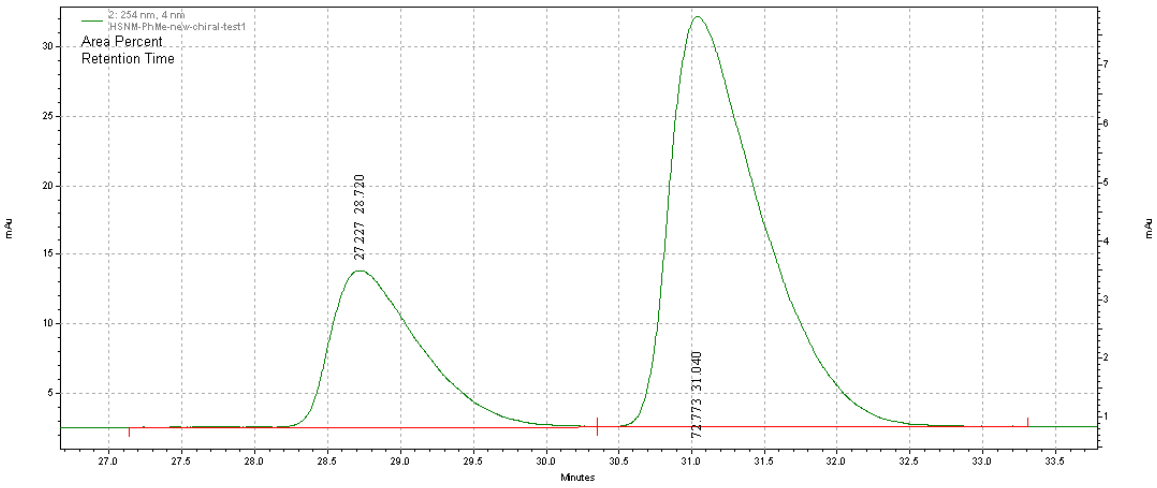

3: 280 nm, 4 nm
HNSM-Chalcone-racemic
Area Percent
Retention Time
mAu
Minutes
49.795 14.272
0.011 15.296
50.185 16.084
0.009 17.516

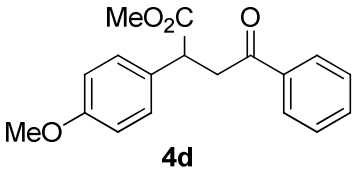

3: 280 nm, 4 nm
HNSM-Chalcone-EXP250
Area Percent
Retention Time
mAu
Minutes
0.165 13.304
96.054 14.324
0.009 15.532
3.772 16.396


MeO2C
O
Me

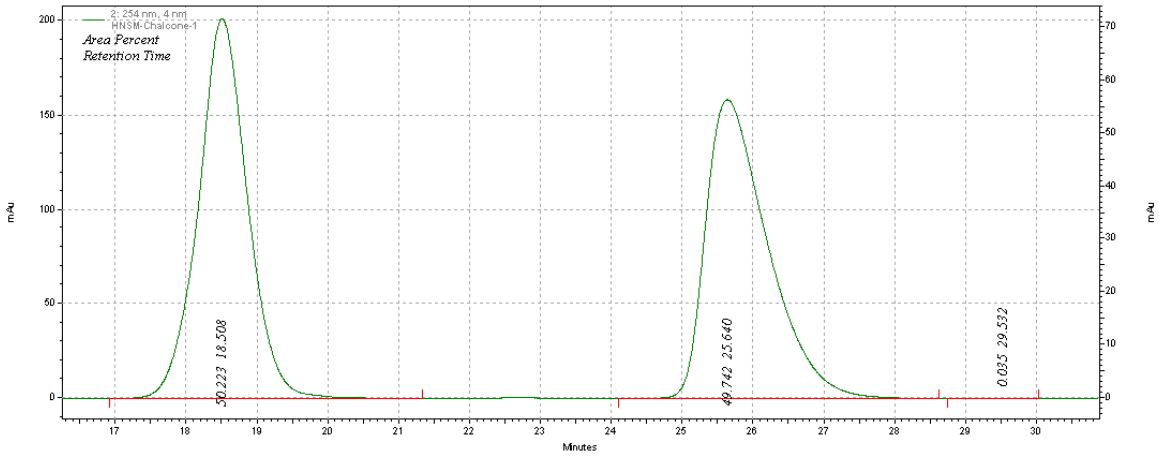
**4b**

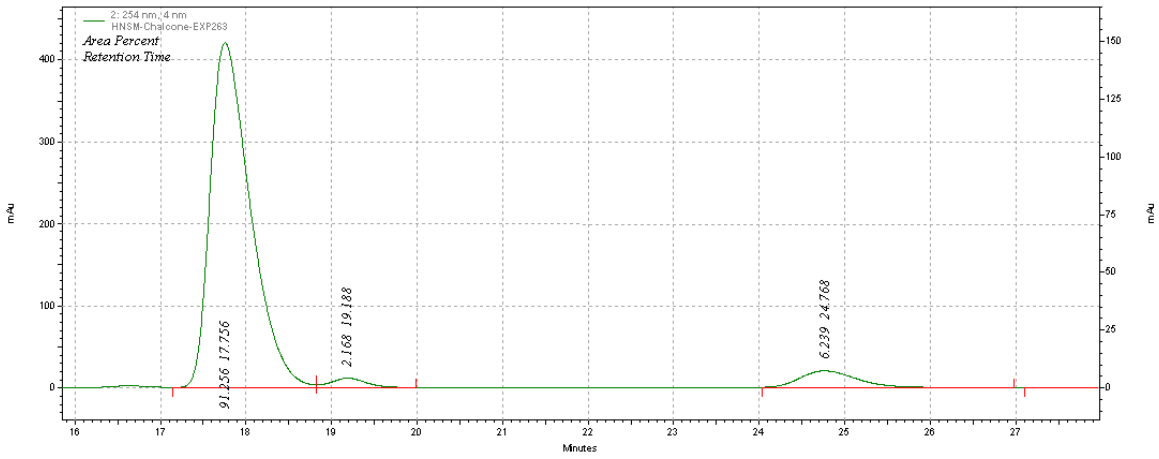

2: 254 nm, 4 nm
HSNM-PhMe-new-chiral-test2
Area Percent
Retention Time
50.896 27.764
1.111 26.856
47.993 30.536
mAu
Minutes

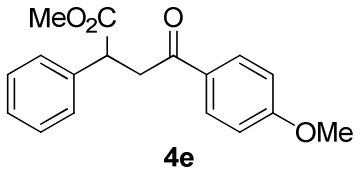

2: 254 nm, 4 nm
HSNM-PhMe-new-chiral-test1
Area Percent
Retention Time
27.227 28.720
72.773 31.040
mAu
Minutes


MeO2C
O
MeO

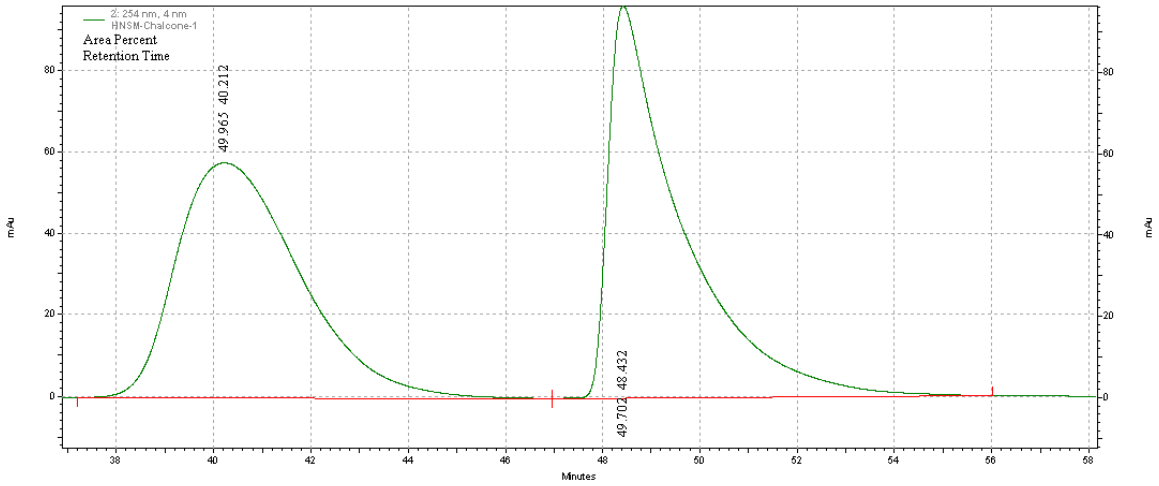
**4d**

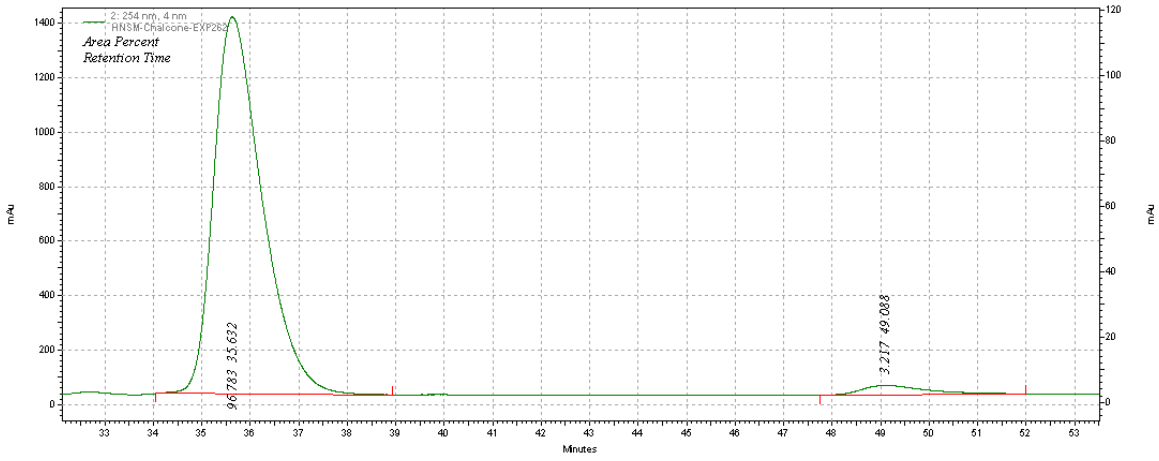

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
50.223 18.508
49.742 25.640
0.035 29.532
mAu
Minutes

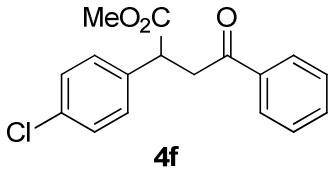

2: 254 nm, 4 nm
HNSM-Chalcone-EXP263
Area Percent
Retention Time
91.256 17.756
2.168 19.188
6.239 24.768
mAu
Minutes


MeO2C
O
OMe

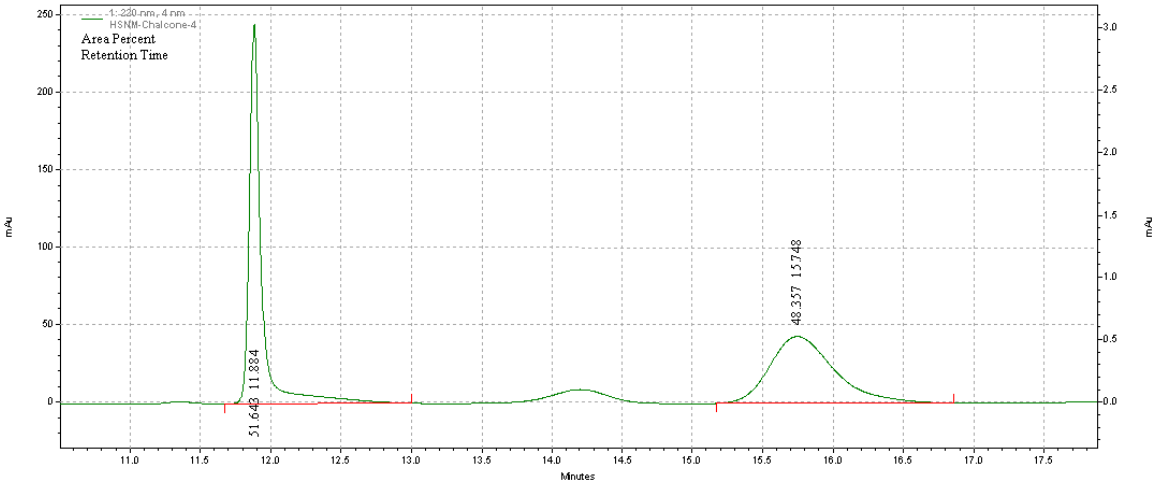
**4e**

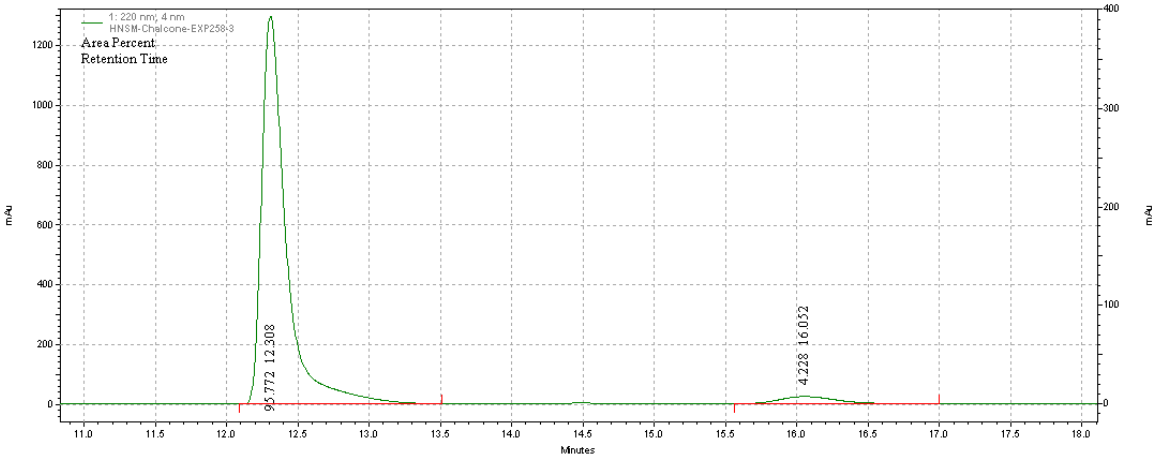

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
49.965 40.212
49.704 48.432
mAu
Minutes

