

H₃PW₁₂O₄₀ Anchored on the Three-Dimensional and Networked SBA-15 as an Efficient and Recyclable Catalyst for Mannich Reaction

Tengfei Zhang, Wei Zhang, Hao Dong, Qing Liu*

Key Laboratory of Low Carbon Energy and Chemical Engineering, College of Chemical and Environmental Engineering, Shandong University of Science and Technology, Qingdao Shandong 266590, China.

***Corresponding author:** Qing Liu, email: gliu@sdu.edu.cn

Received July 22th, 2019; Accepted August 21th, 2019.

DOI for the article: <http://dx.doi.org/10.29356/jmcs.v64i1.1034>

Supplementary Information

(Compound 1) [1]**3-(4-acetylphenyl)-1-phenyl-3-(phenylamino)propan-1-one**

Mp 142-143 °C. ^1H NMR (60 MHz,) δ 7.96 (s, 2H, AR-H), 7.83 (s, 2H, AR-H), 7.43 (s, 3H, AR-H), 7.29 (s, 2H, AR-H), 6.98 (s, 2H, AR-H), 6.79 (s, 1H, AR-H), 6.65 (s, 2H, AR-H, C-H), 5.07 (s, 1H), 3.41 (s, 3H, CH_3) ; IR (KBr) ν (cm $^{-1}$): 694, 757, 1028, 1181, 1288, 1513, 1595, 1667, 3369.

(Compound 2) [2]**1,3-diphenyl-3-(phenylamino)propan-1-one**

Mp 169-170 °C. ^1H NMR (60 MHz,) δ 8.11 (s, 2H, AR-H), 7.66 (s, 3H, AR-H), 7.51 (d, J = 3.1 Hz, 4H, AR-H), 7.41 (s, 2H, AR-H), 7.14 (s, 2H, AR-H), 6.92 (s, 1H, AR-H), 6.80 (s, 2H, AR-H), 6.67 (s, 1H, AR-H), 5.16 (s, 1H, C-H), 3.69 (s, 2H, CH_2) ; IR (KBr) ν (cm $^{-1}$): 622, 1397, 1622, 3233, 3423.

(Compound 3) [3]**2-(phenyl(phenylamino)methyl)cyclohexanone**

Mp 114-115 °C. ^1H NMR (60 MHz,) δ 7.49 (s, 5H, AR-H), 7.29 (s, 2H, AR-H), 6.83 (s, 3H, AR-H), 4.74 (s, 1H, C-H), 3.08 (s, 1H, C-H), 2.59 (s, 2H, CH_2), 2.15 (s, 1H, CH_2), 2.01 (s, 3H, CH_2), 1.73 (s, 2H, CH_2) ; IR (KBr) ν (cm $^{-1}$): 613, 1397, 1622, 3233, 3423.

(Compound 4) [4]**2-((*p*-toluidino)(phenyl)methyl)cyclohexanone**

Mp 118-119 °C. ^1H NMR (60 MHz,) δ 7.61 (s, 4H, AR-H), 7.14 (s, 1H, AR-H), 6.86 (s, 4H, AR-H), 4.85 (s, 1H, C-H), 3.12 (s, 1H, C-H), 2.68 (s, 3H, CH_3), 2.45 (s, 3H, CH_2), 2.11 (s, 5H, CH_2) ; IR (KBr) ν (cm $^{-1}$): 694, 793, 1118, 1316, 1450, 1523, 1622, 1702, 2855, 2927, 3405.

(Compound 5)**3-(*p*-toluidino)-1-phenyl-3-*p*-tolylpropan-1-one**

Mp 135-136 °C. ^1H NMR (60 MHz,) δ 8.62 (s, 2H, AR-H), 8.06 (s, 3H, AR-H), 7.68 (s, 2H, AR-H), 7.45 (s, 2H, AR-H), 7.36 (s, 4H, AR-H), 5.12 (s, 1H, C-H), 3.74 (s, 1H, C-H), 2.56 (s, 6H, CH_3) ; IR (KBr) ν (cm $^{-1}$): 532, 820, 1108, 1397, 1622, 3450.