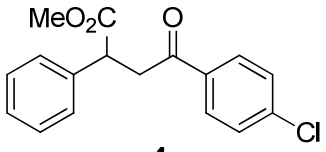

2: 254 nm, 4 nm
HNSM-Chalcone-EXP262
Area Percent
Retention Time
96.783 35.632
3.217 49.088
mAu
Minutes


MeO2C
O
Cl

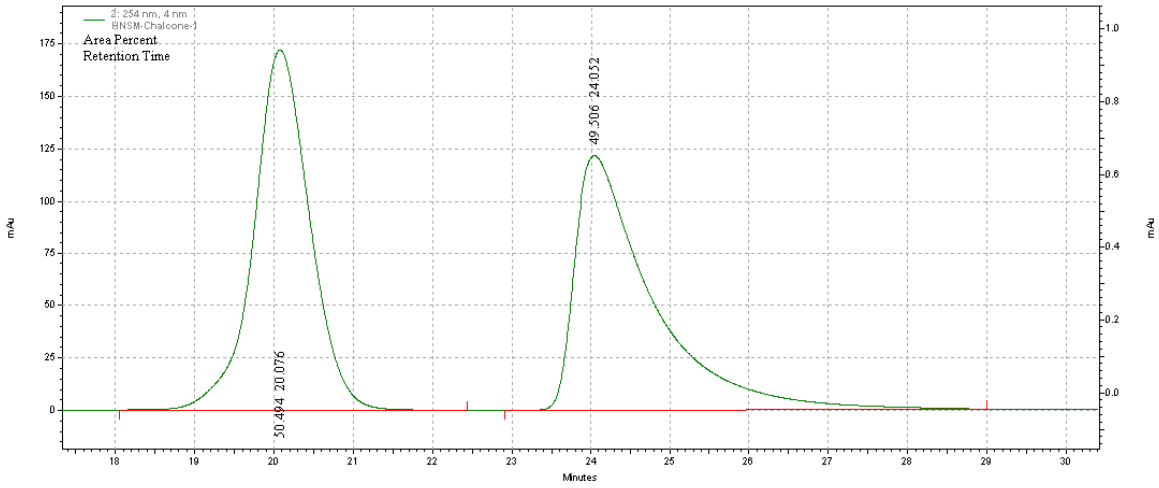
**4f**

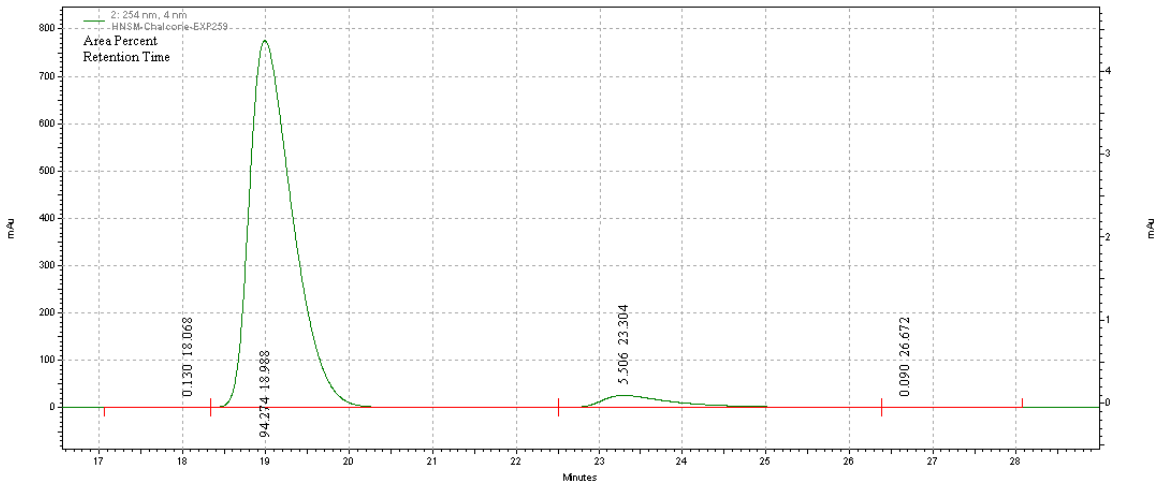

1: 220 nm, 4 nm
HSNM-Chalcone-4
Area Percent
Retention Time
51.643 11.884
48.357 15.748
mAu
Minutes

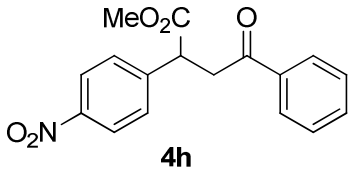

1: 220 nm, 4 nm
HNSM-Chalcone-EXP258-3
Area Percent
Retention Time
95.772 12.308
4.228 16.052
mAu
Minutes


MeO2C
O
Cl

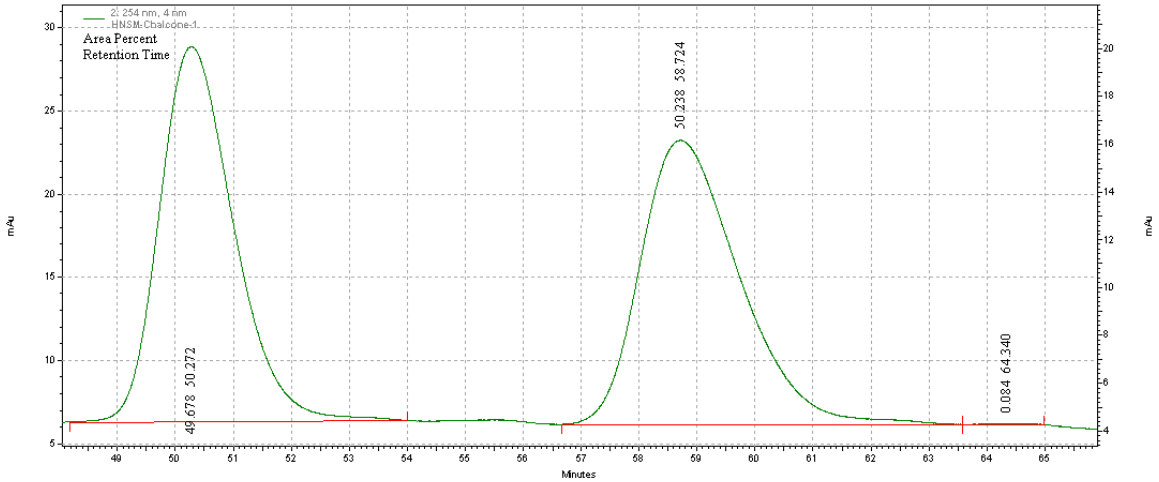
**4g**

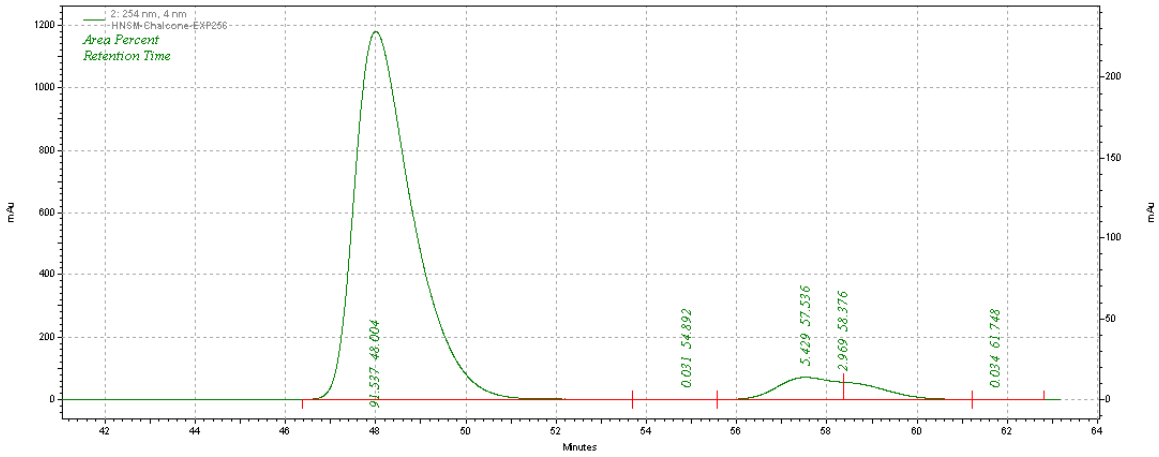

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
50.494 20.076
49.506 24.052
mAu
Minutes

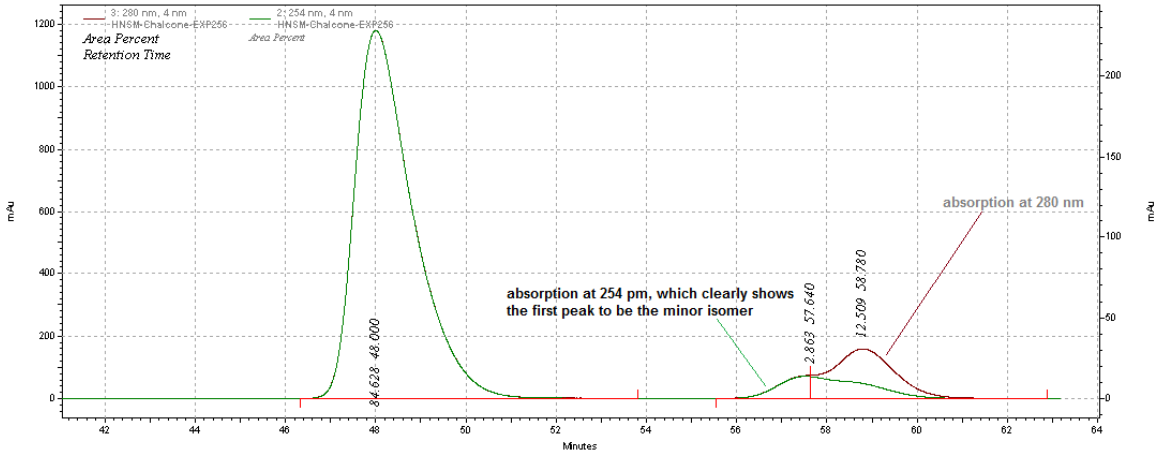

2: 254 nm, 4 nm
HNSM-Chalcone-EXP259
Area Percent
Retention Time
0.130 18.068
94.274 18.988
5.506 23.304
0.090 26.672
mAu
Minutes

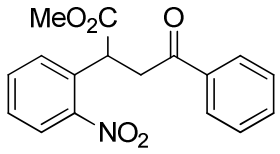

MeO2C
O
O2N

**4h**

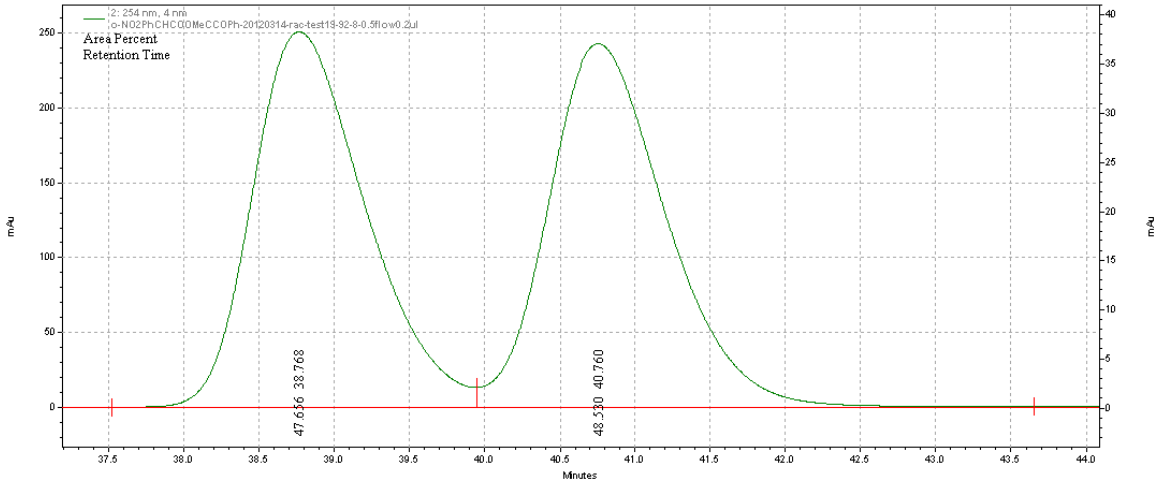

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
49.678 50.272
50.238 58.724
0.084 64.340
mAu
Minutes