(Compound 6) [2]**1-phenyl-3-(phenylamino)-3-*p*-tolylpropan-1-one**

Mp 134-135 °C. ^1H NMR (60 MHz,) δ 8.35 (s, 2H, AR-H), 7.92 (s, 2H, AR-H), 7.77 (s, 2H, AR-H), 7.68 (s, 2H, AR-H), 7.49 (s, 2H, AR-H), 7.15 (s, 2H, AR-H), 7.01 (s, 1H, AR-H), 5.38 (s, 1H, C-H), 4.02 (s, 1H, CH_2), 3.91 (s, 1H, CH_2), 2.81 (s, 3H, CH_3) ; IR (KBr) ν (cm $^{-1}$): 694, 748, 820, 992, 1288, 1513, 1604, 1667, 3387.

(Compound 7) [2]**3-(*p*-toluidino)-1,3-diphenylpropan-1-one**

Mp 167-168 °C. ^1H NMR (60 MHz,) δ 8.12 (s, 2H, AR-H), 7.62 (s, 2H, AR-H), 7.55 (s, 2H, AR-H), 6.84 (s, 2H, AR-H), 6.71 (s, 2H, AR-H), 5.26 (s, 1H, C-H), 3.81 (s, 1H, CH₂), 3.63 (s, 1H, CH₂), 2.46 (s, 3H, CH₃); IR (KBr) ν (cm⁻¹): 694, 793, 1280, 1523, 1612, 1675, 3395.

(Compound 8) [3]**3-(4-chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one**

Mp 114-115 °C. ^1H NMR (60 MHz,) δ 8.22 (s, 2H, AR-H), 8.12 (s, 1H, AR-H), 7.78 (s, 2H, AR-H), 7.59 (s, 2H, AR-H), 7.07 (s, 2H, AR-H), 6.89 (s, 2H, AR-H), 6.77 (s, 1H, AR-H), 5.24 (s, 1H, C-H), 3.79 (s, 1H, CH₂), 3.69 (s, 1H, CH₂); IR (KBr) ν (cm⁻¹): 616, 690, 745, 824, 1219, 1288, 1395, 1488, 1507, 3387.

(Compound 9)**3-(*p*-toluidino)-3-(4-chlorophenyl)-1-phenylpropan-1-one**

Mp 128-129 °C. ^1H NMR (60 MHz,) δ 8.22 (s, 2H, AR-H), 7.95 (s, 1H, AR-H), 7.86 (s, 2H, AR-H), 7.44 (s, 2H, AR-H), 7.12 (s, 2H, AR-H), 6.67 (s, 2H, AR-H), 5.22 (s, 1H, C-H), 3.77 (s, 1H, CH₂), 3.67 (s, 1H, CH₂), 2.46 (s, 3H, CH₃); IR (KBr) ν (cm⁻¹): 685, 803, 1091, 1217, 1288, 1513, 3450.