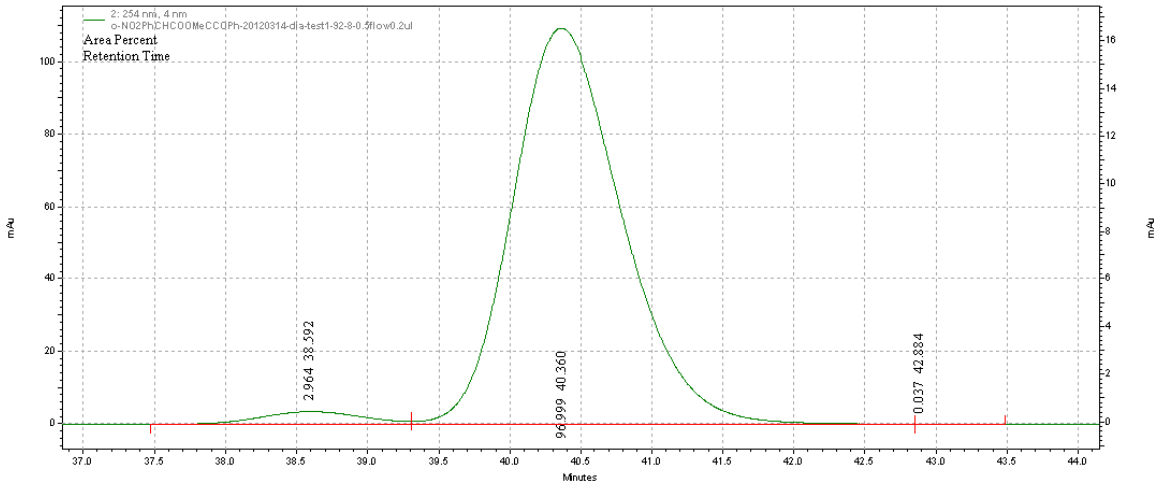

2: 254 nm, 4 nm
HNSM-Chalcone-EXP256
Area Percent
Retention Time
91.537 48.004
0.031 54.892
5.429 57.536
2.969 58.376
0.034 61.748
mAu
Minutes

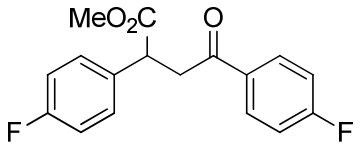

3: 280 nm, 4 nm
HNSM-Chalcone-EXP256
2: 254 nm, 4 nm
HNSM-Chalcone-EXP256
Area Percent
Retention Time
84.628 48.000
2.863 57.640
12.509 58.780
absorption at 280 nm
absorption at 254 pm, which clearly shows
the first peak to be the minor isomer
mAu
Minutes


MeO2C
O
NO2

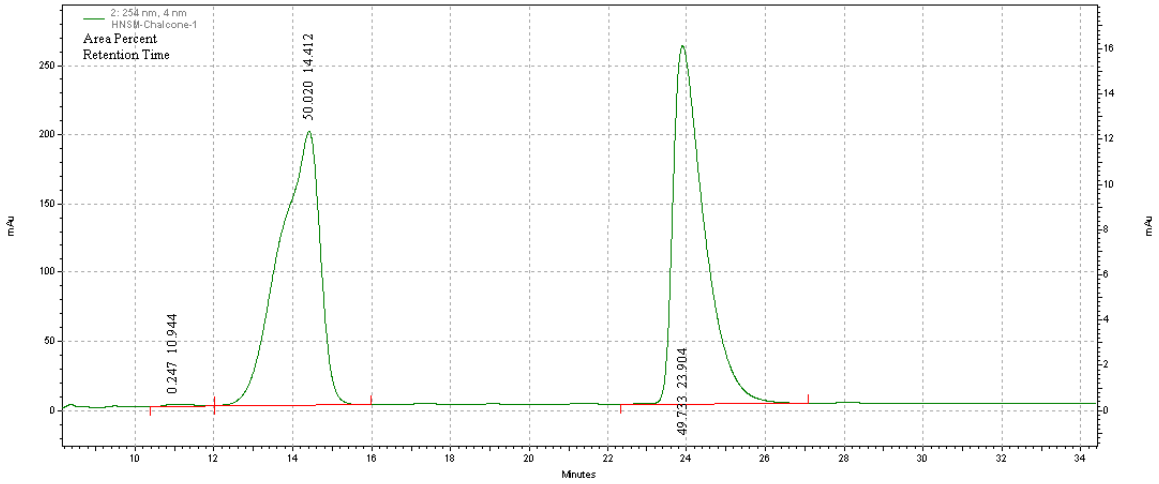
**4i**

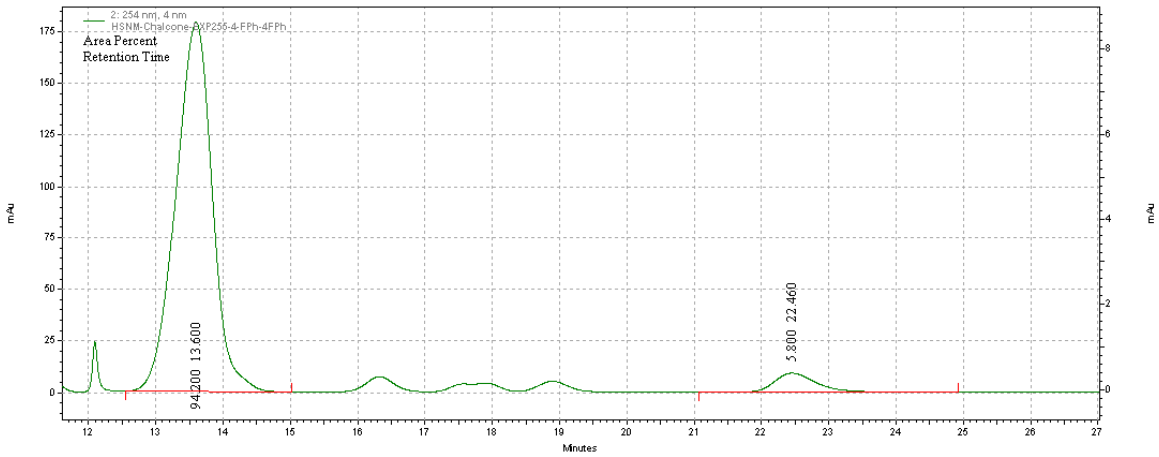

2: 254 nm, 4 nm
o-NO2PhCHCOOMeCCOPh-20120314-rac-test13-92-8-0.5flow0.2ul
Area Percent
Retention Time
47.636 38.768
48.530 40.760
mAu
Minutes

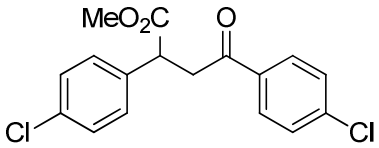

2: 254 nm, 4 nm
o-NO2PhCHCOOMeCCOPh-20120314-dja-test1-92-8-0.5flow0.2ul
Area Percent
Retention Time
2.964 38.592
96.999 40.360
0.037 42.884
mAu
Minutes


MeO2C
O
F
F

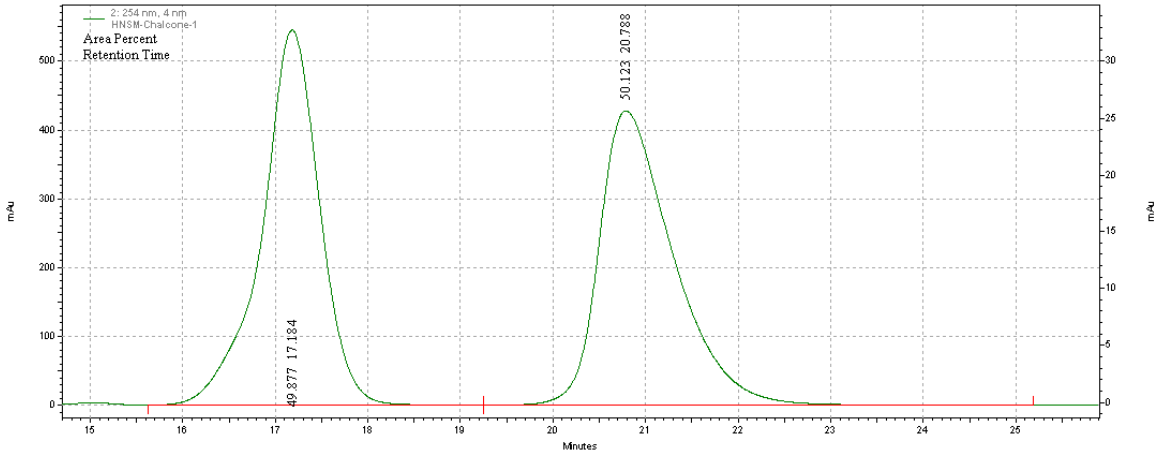
**4j**

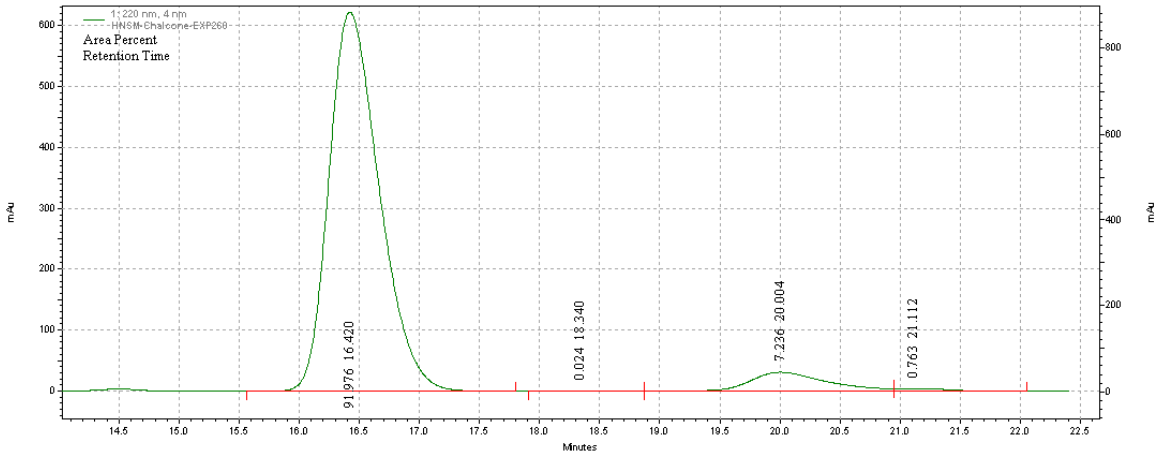

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
50.020 14.412
0.247 10.944
49.733 23.904
mAu
Minutes

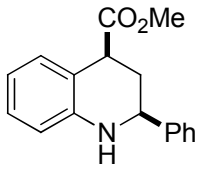

2: 254 nm, 4 nm
HSNM-Chalcone-PXP256-4-FPh-4FPh
Area Percent
Retention Time
94.200 13.600
5.800 22.460
mAu
Minutes


MeO2C
O
Cl
Cl

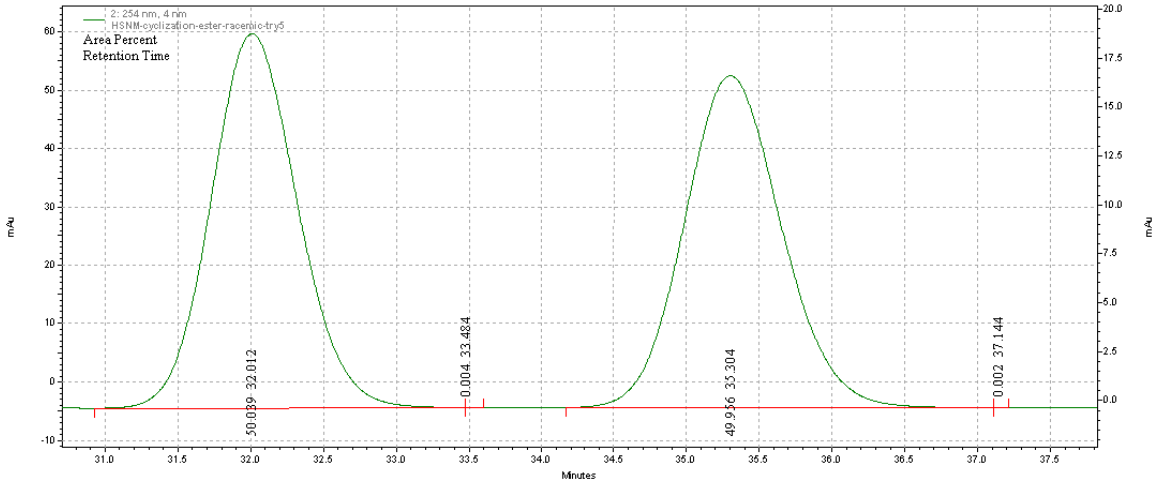
**4k**

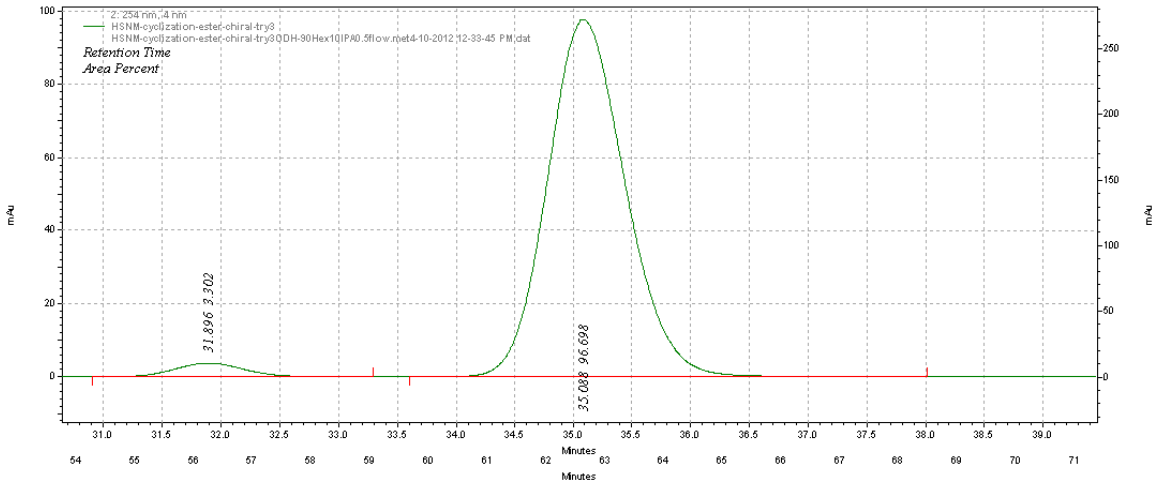

2: 254 nm, 4 nm
HNSM-Chalcone-1
Area Percent
Retention Time
49.877 17.184
50.123 20.788
mAu
Minutes

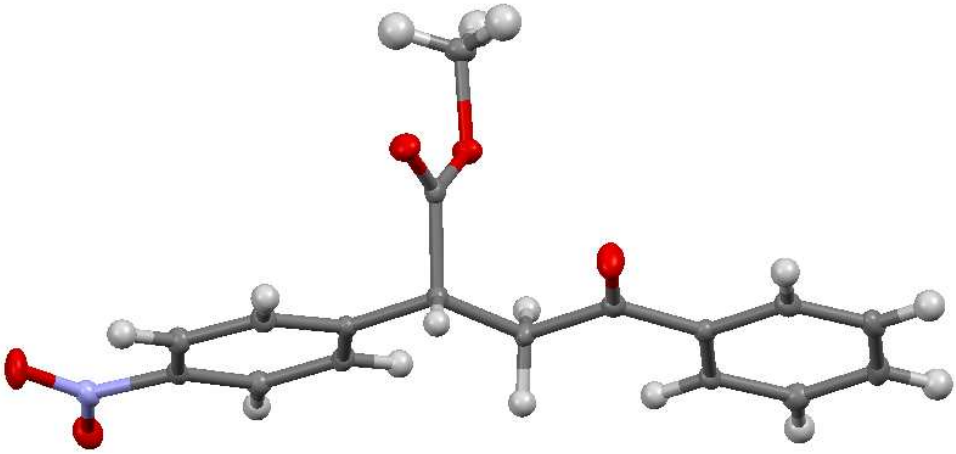

1: 220 nm, 4 nm
HNSM-Chalcone-EXP268
Area Percent
Retention Time
91.976 16.420
0.024 18.340
7.236 20.004
0.763 21.112
mAu
Minutes


CO2Me
N
H
Ph

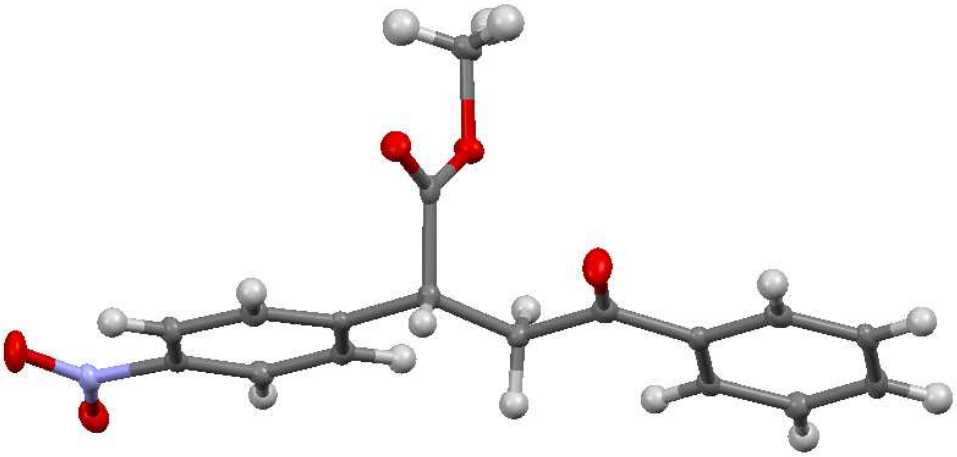
**5**


2: 254 nm, 4 nm
HSNM-cyclization-ester-racemic-try5
Area Percent
Retention Time
50.089 32.012
0.004 33.484
49.956 35.304
0.002 37.144
mAu
Minutes


2: 254 nm, 4 nm
HSNM-cyclization-ester-chiral-try3
HSNM-cyclization-ester-chiral-try3 QDH-90Hex10IPA0.5flow.met4-10-2012 12-33-45 PM.dat
Retention Time
Area Percent
3.302 31.896
96.698 35.088
mAu
Minutes



Crystal Structure of **4h** derived from Mo data



Crystal Structure of **4h** derived from Cu data

**CCDC deposition numbers: CCDC 885740, CCDD 885741**

**Critical Parameters**

$C_{17}H_{15}NO_5$:
Flack x: 0.2(2)
Hooft y: 0.2 (0.1)
Bayesian Statistics: P2(true) 1.000

Table 1. Crystal data and structure refinement for C17H15NO5. (**Cu data**)

| | | |
|---|---|---|
| Identification code | cu_fang053112_0m | |
| Empirical formula | C17 H15 N O5 | |
| Formula weight | 313.30 | |
| Temperature | 100(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Monoclinic | |
| Space group | P 21 | |
| Unit cell dimensions | a = 7.41820(10) Å | α= 90°. |
| | b = 12.6302(2) Å | β= 104.4550(10)°. |
| | c = 7.90890(10) Å | γ = 90°. |
| Volume | 717.554(17) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.450 $Mg/m^3$ | |
| Absorption coefficient | 0.901 $mm^{-1}$ | |
| F(000) | 328 | |
| Crystal size | 0.46 x 0.16 x 0.14 $mm^3$ | |
| Theta range for data collection | 5.78 to 50.42°. | |
| Index ranges | -7<=h<=6, -12<=k<=12, -7<=l<=7 | |
| Reflections collected | 3689 | |
| Independent reflections | 1443 [R(int) = 0.0256] | |
| Completeness to theta = 50.42° | 97.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.99 and 0.85 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 1443 / 1 / 210 | |
| Goodness-of-fit on $F^2$ | 1.098 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0260, wR2 = 0.0650 | |
| R indices (all data) | R1 = 0.0263, wR2 = 0.0652 | |
| Absolute structure parameter | 0.2(2) | |
| Extinction coefficient | 0.0069(9) | |
| Largest diff. peak and hole | 0.152 and -0.150 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for C17H15NO5. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 5557(3) | 6691(2) | 4569(3) | 16(1) |
| C(2) | 5028(3) | 7206(2) | 2980(3) | 18(1) |
| C(3) | 5447(3) | 8269(2) | 2854(3) | 18(1) |
| C(4) | 6401(3) | 8813(2) | 4321(3) | 17(1) |
| C(5) | 6930(3) | 8299(2) | 5905(3) | 15(1) |
| C(6) | 6518(3) | 7234(2) | 6059(3) | 13(1) |
| C(7) | 7132(3) | 6650(2) | 7738(3) | 16(1) |
| C(8) | 8182(3) | 7240(2) | 9340(3) | 15(1) |
| C(9) | 8838(3) | 6532(2) | 10926(3) | 14(1) |
| C(10) | 9875(3) | 7117(2) | 12576(3) | 13(1) |
| C(11) | 10634(3) | 8120(2) | 12534(3) | 15(1) |
| C(12) | 11595(3) | 8618(2) | 14055(3) | 15(1) |
| C(13) | 11813(3) | 8089(2) | 15614(3) | 13(1) |
| C(14) | 11087(3) | 7087(2) | 15702(3) | 16(1) |
| C(15) | 10104(3) | 6611(2) | 14178(3) | 15(1) |
| C(16) | 10130(3) | 5648(2) | 10623(3) | 16(1) |
| C(17) | 12734(3) | 5258(2) | 9499(3) | 21(1) |
| N(1) | 12915(2) | 8580(2) | 17223(2) | 18(1) |
| O(1) | 6826(2) | 5700(1) | 7822(2) | 24(1) |
| O(2) | 13361(2) | 9517(1) | 17168(2) | 23(1) |
| O(3) | 13376(2) | 8033(1) | 18536(2) | 28(1) |
| O(4) | 11325(2) | 6006(1) | 9721(2) | 19(1) |
| O(5) | 10159(2) | 4761(1) | 11187(2) | 21(1) |

Table 3. Bond lengths [Å] and angles [°] for C17H15NO5.

| | |
|---|---|
| C(1)-C(2) | 1.382(3) |
| C(1)-C(6) | 1.395(3) |
| C(1)-H(1) | 0.9500 |
| C(2)-C(3) | 1.388(3) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.382(3) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.379(3) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.392(3) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.487(3) |
| C(7)-O(1) | 1.226(3) |
| C(7)-C(8) | 1.508(3) |
| C(8)-C(9) | 1.518(3) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-C(16) | 1.528(3) |
| C(9)-C(10) | 1.530(3) |
| C(9)-H(9) | 1.0000 |
| C(10)-C(11) | 1.390(3) |
| C(10)-C(15) | 1.391(3) |
| C(11)-C(12) | 1.387(3) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.376(3) |
| C(12)-H(12) | 0.9500 |
| C(13)-C(14) | 1.384(3) |
| C(13)-N(1) | 1.467(3) |
| C(14)-C(15) | 1.381(3) |
| C(14)-H(14) | 0.9500 |
| C(15)-H(15) | 0.9500 |
| C(16)-O(5) | 1.205(3) |
| C(16)-O(4) | 1.347(3) |
| C(17)-O(4) | 1.452(3) |

| | |
|---|---|
| C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 |
| N(1)-O(3) | 1.223(2) |
| N(1)-O(2) | 1.232(2) |

| | |
|---|---|
| C(2)-C(1)-C(6) | 120.5(2) |
| C(2)-C(1)-H(1) | 119.8 |
| C(6)-C(1)-H(1) | 119.8 |
| C(1)-C(2)-C(3) | 120.1(2) |
| C(1)-C(2)-H(2) | 119.9 |
| C(3)-C(2)-H(2) | 119.9 |
| C(4)-C(3)-C(2) | 119.9(2) |
| C(4)-C(3)-H(3) | 120.1 |
| C(2)-C(3)-H(3) | 120.1 |
| C(5)-C(4)-C(3) | 119.9(2) |
| C(5)-C(4)-H(4) | 120.0 |
| C(3)-C(4)-H(4) | 120.0 |
| C(4)-C(5)-C(6) | 121.0(2) |
| C(4)-C(5)-H(5) | 119.5 |
| C(6)-C(5)-H(5) | 119.5 |
| C(5)-C(6)-C(1) | 118.5(2) |
| C(5)-C(6)-C(7) | 122.13(19) |
| C(1)-C(6)-C(7) | 119.3(2) |
| O(1)-C(7)-C(6) | 120.9(2) |
| O(1)-C(7)-C(8) | 120.1(2) |
| C(6)-C(7)-C(8) | 119.0(2) |
| C(7)-C(8)-C(9) | 113.28(19) |
| C(7)-C(8)-H(8A) | 108.9 |
| C(9)-C(8)-H(8A) | 108.9 |
| C(7)-C(8)-H(8B) | 108.9 |
| C(9)-C(8)-H(8B) | 108.9 |
| H(8A)-C(8)-H(8B) | 107.7 |
| C(8)-C(9)-C(16) | 112.60(18) |
| C(8)-C(9)-C(10) | 114.18(17) |
| C(16)-C(9)-C(10) | 106.52(16) |