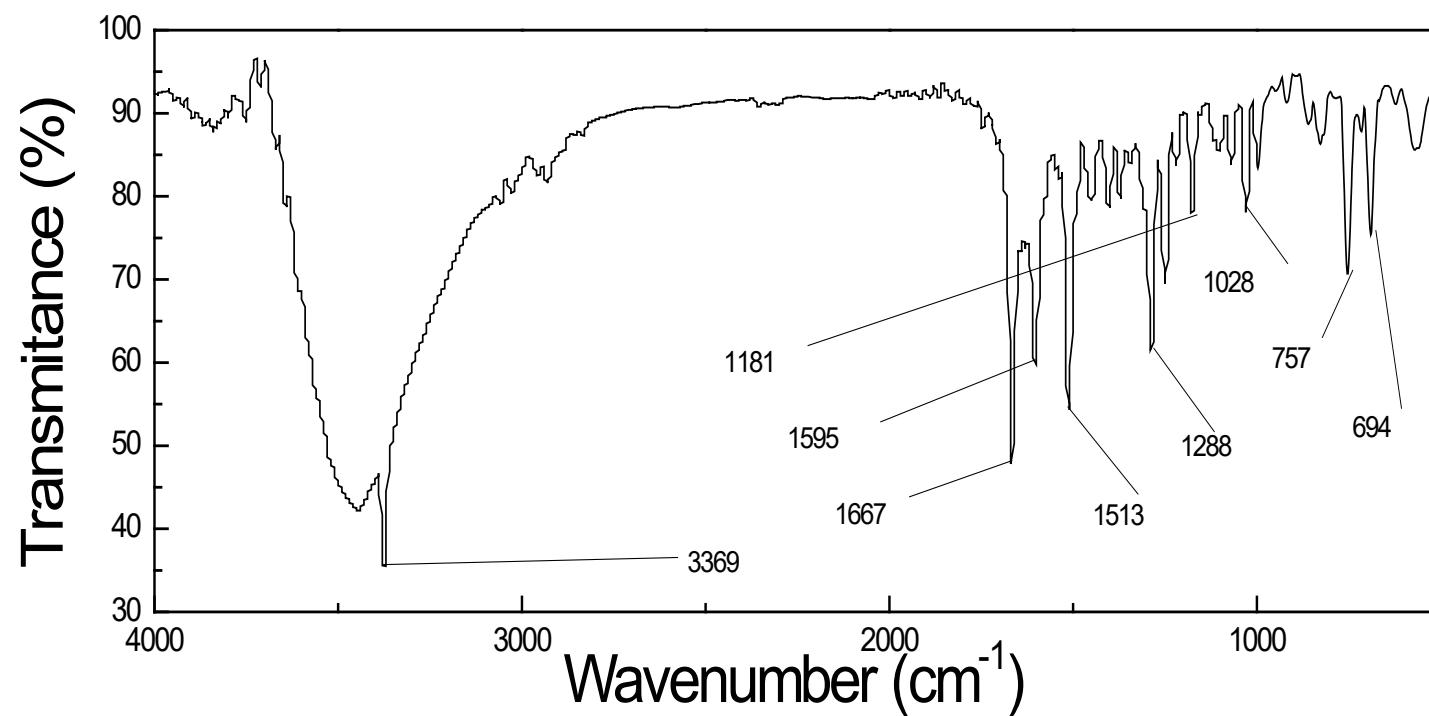
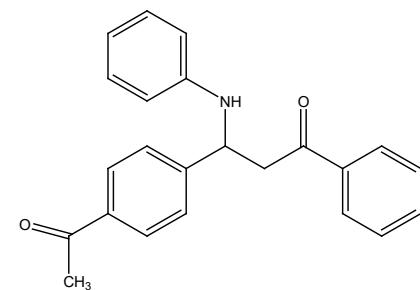
(Compound 10)**3-(*p*-toluidino)-3-(4-acetylphenyl)-1-phenylpropan-1-one**

Mp 152-154 °C. ^1H NMR (60 MHz,) δ 8.50 (s, 4H, AR-H), 7.90 (s, 3H, AR-H), 7.52 (s, 2H, AR-H), 6.91 (d, $J = 5.4$ Hz, 2H, AR-H), 6.52 (s, 2H, AR-H), 5.13 (s, 1H, C-H), 3.97 (s, 2H, CH₂), 3.48 (s, 3H, CH₃), 2.28 (s, 3H, CH₃); IR (KBr) ν (cm⁻¹): 804, 1245, 1288, 1400, 1513, 1622, 1667, 3450.