| | |
|---|---|
| C(8)-C(9)-H(9) | 107.8 |
| C(16)-C(9)-H(9) | 107.8 |
| C(10)-C(9)-H(9) | 107.8 |
| C(11)-C(10)-C(15) | 118.83(18) |
| C(11)-C(10)-C(9) | 122.70(18) |
| C(15)-C(10)-C(9) | 118.46(19) |
| C(12)-C(11)-C(10) | 121.05(19) |
| C(12)-C(11)-H(11) | 119.5 |
| C(10)-C(11)-H(11) | 119.5 |
| C(13)-C(12)-C(11) | 118.5(2) |
| C(13)-C(12)-H(12) | 120.7 |
| C(11)-C(12)-H(12) | 120.7 |
| C(12)-C(13)-C(14) | 121.94(19) |
| C(12)-C(13)-N(1) | 119.1(2) |
| C(14)-C(13)-N(1) | 118.86(19) |
| C(15)-C(14)-C(13) | 118.77(19) |
| C(15)-C(14)-H(14) | 120.6 |
| C(13)-C(14)-H(14) | 120.6 |
| C(14)-C(15)-C(10) | 120.9(2) |
| C(14)-C(15)-H(15) | 119.6 |
| C(10)-C(15)-H(15) | 119.6 |
| O(5)-C(16)-O(4) | 123.7(2) |
| O(5)-C(16)-C(9) | 125.1(2) |
| O(4)-C(16)-C(9) | 111.09(19) |
| O(4)-C(17)-H(17A) | 109.5 |
| O(4)-C(17)-H(17B) | 109.5 |
| H(17A)-C(17)-H(17B) | 109.5 |
| O(4)-C(17)-H(17C) | 109.5 |
| H(17A)-C(17)-H(17C) | 109.5 |
| H(17B)-C(17)-H(17C) | 109.5 |
| O(3)-N(1)-O(2) | 123.49(18) |
| O(3)-N(1)-C(13) | 118.31(19) |
| O(2)-N(1)-C(13) | 118.17(18) |
| C(16)-O(4)-C(17) | 115.76(17) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for C17H15NO5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| C(1) | 13(1) | 16(1) | 19(1) | -2(1) | 3(1) | -1(1) |
| C(2) | 12(1) | 27(2) | 16(2) | -8(1) | 4(1) | -3(1) |
| C(3) | 14(1) | 28(2) | 11(1) | 2(1) | 2(1) | 1(1) |
| C(4) | 16(1) | 17(1) | 20(1) | 1(1) | 8(1) | 1(1) |
| C(5) | 11(1) | 22(2) | 12(1) | -5(1) | 2(1) | 0(1) |
| C(6) | 11(1) | 12(2) | 16(1) | -2(1) | 4(1) | -1(1) |
| C(7) | 12(1) | 16(2) | 21(1) | -2(1) | 5(1) | 1(1) |
| C(8) | 15(1) | 14(1) | 16(1) | 0(1) | 3(1) | 2(1) |
| C(9) | 11(1) | 16(1) | 16(1) | 0(1) | 4(1) | 1(1) |
| C(10) | 8(1) | 17(1) | 13(1) | -1(1) | 4(1) | 4(1) |
| C(11) | 14(1) | 18(1) | 14(1) | 3(1) | 4(1) | 3(1) |
| C(12) | 14(1) | 14(1) | 17(1) | -4(1) | 5(1) | -1(1) |
| C(13) | 12(1) | 16(2) | 11(1) | -3(1) | 2(1) | 2(1) |
| C(14) | 16(1) | 18(1) | 15(1) | 3(1) | 6(1) | 3(1) |
| C(15) | 15(1) | 14(1) | 17(1) | -1(1) | 4(1) | -2(1) |
| C(16) | 17(1) | 21(2) | 8(1) | -1(1) | -1(1) | -4(1) |
| C(17) | 20(1) | 21(1) | 26(1) | -3(1) | 10(1) | 4(1) |
| N(1) | 17(1) | 22(2) | 14(1) | -1(1) | 4(1) | 1(1) |
| O(1) | 35(1) | 15(1) | 20(1) | -1(1) | 0(1) | -4(1) |
| O(2) | 24(1) | 21(1) | 23(1) | -4(1) | 6(1) | -6(1) |
| O(3) | 36(1) | 30(1) | 13(1) | 4(1) | -2(1) | -2(1) |
| O(4) | 20(1) | 17(1) | 22(1) | 1(1) | 10(1) | 1(1) |
| O(5) | 26(1) | 15(1) | 22(1) | 6(1) | 7(1) | 1(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^{-3}$) for C17H15NO5.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 5265 | 5964 | 4647 | 19 |
| H(2) | 4374 | 6831 | 1971 | 22 |
| H(3) | 5079 | 8622 | 1762 | 21 |
| H(4) | 6692 | 9541 | 4238 | 21 |
| H(5) | 7586 | 8678 | 6909 | 18 |
| H(8A) | 7369 | 7800 | 9619 | 18 |
| H(8B) | 9277 | 7589 | 9080 | 18 |
| H(9) | 7714 | 6193 | 11176 | 17 |
| H(11) | 10492 | 8470 | 11445 | 18 |
| H(12) | 12092 | 9309 | 14022 | 18 |
| H(14) | 11262 | 6733 | 16790 | 19 |
| H(15) | 9577 | 5930 | 14224 | 19 |
| H(17A) | 13393 | 4977 | 10641 | 32 |
| H(17B) | 13622 | 5617 | 8962 | 32 |
| H(17C) | 12139 | 4676 | 8746 | 32 |

Table 6. Torsion angles [°] for C17H15NO5.

| | |
|---|---|
| C(6)-C(1)-C(2)-C(3) | -0.1(3) |
| C(1)-C(2)-C(3)-C(4) | 0.2(3) |
| C(2)-C(3)-C(4)-C(5) | -0.1(3) |
| C(3)-C(4)-C(5)-C(6) | 0.0(3) |
| C(4)-C(5)-C(6)-C(1) | 0.1(3) |
| C(4)-C(5)-C(6)-C(7) | 177.8(2) |
| C(2)-C(1)-C(6)-C(5) | -0.1(3) |
| C(2)-C(1)-C(6)-C(7) | -177.8(2) |
| C(5)-C(6)-C(7)-O(1) | -176.6(2) |
| C(1)-C(6)-C(7)-O(1) | 1.0(3) |
| C(5)-C(6)-C(7)-C(8) | 1.9(3) |
| C(1)-C(6)-C(7)-C(8) | 179.54(18) |
| O(1)-C(7)-C(8)-C(9) | 2.6(3) |
| C(6)-C(7)-C(8)-C(9) | -175.96(19) |
| C(7)-C(8)-C(9)-C(16) | 59.5(2) |
| C(7)-C(8)-C(9)-C(10) | -178.91(17) |
| C(8)-C(9)-C(10)-C(11) | -18.0(3) |
| C(16)-C(9)-C(10)-C(11) | 106.9(2) |
| C(8)-C(9)-C(10)-C(15) | 163.5(2) |
| C(16)-C(9)-C(10)-C(15) | -71.6(2) |
| C(15)-C(10)-C(11)-C(12) | -0.4(3) |
| C(9)-C(10)-C(11)-C(12) | -178.85(18) |
| C(10)-C(11)-C(12)-C(13) | 1.1(3) |
| C(11)-C(12)-C(13)-C(14) | -0.7(3) |
| C(11)-C(12)-C(13)-N(1) | 176.61(17) |
| C(12)-C(13)-C(14)-C(15) | -0.5(3) |
| N(1)-C(13)-C(14)-C(15) | -177.83(19) |
| C(13)-C(14)-C(15)-C(10) | 1.3(3) |
| C(11)-C(10)-C(15)-C(14) | -0.9(3) |
| C(9)-C(10)-C(15)-C(14) | 177.66(18) |
| C(8)-C(9)-C(16)-O(5) | -143.3(2) |
| C(10)-C(9)-C(16)-O(5) | 90.9(2) |
| C(8)-C(9)-C(16)-O(4) | 40.4(2) |
| C(10)-C(9)-C(16)-O(4) | -85.50(19) |

| | |
|---|---|
| C(12)-C(13)-N(1)-O(3) | -167.15(19) |
| C(14)-C(13)-N(1)-O(3) | 10.2(3) |
| C(12)-C(13)-N(1)-O(2) | 11.3(3) |
| C(14)-C(13)-N(1)-O(2) | -171.34(18) |
| O(5)-C(16)-O(4)-C(17) | -1.9(3) |
| C(9)-C(16)-O(4)-C(17) | 174.47(16) |

Symmetry transformations used to generate equivalent atoms:

Table 1. Crystal data and structure refinement for C17H15NO5 (**Mo data**).

| | | |
|---|---|---|
| Identification code | mo_fang053112_0m | |
| Empirical formula | C17 H15 N O5 | |
| Formula weight | 313.30 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P 21 | |
| Unit cell dimensions | a = 7.4205(4) Å | α= 90°. |
| | b = 12.6395(7) Å | β= 104.4190(10)°. |
| | c = 7.9161(5) Å | γ = 90°. |
| Volume | 719.08(7) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.447 $Mg/m^3$ | |
| Absorption coefficient | 0.108 $mm^{-1}$ | |
| F(000) | 328 | |
| Crystal size | 0.46 x 0.16 x 0.14 $mm^3$ | |
| Theta range for data collection | 2.66 to 30.64°. | |
| Index ranges | -10<=h<=10, -18<=k<=17, -11<=l<=11 | |
| Reflections collected | 17872 | |
| Independent reflections | 4367 [R(int) = 0.0444] | |
| Completeness to theta = 30.64° | 99.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.99 and 0.84 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4367 / 1 / 209 | |
| Goodness-of-fit on $F^2$ | 1.055 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0408, wR2 = 0.0910 | |
| R indices (all data) | R1 = 0.0543, wR2 = 0.0969 | |
| Absolute structure parameter | 0.6(8) | |
| Largest diff. peak and hole | 0.301 and -0.254 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for C17H15NO5. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 9446(2) | 1473(1) | 15435(2) | 16(1) |
| C(2) | 9974(2) | 1993(1) | 17027(2) | 18(1) |
| C(3) | 9559(2) | 3057(1) | 17155(2) | 18(1) |
| C(4) | 8596(2) | 3607(1) | 15683(2) | 17(1) |
| C(5) | 8063(2) | 3092(1) | 14082(2) | 14(1) |
| C(6) | 8484(2) | 2024(1) | 13942(2) | 13(1) |
| C(7) | 7870(2) | 1431(1) | 12259(2) | 14(1) |
| C(8) | 6823(2) | 2032(1) | 10661(2) | 14(1) |
| C(9) | 6163(2) | 1317(1) | 9073(2) | 13(1) |
| C(10) | 5127(2) | 1906(1) | 7427(2) | 13(1) |
| C(11) | 4368(2) | 2910(1) | 7473(2) | 14(1) |
| C(12) | 3406(2) | 3412(1) | 5948(2) | 14(1) |
| C(13) | 3182(2) | 2876(1) | 4386(2) | 14(1) |
| C(14) | 3911(2) | 1870(1) | 4290(2) | 16(1) |
| C(15) | 4899(2) | 1395(1) | 5817(2) | 15(1) |
| C(16) | 4870(2) | 437(1) | 9376(2) | 14(1) |
| C(17) | 2267(2) | 42(1) | 10500(2) | 20(1) |
| N(1) | 2082(2) | 3371(1) | 2778(2) | 17(1) |
| O(1) | 8174(2) | 486(1) | 12179(1) | 23(1) |
| O(2) | 1638(2) | 4305(1) | 2831(1) | 21(1) |
| O(3) | 1618(2) | 2818(1) | 1462(2) | 27(1) |
| O(4) | 3673(1) | 793(1) | 10278(1) | 17(1) |
| O(5) | 4842(2) | -452(1) | 8819(1) | 20(1) |

Table 3. Bond lengths [Å] and angles [°] for C17H15NO5.

| | |
|---|---|
| C(1)-C(2) | 1.389(2) |
| C(1)-C(6) | 1.404(2) |
| C(1)-H(1) | 0.9500 |
| C(2)-C(3) | 1.389(2) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.392(2) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.392(2) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.396(2) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.498(2) |
| C(7)-O(1) | 1.220(2) |
| C(7)-C(8) | 1.513(2) |
| C(8)-C(9) | 1.526(2) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-C(16) | 1.527(2) |
| C(9)-C(10) | 1.5310(19) |
| C(9)-H(9) | 1.0000 |
| C(10)-C(11) | 1.393(2) |
| C(10)-C(15) | 1.400(2) |
| C(11)-C(12) | 1.393(2) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.384(2) |
| C(12)-H(12) | 0.9500 |
| C(13)-C(14) | 1.391(2) |
| C(13)-N(1) | 1.4696(18) |
| C(14)-C(15) | 1.384(2) |
| C(14)-H(14) | 0.9500 |
| C(15)-H(15) | 0.9500 |
| C(16)-O(5) | 1.2058(18) |
| C(16)-O(4) | 1.3480(18) |
| C(17)-O(4) | 1.4536(18) |

| | |
|---|---|
| C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 |
| N(1)-O(3) | 1.2299(17) |
| N(1)-O(2) | 1.2302(17) |

| | |
|---|---|
| C(2)-C(1)-C(6) | 119.91(14) |
| C(2)-C(1)-H(1) | 120.0 |
| C(6)-C(1)-H(1) | 120.0 |
| C(1)-C(2)-C(3) | 120.43(14) |
| C(1)-C(2)-H(2) | 119.8 |
| C(3)-C(2)-H(2) | 119.8 |
| C(2)-C(3)-C(4) | 119.98(14) |
| C(2)-C(3)-H(3) | 120.0 |
| C(4)-C(3)-H(3) | 120.0 |
| C(5)-C(4)-C(3) | 119.92(15) |
| C(5)-C(4)-H(4) | 120.0 |
| C(3)-C(4)-H(4) | 120.0 |
| C(4)-C(5)-C(6) | 120.40(14) |
| C(4)-C(5)-H(5) | 119.8 |
| C(6)-C(5)-H(5) | 119.8 |
| C(5)-C(6)-C(1) | 119.36(14) |
| C(5)-C(6)-C(7) | 121.98(13) |
| C(1)-C(6)-C(7) | 118.60(14) |
| O(1)-C(7)-C(6) | 121.13(13) |
| O(1)-C(7)-C(8) | 120.70(13) |
| C(6)-C(7)-C(8) | 118.15(13) |
| C(7)-C(8)-C(9) | 112.60(12) |
| C(7)-C(8)-H(8A) | 109.1 |
| C(9)-C(8)-H(8A) | 109.1 |
| C(7)-C(8)-H(8B) | 109.1 |
| C(9)-C(8)-H(8B) | 109.1 |
| H(8A)-C(8)-H(8B) | 107.8 |
| C(8)-C(9)-C(16) | 112.77(12) |
| C(8)-C(9)-C(10) | 113.81(12) |
| C(16)-C(9)-C(10) | 106.60(11) |

| | |
|---|---|
| C(8)-C(9)-H(9) | 107.8 |
| C(16)-C(9)-H(9) | 107.8 |
| C(10)-C(9)-H(9) | 107.8 |
| C(11)-C(10)-C(15) | 119.06(13) |
| C(11)-C(10)-C(9) | 122.78(12) |
| C(15)-C(10)-C(9) | 118.14(13) |
| C(12)-C(11)-C(10) | 121.05(13) |
| C(12)-C(11)-H(11) | 119.5 |
| C(10)-C(11)-H(11) | 119.5 |
| C(13)-C(12)-C(11) | 118.24(14) |
| C(13)-C(12)-H(12) | 120.9 |
| C(11)-C(12)-H(12) | 120.9 |
| C(12)-C(13)-C(14) | 122.30(13) |
| C(12)-C(13)-N(1) | 118.86(14) |
| C(14)-C(13)-N(1) | 118.81(13) |
| C(15)-C(14)-C(13) | 118.50(13) |
| C(15)-C(14)-H(14) | 120.7 |
| C(13)-C(14)-H(14) | 120.7 |
| C(14)-C(15)-C(10) | 120.82(14) |
| C(14)-C(15)-H(15) | 119.6 |
| C(10)-C(15)-H(15) | 119.6 |
| O(5)-C(16)-O(4) | 123.49(14) |
| O(5)-C(16)-C(9) | 125.16(14) |
| O(4)-C(16)-C(9) | 111.26(12) |
| O(4)-C(17)-H(17A) | 109.5 |
| O(4)-C(17)-H(17B) | 109.5 |
| H(17A)-C(17)-H(17B) | 109.5 |
| O(4)-C(17)-H(17C) | 109.5 |
| H(17A)-C(17)-H(17C) | 109.5 |
| H(17B)-C(17)-H(17C) | 109.5 |
| O(3)-N(1)-O(2) | 123.61(13) |
| O(3)-N(1)-C(13) | 118.00(13) |
| O(2)-N(1)-C(13) | 118.36(13) |
| C(16)-O(4)-C(17) | 115.70(12) |

---

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for C17H15NO5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| C(1) | 15(1) | 16(1) | 17(1) | 4(1) | 3(1) | 0(1) |
| C(2) | 14(1) | 25(1) | 14(1) | 5(1) | 2(1) | 0(1) |
| C(3) | 16(1) | 25(1) | 12(1) | -2(1) | 2(1) | -1(1) |
| C(4) | 18(1) | 17(1) | 18(1) | -2(1) | 6(1) | -1(1) |
| C(5) | 14(1) | 14(1) | 13(1) | 3(1) | 1(1) | 0(1) |
| C(6) | 11(1) | 15(1) | 13(1) | 1(1) | 2(1) | -1(1) |
| C(7) | 13(1) | 15(1) | 15(1) | 2(1) | 2(1) | -1(1) |
| C(8) | 16(1) | 13(1) | 12(1) | 1(1) | -1(1) | -2(1) |
| C(9) | 14(1) | 14(1) | 11(1) | 0(1) | 2(1) | 0(1) |
| C(10) | 12(1) | 15(1) | 11(1) | 1(1) | 2(1) | -2(1) |
| C(11) | 15(1) | 15(1) | 14(1) | -2(1) | 2(1) | -2(1) |
| C(12) | 14(1) | 14(1) | 15(1) | 0(1) | 4(1) | 0(1) |
| C(13) | 13(1) | 16(1) | 12(1) | 3(1) | 2(1) | -1(1) |
| C(14) | 18(1) | 17(1) | 13(1) | -2(1) | 5(1) | -1(1) |
| C(15) | 16(1) | 14(1) | 15(1) | 0(1) | 4(1) | 2(1) |
| C(16) | 15(1) | 16(1) | 11(1) | 1(1) | 0(1) | 0(1) |
| C(17) | 18(1) | 19(1) | 24(1) | 2(1) | 7(1) | -3(1) |
| N(1) | 16(1) | 20(1) | 14(1) | 3(1) | 4(1) | 0(1) |
| O(1) | 33(1) | 15(1) | 19(1) | 0(1) | 0(1) | 3(1) |
| O(2) | 22(1) | 20(1) | 21(1) | 4(1) | 4(1) | 5(1) |
| O(3) | 35(1) | 30(1) | 13(1) | -1(1) | -3(1) | 2(1) |
| O(4) | 18(1) | 16(1) | 20(1) | -2(1) | 7(1) | -2(1) |
| O(5) | 23(1) | 15(1) | 21(1) | -3(1) | 6(1) | -3(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^{-3}$) for C17H15NO5.

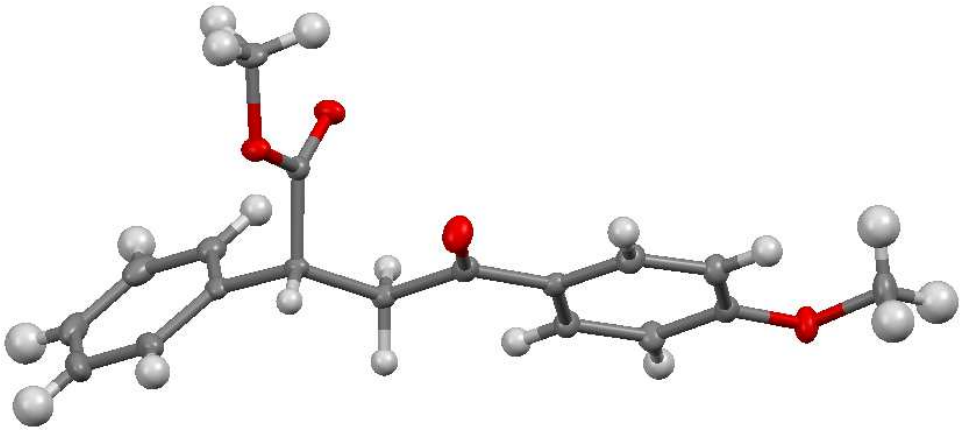
| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 9735 | 745 | 15356 | 19 |
| H(2) | 10624 | 1618 | 18036 | 22 |
| H(3) | 9933 | 3409 | 18246 | 22 |
| H(4) | 8303 | 4333 | 15770 | 21 |
| H(5) | 7408 | 3469 | 13078 | 17 |
| H(8A) | 7642 | 2589 | 10384 | 17 |
| H(8B) | 5732 | 2384 | 10923 | 17 |
| H(9) | 7286 | 978 | 8821 | 16 |
| H(11) | 4508 | 3258 | 8561 | 17 |
| H(12) | 2917 | 4104 | 5980 | 17 |
| H(14) | 3735 | 1516 | 3202 | 19 |
| H(15) | 5429 | 715 | 5773 | 18 |
| H(17A) | 1619 | -246 | 9361 | 30 |
| H(17B) | 1369 | 401 | 11026 | 30 |
| H(17C) | 2863 | -535 | 11264 | 30 |

Table 6. Torsion angles [°] for C17H15NO5.

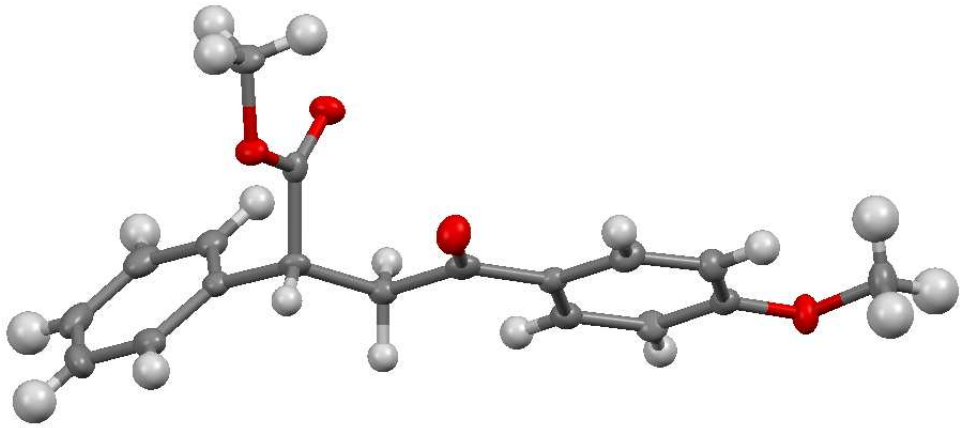
| | |
|---|---|
| C(6)-C(1)-C(2)-C(3) | -0.1(2) |
| C(1)-C(2)-C(3)-C(4) | 0.5(2) |
| C(2)-C(3)-C(4)-C(5) | -0.5(2) |
| C(3)-C(4)-C(5)-C(6) | 0.1(2) |
| C(4)-C(5)-C(6)-C(1) | 0.3(2) |
| C(4)-C(5)-C(6)-C(7) | 177.55(14) |
| C(2)-C(1)-C(6)-C(5) | -0.3(2) |
| C(2)-C(1)-C(6)-C(7) | -177.67(14) |
| C(5)-C(6)-C(7)-O(1) | -176.49(14) |
| C(1)-C(6)-C(7)-O(1) | 0.8(2) |
| C(5)-C(6)-C(7)-C(8) | 2.3(2) |
| C(1)-C(6)-C(7)-C(8) | 179.60(13) |
| O(1)-C(7)-C(8)-C(9) | 2.9(2) |
| C(6)-C(7)-C(8)-C(9) | -175.94(12) |
| C(7)-C(8)-C(9)-C(16) | 59.36(16) |
| C(7)-C(8)-C(9)-C(10) | -179.05(11) |
| C(8)-C(9)-C(10)-C(11) | -18.26(19) |
| C(16)-C(9)-C(10)-C(11) | 106.70(15) |
| C(8)-C(9)-C(10)-C(15) | 163.37(13) |
| C(16)-C(9)-C(10)-C(15) | -71.68(16) |
| C(15)-C(10)-C(11)-C(12) | -0.5(2) |
| C(9)-C(10)-C(11)-C(12) | -178.87(13) |
| C(10)-C(11)-C(12)-C(13) | 1.6(2) |
| C(11)-C(12)-C(13)-C(14) | -1.3(2) |
| C(11)-C(12)-C(13)-N(1) | 176.65(13) |
| C(12)-C(13)-C(14)-C(15) | -0.2(2) |
| N(1)-C(13)-C(14)-C(15) | -178.11(13) |
| C(13)-C(14)-C(15)-C(10) | 1.3(2) |
| C(11)-C(10)-C(15)-C(14) | -1.0(2) |
| C(9)-C(10)-C(15)-C(14) | 177.44(13) |
| C(8)-C(9)-C(16)-O(5) | -143.12(15) |
| C(10)-C(9)-C(16)-O(5) | 91.28(17) |
| C(8)-C(9)-C(16)-O(4) | 40.28(16) |
| C(10)-C(9)-C(16)-O(4) | -85.31(14) |

| | |
|---|---|
| C(12)-C(13)-N(1)-O(3) | -167.41(14) |
| C(14)-C(13)-N(1)-O(3) | 10.6(2) |
| C(12)-C(13)-N(1)-O(2) | 10.9(2) |
| C(14)-C(13)-N(1)-O(2) | -171.15(13) |
| O(5)-C(16)-O(4)-C(17) | -2.0(2) |
| C(9)-C(16)-O(4)-C(17) | 174.61(12) |

Symmetry transformations used to generate equivalent atoms:



Crystal Structure of **4e** derived from Mo data



Crystal Structure of **4e** derived from Cu data

**CCDC deposition numbers: CCDC 885742, CCDD 885743**

**Critical Parameters**

$C_{18}H_{18}O_4$
Flack x: 0.0(2)
Hooft y: -0.02(0.07)
Bayesian Statistics: P2(true) 1.000

structure refinement for C18H18O4 (**Cu data**).

| | | |
|---|---|---|
| Identification code | cu_fang060212_0m | |
| Empirical formula | C18 H18 O4 | |
| Formula weight | 298.32 | |
| Temperature | 100(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Orthorhombic | |
| Space group | P2(1)2(1)2(1) | |
| Unit cell dimensions | a = 7.7671(2) Å | α= 90°. |
| | b = 7.9627(2) Å | β= 90°. |
| | c = 24.9339(5) Å | γ = 90°. |
| Volume | 1542.09(6) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.285 $Mg/m^3$ | |
| Absorption coefficient | 0.738 $mm^{-1}$ | |
| F(000) | 632 | |
| Crystal size | 0.44 x 0.44 x 0.16 $mm^3$ | |
| Theta range for data collection | 3.55 to 44.38°. | |
| Index ranges | -7<=h<=7, -7<=k<=7, -21<=l<=22 | |
| Reflections collected | 10685 | |
| Independent reflections | 1194 [R(int) = 0.0374] | |
| Completeness to theta = 44.38° | 99.3 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.89 and 0.72 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 1194 / 0 / 202 | |
| Goodness-of-fit on $F^2$ | 1.073 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0245, wR2 = 0.0604 | |
| R indices (all data) | R1 = 0.0245, wR2 = 0.0606 | |
| Absolute structure parameter | 0.0(2) | |
| Extinction coefficient | 0.0114(7) | |
| Largest diff. peak and hole | 0.189 and -0.172 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for C18H18O4. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 1463(3) | 6442(3) | 8902(1) | 28(1) |
| C(2) | 2068(3) | 7326(3) | 9343(1) | 38(1) |
| C(3) | 3751(4) | 7890(3) | 9357(1) | 38(1) |
| C(4) | 4826(3) | 7564(3) | 8931(1) | 33(1) |
| C(5) | 4219(3) | 6679(3) | 8491(1) | 25(1) |
| C(6) | 2532(3) | 6112(2) | 8469(1) | 20(1) |
| C(7) | 1872(3) | 5183(2) | 7975(1) | 20(1) |
| C(8) | 2019(2) | 6276(2) | 7474(1) | 20(1) |
| C(9) | 1547(3) | 5375(3) | 6967(1) | 22(1) |
| C(10) | 1797(2) | 6227(3) | 6445(1) | 18(1) |
| C(11) | 2454(2) | 7851(3) | 6406(1) | 23(1) |
| C(12) | 2679(2) | 8590(2) | 5908(1) | 23(1) |
| C(13) | 2270(2) | 7735(3) | 5445(1) | 21(1) |
| C(14) | 1626(2) | 6115(3) | 5477(1) | 25(1) |
| C(15) | 1389(2) | 5394(3) | 5973(1) | 25(1) |
| C(16) | 2885(3) | 3581(3) | 7906(1) | 19(1) |
| C(17) | 3029(3) | 694(3) | 8107(1) | 28(1) |
| C(18) | 2306(3) | 7701(3) | 4486(1) | 35(1) |
| O(1) | 4273(2) | 3498(2) | 7687(1) | 25(1) |
| O(2) | 2112(2) | 2270(2) | 8138(1) | 22(1) |
| O(3) | 973(2) | 3931(2) | 6985(1) | 30(1) |
| O(4) | 2554(2) | 8581(2) | 4979(1) | 29(1) |

Table 3. Bond lengths [Å] and angles [°] for C18H18O4.