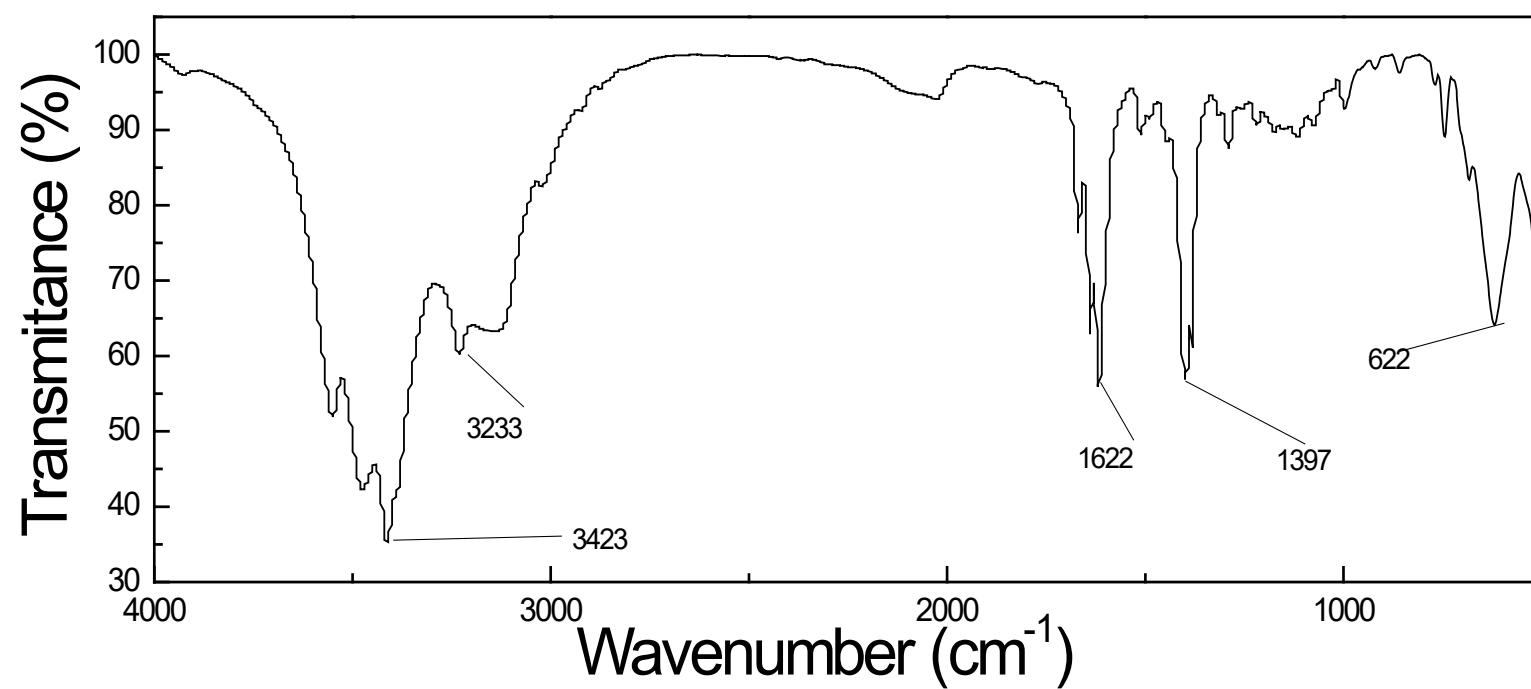
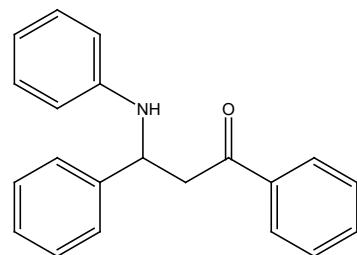
(Compound 11) [4]**2-((*p*-toluidino)(4-chlorophenyl)methyl)cyclohexanone**

Mp 120-121 °C. ^1H NMR (60 MHz,) δ 7.82 (s, 2H, AR-H), 7.39 (s, 2H, AR-H), 6.87 (s, 2H, AR-H), 6.54 (s, 2H, AR-H), 4.68 (s, 1H, C-H), 2.83 (s, 1H, C-H), 2.42 (s, 2H, CH₂), 2.02 (s, 3H, CH₃), 1.84 (s, 5H, CH₂); IR (KBr) ν (cm⁻¹): 1091, 1502, 1525, 1635, 3457.