| | |
|---|---|
| C(1)-C(2) | 1.386(3) |
| C(1)-C(6) | 1.387(3) |
| C(1)-H(1) | 0.9500 |
| C(2)-C(3) | 1.383(3) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.375(3) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.387(3) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.387(3) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.526(3) |
| C(7)-C(16) | 1.509(3) |
| C(7)-C(8) | 1.528(2) |
| C(7)-H(7) | 1.0000 |
| C(8)-C(9) | 1.499(3) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-O(3) | 1.234(2) |
| C(9)-C(10) | 1.480(3) |
| C(10)-C(15) | 1.388(3) |
| C(10)-C(11) | 1.393(3) |
| C(11)-C(12) | 1.384(3) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.378(3) |
| C(12)-H(12) | 0.9500 |
| C(13)-O(4) | 1.361(2) |
| C(13)-C(14) | 1.386(3) |
| C(14)-C(15) | 1.377(3) |
| C(14)-H(14) | 0.9500 |
| C(15)-H(15) | 0.9500 |
| C(16)-O(1) | 1.210(2) |
| C(16)-O(2) | 1.335(2) |
| C(17)-O(2) | 1.445(2) |

| | |
|---|---|
| C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 |
| C(18)-O(4) | 1.428(2) |
| C(18)-H(18A) | 0.9800 |
| C(18)-H(18B) | 0.9800 |
| C(18)-H(18C) | 0.9800 |

| | |
|---|---|
| C(2)-C(1)-C(6) | 120.65(19) |
| C(2)-C(1)-H(1) | 119.7 |
| C(6)-C(1)-H(1) | 119.7 |
| C(3)-C(2)-C(1) | 120.4(2) |
| C(3)-C(2)-H(2) | 119.8 |
| C(1)-C(2)-H(2) | 119.8 |
| C(4)-C(3)-C(2) | 119.5(2) |
| C(4)-C(3)-H(3) | 120.2 |
| C(2)-C(3)-H(3) | 120.2 |
| C(3)-C(4)-C(5) | 120.0(2) |
| C(3)-C(4)-H(4) | 120.0 |
| C(5)-C(4)-H(4) | 120.0 |
| C(6)-C(5)-C(4) | 121.1(2) |
| C(6)-C(5)-H(5) | 119.4 |
| C(4)-C(5)-H(5) | 119.4 |
| C(1)-C(6)-C(5) | 118.28(18) |
| C(1)-C(6)-C(7) | 121.28(18) |
| C(5)-C(6)-C(7) | 120.43(18) |
| C(16)-C(7)-C(6) | 109.12(15) |
| C(16)-C(7)-C(8) | 110.44(15) |
| C(6)-C(7)-C(8) | 111.05(16) |
| C(16)-C(7)-H(7) | 108.7 |
| C(6)-C(7)-H(7) | 108.7 |
| C(8)-C(7)-H(7) | 108.7 |
| C(9)-C(8)-C(7) | 113.54(17) |
| C(9)-C(8)-H(8A) | 108.9 |
| C(7)-C(8)-H(8A) | 108.9 |
| C(9)-C(8)-H(8B) | 108.9 |

| | |
|---|---|
| C(7)-C(8)-H(8B) | 108.9 |
| H(8A)-C(8)-H(8B) | 107.7 |
| O(3)-C(9)-C(10) | 120.43(18) |
| O(3)-C(9)-C(8) | 120.25(19) |
| C(10)-C(9)-C(8) | 119.3(2) |
| C(15)-C(10)-C(11) | 117.85(19) |
| C(15)-C(10)-C(9) | 119.73(19) |
| C(11)-C(10)-C(9) | 122.41(19) |
| C(12)-C(11)-C(10) | 120.29(18) |
| C(12)-C(11)-H(11) | 119.9 |
| C(10)-C(11)-H(11) | 119.9 |
| C(13)-C(12)-C(11) | 120.81(18) |
| C(13)-C(12)-H(12) | 119.6 |
| C(11)-C(12)-H(12) | 119.6 |
| O(4)-C(13)-C(12) | 115.72(18) |
| O(4)-C(13)-C(14) | 124.62(18) |
| C(12)-C(13)-C(14) | 119.66(19) |
| C(15)-C(14)-C(13) | 119.20(19) |
| C(15)-C(14)-H(14) | 120.4 |
| C(13)-C(14)-H(14) | 120.4 |
| C(14)-C(15)-C(10) | 122.19(19) |
| C(14)-C(15)-H(15) | 118.9 |
| C(10)-C(15)-H(15) | 118.9 |
| O(1)-C(16)-O(2) | 123.61(19) |
| O(1)-C(16)-C(7) | 124.2(2) |
| O(2)-C(16)-C(7) | 112.13(19) |
| O(2)-C(17)-H(17A) | 109.5 |
| O(2)-C(17)-H(17B) | 109.5 |
| H(17A)-C(17)-H(17B) | 109.5 |
| O(2)-C(17)-H(17C) | 109.5 |
| H(17A)-C(17)-H(17C) | 109.5 |
| H(17B)-C(17)-H(17C) | 109.5 |
| O(4)-C(18)-H(18A) | 109.5 |
| O(4)-C(18)-H(18B) | 109.5 |
| H(18A)-C(18)-H(18B) | 109.5 |
| O(4)-C(18)-H(18C) | 109.5 |

| | |
|---|---|
| H(18A)-C(18)-H(18C) | 109.5 |
| H(18B)-C(18)-H(18C) | 109.5 |
| C(16)-O(2)-C(17) | 115.72(15) |
| C(13)-O(4)-C(18) | 118.07(15) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for C18H18O4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| C(1) | 29(1) | 26(1) | 28(1) | 5(1) | 4(1) | 7(1) |
| C(2) | 55(2) | 38(1) | 22(1) | -3(1) | 2(1) | 17(2) |
| C(3) | 56(2) | 28(1) | 31(2) | -1(1) | -18(2) | 13(1) |
| C(4) | 39(1) | 22(1) | 38(2) | 0(1) | -12(2) | 6(1) |
| C(5) | 31(2) | 19(1) | 26(1) | 0(1) | -3(1) | 6(1) |
| C(6) | 23(1) | 17(1) | 21(1) | 1(1) | -2(1) | 4(1) |
| C(7) | 12(1) | 21(1) | 26(1) | 1(1) | 2(1) | 2(1) |
| C(8) | 18(1) | 18(1) | 22(1) | 0(1) | 1(1) | 1(1) |
| C(9) | 13(1) | 21(2) | 32(2) | 0(1) | 0(1) | 2(1) |
| C(10) | 16(1) | 18(2) | 22(2) | 1(1) | -1(1) | 1(1) |
| C(11) | 21(1) | 22(2) | 26(1) | -2(1) | 0(1) | 3(1) |
| C(12) | 26(1) | 16(1) | 27(2) | 0(1) | 0(1) | -4(1) |
| C(13) | 18(1) | 22(2) | 23(2) | 2(1) | 0(1) | 2(1) |
| C(14) | 26(1) | 27(2) | 22(2) | -4(1) | -4(1) | -1(1) |
| C(15) | 24(1) | 19(1) | 32(2) | 2(1) | -4(1) | -3(1) |
| C(16) | 19(1) | 23(2) | 16(1) | 1(1) | -2(1) | -3(1) |
| C(17) | 29(1) | 18(1) | 36(1) | 2(1) | 3(1) | 3(1) |
| C(18) | 44(2) | 42(1) | 20(1) | -1(1) | -2(1) | -7(1) |
| O(1) | 20(1) | 24(1) | 31(1) | 0(1) | 5(1) | 1(1) |
| O(2) | 22(1) | 16(1) | 28(1) | 4(1) | 3(1) | 0(1) |
| O(3) | 32(1) | 27(1) | 30(1) | 2(1) | -4(1) | -8(1) |
| O(4) | 39(1) | 29(1) | 18(1) | 2(1) | 0(1) | -5(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^{-3}$) for C18H18O4.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 305 | 6059 | 8897 | 34 |
| H(2) | 1322 | 7545 | 9636 | 46 |
| H(3) | 4162 | 8498 | 9659 | 46 |
| H(4) | 5985 | 7946 | 8939 | 40 |
| H(5) | 4972 | 6456 | 8199 | 30 |
| H(7) | 634 | 4890 | 8032 | 23 |
| H(8A) | 1259 | 7265 | 7516 | 23 |
| H(8B) | 3217 | 6689 | 7443 | 23 |
| H(11) | 2749 | 8453 | 6721 | 27 |
| H(12) | 3121 | 9700 | 5886 | 27 |
| H(14) | 1352 | 5508 | 5160 | 30 |
| H(15) | 930 | 4290 | 5993 | 30 |
| H(17A) | 3219 | 399 | 7730 | 41 |
| H(17B) | 2348 | -189 | 8281 | 41 |
| H(17C) | 4141 | 802 | 8289 | 41 |
| H(18A) | 3006 | 6678 | 4485 | 53 |
| H(18B) | 2652 | 8421 | 4186 | 53 |
| H(18C) | 1088 | 7402 | 4448 | 53 |

Table 6. Torsion angles [°] for C18H18O4.

| | |
|---|---|
| C(6)-C(1)-C(2)-C(3) | -0.1(3) |
| C(1)-C(2)-C(3)-C(4) | -0.2(3) |
| C(2)-C(3)-C(4)-C(5) | 0.1(3) |
| C(3)-C(4)-C(5)-C(6) | 0.2(3) |
| C(2)-C(1)-C(6)-C(5) | 0.4(3) |
| C(2)-C(1)-C(6)-C(7) | -178.36(17) |
| C(4)-C(5)-C(6)-C(1) | -0.5(3) |
| C(4)-C(5)-C(6)-C(7) | 178.31(17) |
| C(1)-C(6)-C(7)-C(16) | -119.0(2) |
| C(5)-C(6)-C(7)-C(16) | 62.3(2) |
| C(1)-C(6)-C(7)-C(8) | 119.1(2) |
| C(5)-C(6)-C(7)-C(8) | -59.7(2) |
| C(16)-C(7)-C(8)-C(9) | 53.3(2) |
| C(6)-C(7)-C(8)-C(9) | 174.54(15) |
| C(7)-C(8)-C(9)-O(3) | 5.3(3) |
| C(7)-C(8)-C(9)-C(10) | -173.87(16) |
| O(3)-C(9)-C(10)-C(15) | -0.2(3) |
| C(8)-C(9)-C(10)-C(15) | 179.02(17) |
| O(3)-C(9)-C(10)-C(11) | -179.45(17) |
| C(8)-C(9)-C(10)-C(11) | -0.3(3) |
| C(15)-C(10)-C(11)-C(12) | 0.1(3) |
| C(9)-C(10)-C(11)-C(12) | 179.44(17) |
| C(10)-C(11)-C(12)-C(13) | -0.4(3) |
| C(11)-C(12)-C(13)-O(4) | -179.62(15) |
| C(11)-C(12)-C(13)-C(14) | 0.0(3) |
| O(4)-C(13)-C(14)-C(15) | -179.75(17) |
| C(12)-C(13)-C(14)-C(15) | 0.7(3) |
| C(13)-C(14)-C(15)-C(10) | -1.0(3) |
| C(11)-C(10)-C(15)-C(14) | 0.6(3) |
| C(9)-C(10)-C(15)-C(14) | -178.76(17) |
| C(6)-C(7)-C(16)-O(1) | -83.2(2) |
| C(8)-C(7)-C(16)-O(1) | 39.2(2) |
| C(6)-C(7)-C(16)-O(2) | 93.80(18) |
| C(8)-C(7)-C(16)-O(2) | -143.87(16) |

| | |
|---|---|
| O(1)-C(16)-O(2)-C(17) | -0.9(2) |
| C(7)-C(16)-O(2)-C(17) | -177.86(15) |
| C(12)-C(13)-O(4)-C(18) | 174.27(16) |
| C(14)-C(13)-O(4)-C(18) | -5.3(3) |

Symmetry transformations used to generate equivalent atoms:

Table 1. Crystal data and structure refinement for C18H18O4 (**Mo data**).

| | | |
|---|---|---|
| Identification code | mo_fang060212_0m | |
| Empirical formula | C18 H18 O4 | |
| Formula weight | 298.32 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Orthorhombic | |
| Space group | P2(1)2(1)2(1) | |
| Unit cell dimensions | a = 7.7714(4) Å | α= 90°. |
| | b = 7.9651(4) Å | β= 90°. |
| | c = 24.9654(14) Å | γ = 90°. |
| Volume | 1545.36(14) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.282 $Mg/m^3$ | |
| Absorption coefficient | 0.090 $mm^{-1}$ | |
| F(000) | 632 | |
| Crystal size | 0.44 x 0.44 x 0.16 $mm^3$ | |
| Theta range for data collection | 1.63 to 30.50°. | |
| Index ranges | -10<=h<=11, -11<=k<=11, -35<=l<=35 | |
| Reflections collected | 38337 | |
| Independent reflections | 4667 [R(int) = 0.0328] | |
| Completeness to theta = 29.00° | 100.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9855 and 0.9614 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4667 / 0 / 201 | |
| Goodness-of-fit on $F^2$ | 1.043 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0319, wR2 = 0.0816 | |
| R indices (all data) | R1 = 0.0355, wR2 = 0.0838 | |
| Absolute structure parameter | -0.5(6) | |
| Largest diff. peak and hole | 0.277 and -0.216 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for C18H18O4. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 1458(2) | 6438(1) | 3902(1) | 24(1) |
| C(2) | 2063(2) | 7327(2) | 4345(1) | 33(1) |
| C(3) | 3748(2) | 7895(2) | 4359(1) | 34(1) |
| C(4) | 4832(2) | 7566(1) | 3933(1) | 28(1) |
| C(5) | 4235(1) | 6678(1) | 3488(1) | 21(1) |
| C(6) | 2535(1) | 6112(1) | 3469(1) | 17(1) |
| C(7) | 1862(1) | 5188(1) | 2976(1) | 14(1) |
| C(8) | 2022(1) | 6284(1) | 2474(1) | 15(1) |
| C(9) | 1539(1) | 5359(1) | 1968(1) | 17(1) |
| C(10) | 1797(1) | 6226(1) | 1445(1) | 15(1) |
| C(11) | 2455(1) | 7859(1) | 1407(1) | 18(1) |
| C(12) | 2682(1) | 8607(1) | 911(1) | 20(1) |
| C(13) | 2268(1) | 7732(1) | 443(1) | 18(1) |
| C(14) | 1618(1) | 6103(1) | 473(1) | 20(1) |
| C(15) | 1383(1) | 5379(1) | 973(1) | 20(1) |
| C(16) | 2891(1) | 3582(1) | 2905(1) | 14(1) |
| C(17) | 3034(1) | 698(1) | 3107(1) | 22(1) |
| C(18) | 2303(2) | 7704(2) | -515(1) | 29(1) |
| O(1) | 4276(1) | 3496(1) | 2687(1) | 19(1) |
| O(2) | 2108(1) | 2271(1) | 3138(1) | 17(1) |
| O(3) | 970(1) | 3929(1) | 1985(1) | 24(1) |
| O(4) | 2554(1) | 8587(1) | -21(1) | 25(1) |