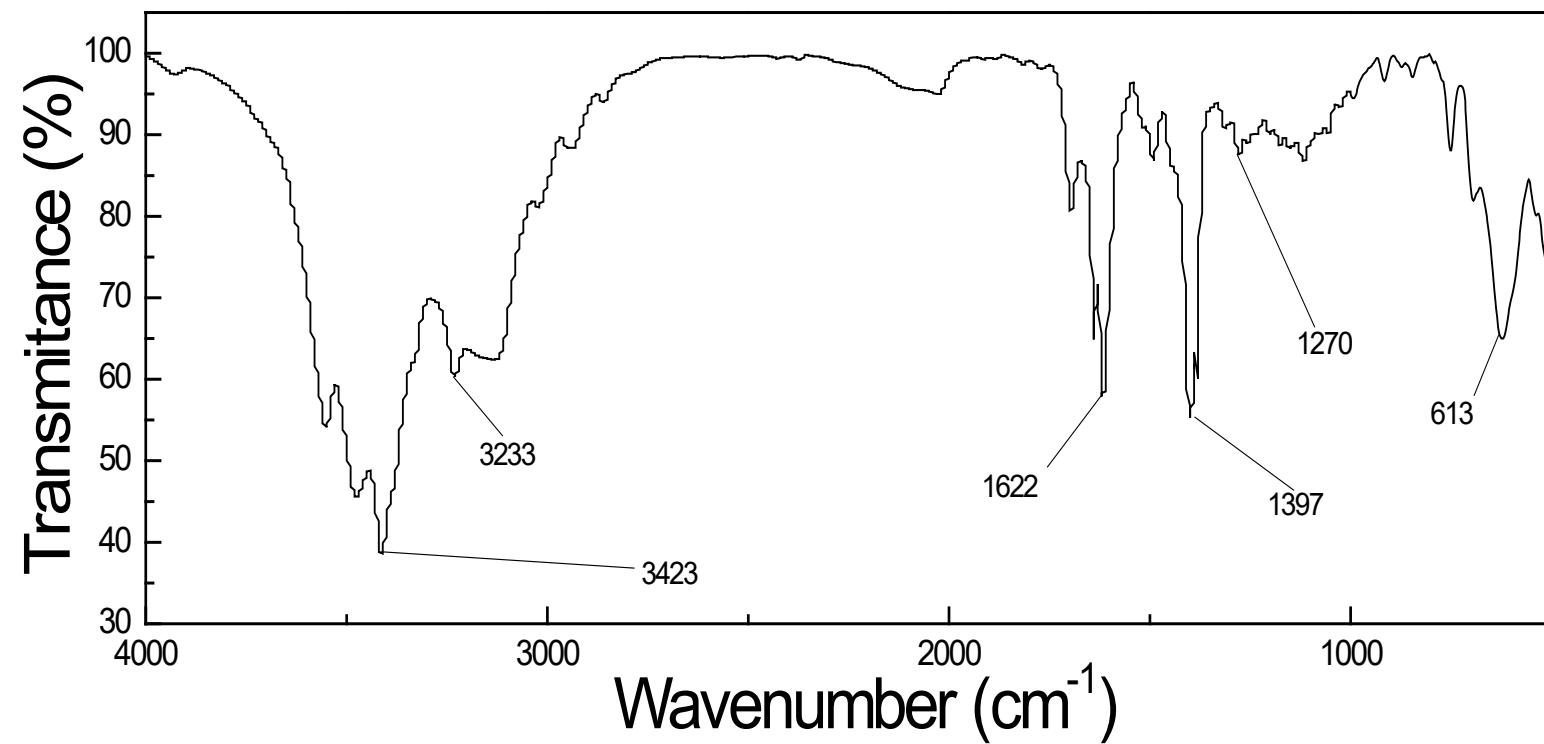
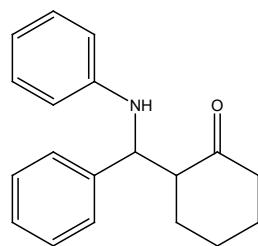
Compound 1



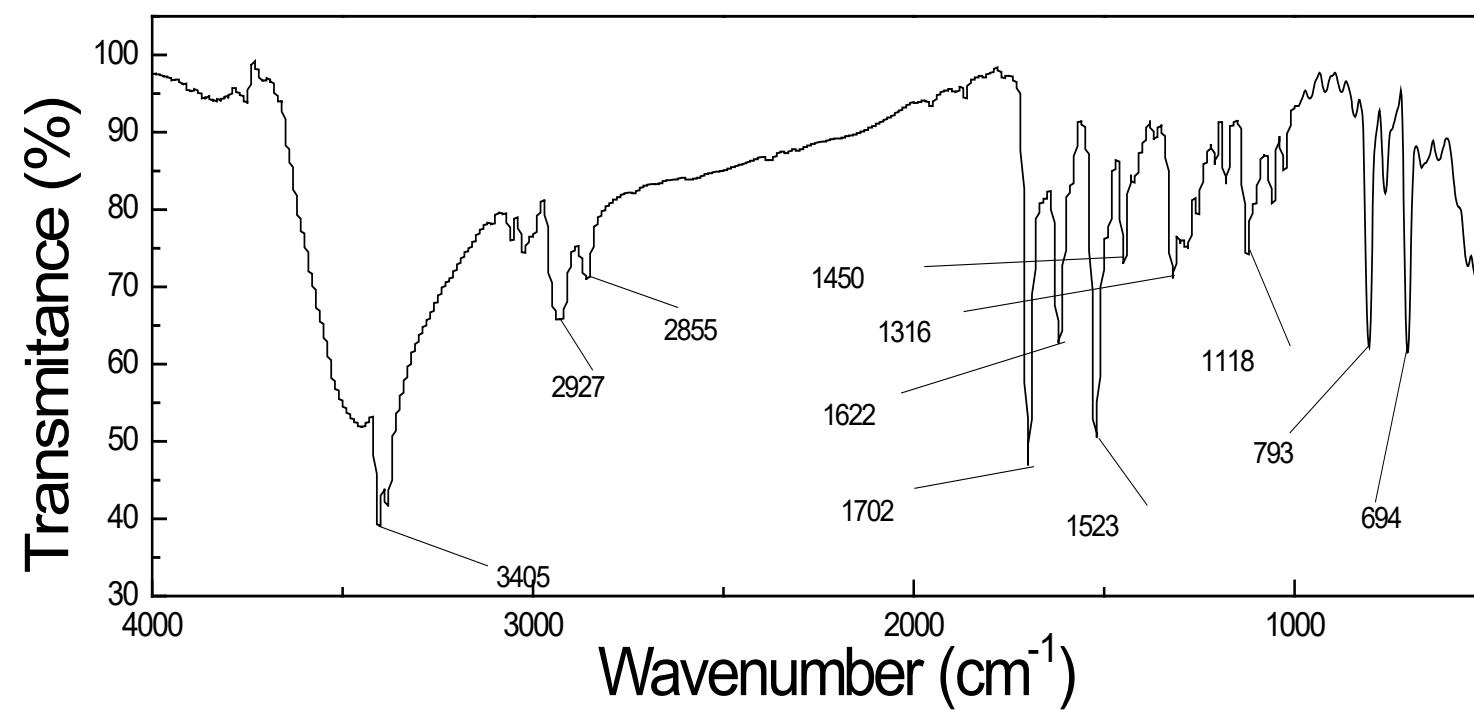
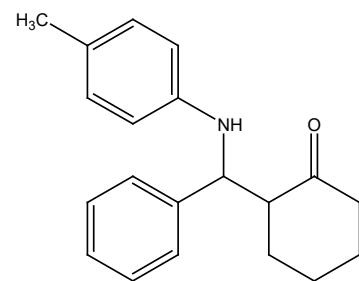
Compound 2



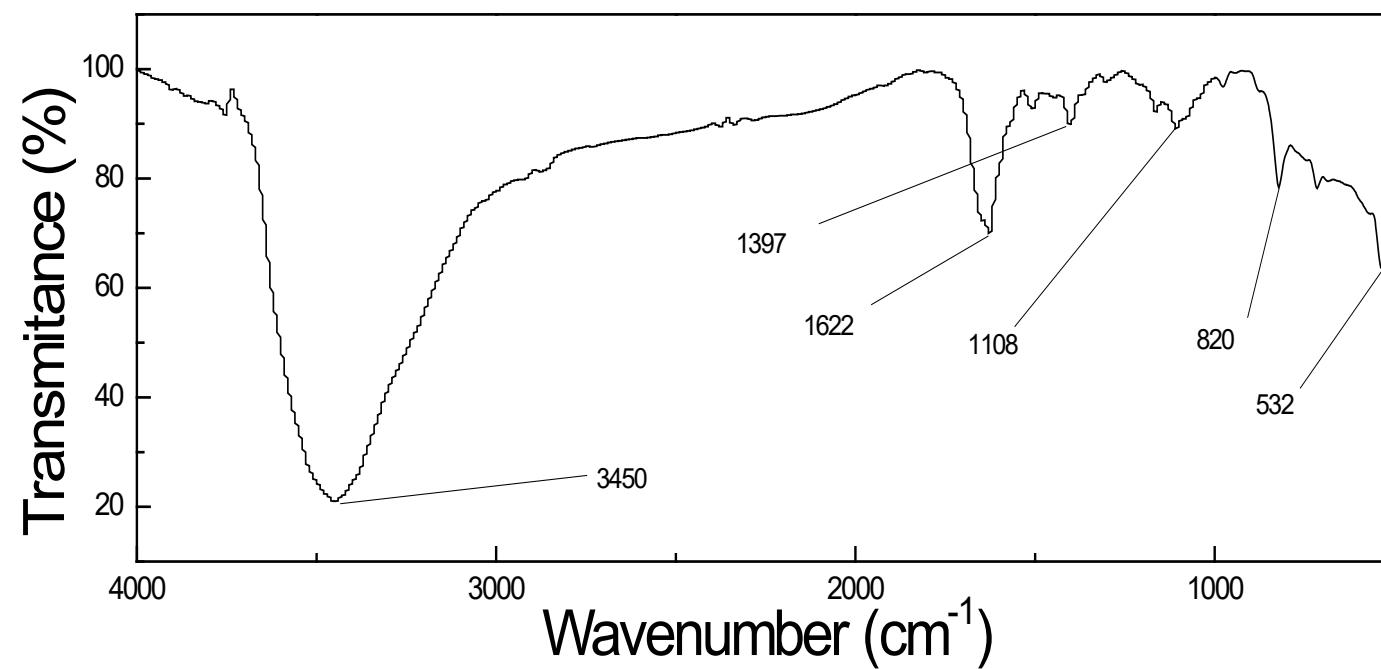
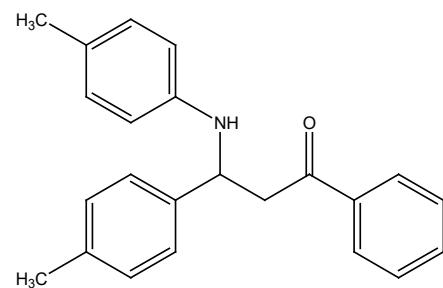
Compound 3