Table 3. Bond lengths [Å] and angles [°] for C18H18O4.

| | |
|---|---|
| C(1)-C(6) | 1.3918(14) |
| C(1)-C(2) | 1.3940(16) |
| C(1)-H(1) | 0.9500 |
| C(2)-C(3) | 1.386(2) |
| C(2)-H(2) | 0.9500 |
| C(3)-C(4) | 1.3818(19) |
| C(3)-H(3) | 0.9500 |
| C(4)-C(5) | 1.3952(14) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.3976(15) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.5262(13) |
| C(7)-C(16) | 1.5191(13) |
| C(7)-C(8) | 1.5307(12) |
| C(7)-H(7) | 1.0000 |
| C(8)-C(9) | 1.5107(13) |
| C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 |
| C(9)-O(3) | 1.2227(12) |
| C(9)-C(10) | 1.4899(13) |
| C(10)-C(15) | 1.3950(13) |
| C(10)-C(11) | 1.4008(13) |
| C(11)-C(12) | 1.3851(13) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.3980(13) |
| C(12)-H(12) | 0.9500 |
| C(13)-O(4) | 1.3613(11) |
| C(13)-C(14) | 1.3945(14) |
| C(14)-C(15) | 1.3869(14) |
| C(14)-H(14) | 0.9500 |
| C(15)-H(15) | 0.9500 |
| C(16)-O(1) | 1.2083(12) |
| C(16)-O(2) | 1.3399(11) |
| C(17)-O(2) | 1.4466(11) |

| | |
|---|---|
| C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 |
| C(18)-O(4) | 1.4330(12) |
| C(18)-H(18A) | 0.9800 |
| C(18)-H(18B) | 0.9800 |
| C(18)-H(18C) | 0.9800 |

| | |
|---|---|
| C(6)-C(1)-C(2) | 120.57(11) |
| C(6)-C(1)-H(1) | 119.7 |
| C(2)-C(1)-H(1) | 119.7 |
| C(3)-C(2)-C(1) | 120.30(11) |
| C(3)-C(2)-H(2) | 119.9 |
| C(1)-C(2)-H(2) | 119.9 |
| C(4)-C(3)-C(2) | 119.63(11) |
| C(4)-C(3)-H(3) | 120.2 |
| C(2)-C(3)-H(3) | 120.2 |
| C(3)-C(4)-C(5) | 120.37(11) |
| C(3)-C(4)-H(4) | 119.8 |
| C(5)-C(4)-H(4) | 119.8 |
| C(4)-C(5)-C(6) | 120.40(10) |
| C(4)-C(5)-H(5) | 119.8 |
| C(6)-C(5)-H(5) | 119.8 |
| C(1)-C(6)-C(5) | 118.72(9) |
| C(1)-C(6)-C(7) | 120.74(9) |
| C(5)-C(6)-C(7) | 120.52(8) |
| C(16)-C(7)-C(6) | 108.61(7) |
| C(16)-C(7)-C(8) | 110.05(7) |
| C(6)-C(7)-C(8) | 110.94(7) |
| C(16)-C(7)-H(7) | 109.1 |
| C(6)-C(7)-H(7) | 109.1 |
| C(8)-C(7)-H(7) | 109.1 |
| C(9)-C(8)-C(7) | 112.76(8) |
| C(9)-C(8)-H(8A) | 109.0 |
| C(7)-C(8)-H(8A) | 109.0 |
| C(9)-C(8)-H(8B) | 109.0 |

| | |
|---|---|
| C(7)-C(8)-H(8B) | 109.0 |
| H(8A)-C(8)-H(8B) | 107.8 |
| O(3)-C(9)-C(10) | 120.77(9) |
| O(3)-C(9)-C(8) | 120.93(9) |
| C(10)-C(9)-C(8) | 118.30(8) |
| C(15)-C(10)-C(11) | 118.39(9) |
| C(15)-C(10)-C(9) | 118.98(8) |
| C(11)-C(10)-C(9) | 122.62(8) |
| C(12)-C(11)-C(10) | 120.46(9) |
| C(12)-C(11)-H(11) | 119.8 |
| C(10)-C(11)-H(11) | 119.8 |
| C(11)-C(12)-C(13) | 120.21(8) |
| C(11)-C(12)-H(12) | 119.9 |
| C(13)-C(12)-H(12) | 119.9 |
| O(4)-C(13)-C(14) | 124.82(9) |
| O(4)-C(13)-C(12) | 115.07(8) |
| C(14)-C(13)-C(12) | 120.11(9) |
| C(15)-C(14)-C(13) | 118.92(9) |
| C(15)-C(14)-H(14) | 120.5 |
| C(13)-C(14)-H(14) | 120.5 |
| C(14)-C(15)-C(10) | 121.90(9) |
| C(14)-C(15)-H(15) | 119.1 |
| C(10)-C(15)-H(15) | 119.1 |
| O(1)-C(16)-O(2) | 123.74(8) |
| O(1)-C(16)-C(7) | 124.63(8) |
| O(2)-C(16)-C(7) | 111.56(7) |
| O(2)-C(17)-H(17A) | 109.5 |
| O(2)-C(17)-H(17B) | 109.5 |
| H(17A)-C(17)-H(17B) | 109.5 |
| O(2)-C(17)-H(17C) | 109.5 |
| H(17A)-C(17)-H(17C) | 109.5 |
| H(17B)-C(17)-H(17C) | 109.5 |
| O(4)-C(18)-H(18A) | 109.5 |
| O(4)-C(18)-H(18B) | 109.5 |
| H(18A)-C(18)-H(18B) | 109.5 |
| O(4)-C(18)-H(18C) | 109.5 |

| | |
|---|---|
| H(18A)-C(18)-H(18C) | 109.5 |
| H(18B)-C(18)-H(18C) | 109.5 |
| C(16)-O(2)-C(17) | 115.22(7) |
| C(13)-O(4)-C(18) | 117.66(8) |

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for C18H18O4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| C(1) | 30(1) | 24(1) | 20(1) | 2(1) | 4(1) | 8(1) |
| C(2) | 50(1) | 32(1) | 17(1) | -2(1) | 1(1) | 17(1) |
| C(3) | 55(1) | 23(1) | 23(1) | -5(1) | -16(1) | 13(1) |
| C(4) | 34(1) | 17(1) | 34(1) | -1(1) | -15(1) | 4(1) |
| C(5) | 24(1) | 16(1) | 22(1) | 0(1) | -3(1) | 2(1) |
| C(6) | 22(1) | 12(1) | 16(1) | 2(1) | 0(1) | 4(1) |
| C(7) | 13(1) | 14(1) | 16(1) | 2(1) | 2(1) | 2(1) |
| C(8) | 17(1) | 14(1) | 16(1) | 2(1) | 0(1) | 0(1) |
| C(9) | 14(1) | 17(1) | 19(1) | 2(1) | 0(1) | -1(1) |
| C(10) | 13(1) | 15(1) | 17(1) | 1(1) | -1(1) | 0(1) |
| C(11) | 21(1) | 15(1) | 17(1) | -1(1) | -2(1) | -1(1) |
| C(12) | 25(1) | 14(1) | 20(1) | 1(1) | 0(1) | -3(1) |
| C(13) | 18(1) | 19(1) | 16(1) | 1(1) | 0(1) | 1(1) |
| C(14) | 23(1) | 20(1) | 18(1) | -2(1) | -3(1) | -3(1) |
| C(15) | 21(1) | 17(1) | 22(1) | 0(1) | -3(1) | -3(1) |
| C(16) | 16(1) | 14(1) | 14(1) | 0(1) | 0(1) | 0(1) |
| C(17) | 24(1) | 13(1) | 28(1) | 3(1) | 3(1) | 2(1) |
| C(18) | 38(1) | 34(1) | 15(1) | -1(1) | -2(1) | -7(1) |
| O(1) | 17(1) | 17(1) | 24(1) | 2(1) | 5(1) | 1(1) |
| O(2) | 18(1) | 13(1) | 21(1) | 3(1) | 4(1) | 1(1) |
| O(3) | 29(1) | 19(1) | 24(1) | 3(1) | -4(1) | -9(1) |
| O(4) | 37(1) | 22(1) | 15(1) | 2(1) | 0(1) | -4(1) |

Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^{-3}$) for C18H18O4.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 302 | 6053 | 3896 | 29 |
| H(2) | 1316 | 7544 | 4638 | 40 |
| H(3) | 4155 | 8507 | 4660 | 41 |
| H(4) | 5990 | 7947 | 3943 | 34 |
| H(5) | 4990 | 6458 | 3198 | 25 |
| H(7) | 624 | 4896 | 3033 | 17 |
| H(8A) | 1267 | 7277 | 2514 | 18 |
| H(8B) | 3222 | 6687 | 2443 | 18 |
| H(11) | 2747 | 8457 | 1723 | 21 |
| H(12) | 3120 | 9718 | 889 | 24 |
| H(14) | 1339 | 5499 | 157 | 24 |
| H(15) | 928 | 4274 | 995 | 24 |
| H(17A) | 3230 | 407 | 2731 | 32 |
| H(17B) | 2356 | -188 | 3279 | 32 |
| H(17C) | 4143 | 811 | 3291 | 32 |
| H(18A) | 3014 | 6688 | -517 | 44 |
| H(18B) | 2634 | 8429 | -815 | 44 |
| H(18C) | 1088 | 7392 | -550 | 44 |

Table 6. Torsion angles [°] for C18H18O4.

| | |
|---|---|
| C(6)-C(1)-C(2)-C(3) | -0.06(17) |
| C(1)-C(2)-C(3)-C(4) | -0.46(17) |
| C(2)-C(3)-C(4)-C(5) | 0.48(17) |
| C(3)-C(4)-C(5)-C(6) | 0.01(16) |
| C(2)-C(1)-C(6)-C(5) | 0.54(15) |
| C(2)-C(1)-C(6)-C(7) | -178.03(9) |
| C(4)-C(5)-C(6)-C(1) | -0.52(14) |
| C(4)-C(5)-C(6)-C(7) | 178.06(9) |
| C(1)-C(6)-C(7)-C(16) | -119.46(9) |
| C(5)-C(6)-C(7)-C(16) | 61.99(11) |
| C(1)-C(6)-C(7)-C(8) | 119.46(10) |
| C(5)-C(6)-C(7)-C(8) | -59.09(11) |
| C(16)-C(7)-C(8)-C(9) | 53.98(10) |
| C(6)-C(7)-C(8)-C(9) | 174.21(7) |
| C(7)-C(8)-C(9)-O(3) | 4.93(13) |
| C(7)-C(8)-C(9)-C(10) | -174.19(8) |
| O(3)-C(9)-C(10)-C(15) | -0.13(14) |
| C(8)-C(9)-C(10)-C(15) | 178.99(9) |
| O(3)-C(9)-C(10)-C(11) | -179.62(10) |
| C(8)-C(9)-C(10)-C(11) | -0.50(13) |
| C(15)-C(10)-C(11)-C(12) | 0.10(14) |
| C(9)-C(10)-C(11)-C(12) | 179.60(9) |
| C(10)-C(11)-C(12)-C(13) | -0.49(15) |
| C(11)-C(12)-C(13)-O(4) | -179.62(9) |
| C(11)-C(12)-C(13)-C(14) | 0.29(15) |
| O(4)-C(13)-C(14)-C(15) | -179.80(10) |
| C(12)-C(13)-C(14)-C(15) | 0.30(15) |
| C(13)-C(14)-C(15)-C(10) | -0.70(15) |
| C(11)-C(10)-C(15)-C(14) | 0.50(15) |
| C(9)-C(10)-C(15)-C(14) | -179.01(9) |
| C(6)-C(7)-C(16)-O(1) | -82.93(11) |
| C(8)-C(7)-C(16)-O(1) | 38.69(12) |
| C(6)-C(7)-C(16)-O(2) | 94.16(9) |
| C(8)-C(7)-C(16)-O(2) | -144.21(8) |

| | |
|---|---|
| O(1)-C(16)-O(2)-C(17) | -0.66(13) |
| C(7)-C(16)-O(2)-C(17) | -177.79(8) |
| C(14)-C(13)-O(4)-C(18) | -5.41(15) |
| C(12)-C(13)-O(4)-C(18) | 174.49(9) |

Symmetry transformations used to generate equivalent atoms:

[1] Trost, B. M.; Braslau, R. *J. Org. Chem.* **1988**, *53*, 532-537.

[2] The $^{1}$H NMR data are consistent with those reported in the literature, Zhao, W.-J.; Yan, M.; Huang, D.; Ji, S.-J. *Tetrahedron* **2005**, *61*, 5585-5593.

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