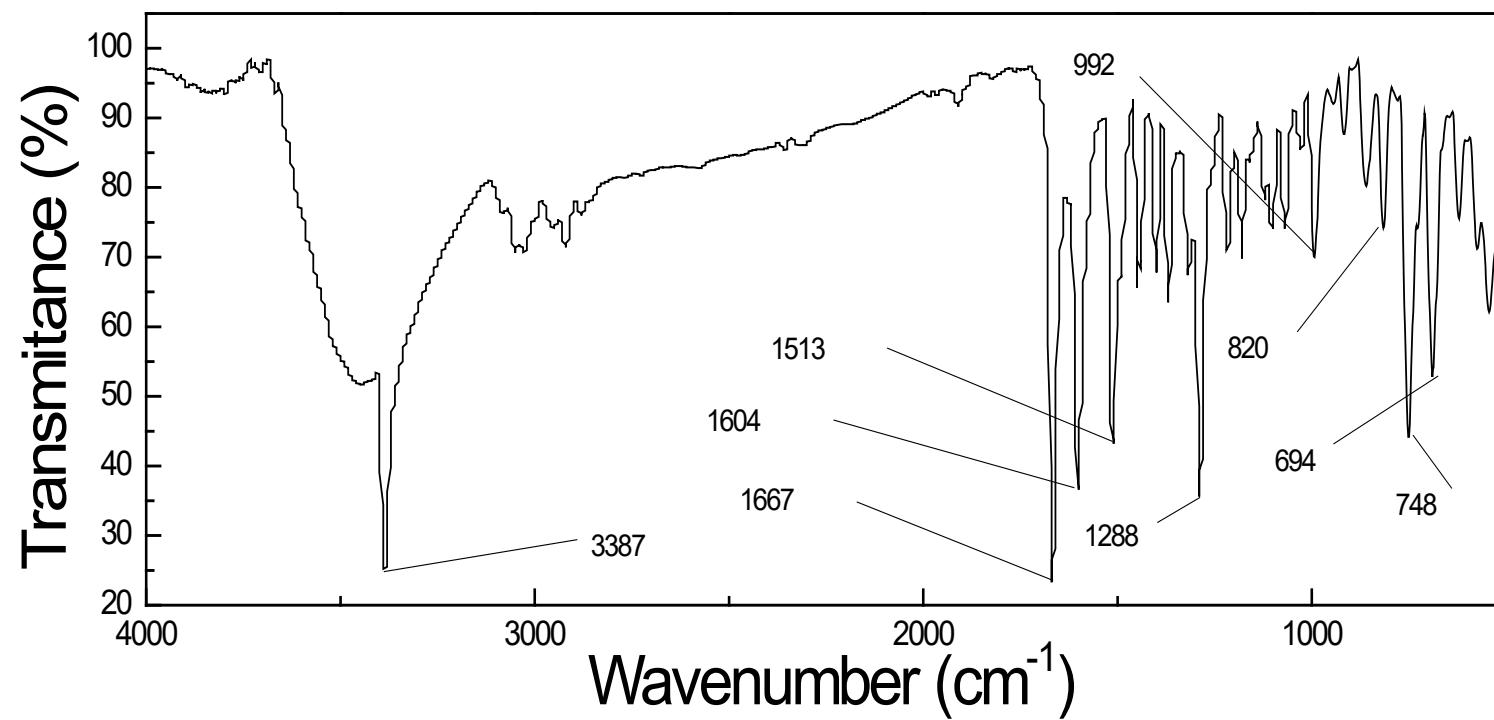
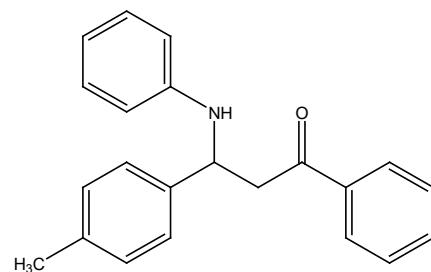
Compound 4



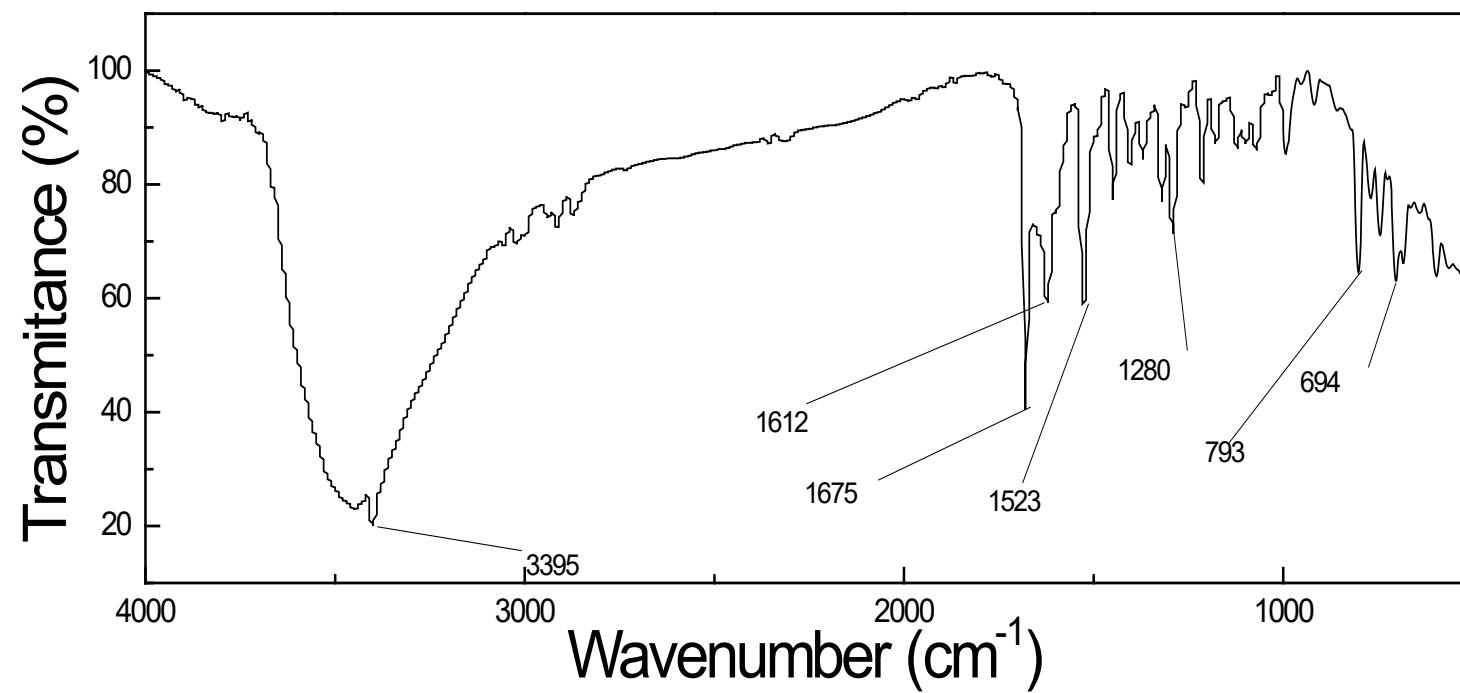
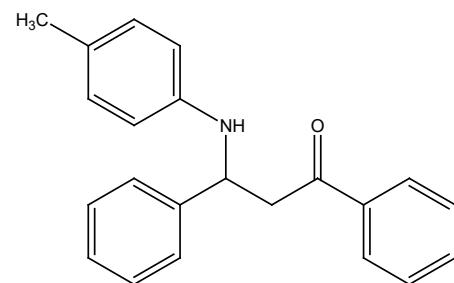
Compound 5



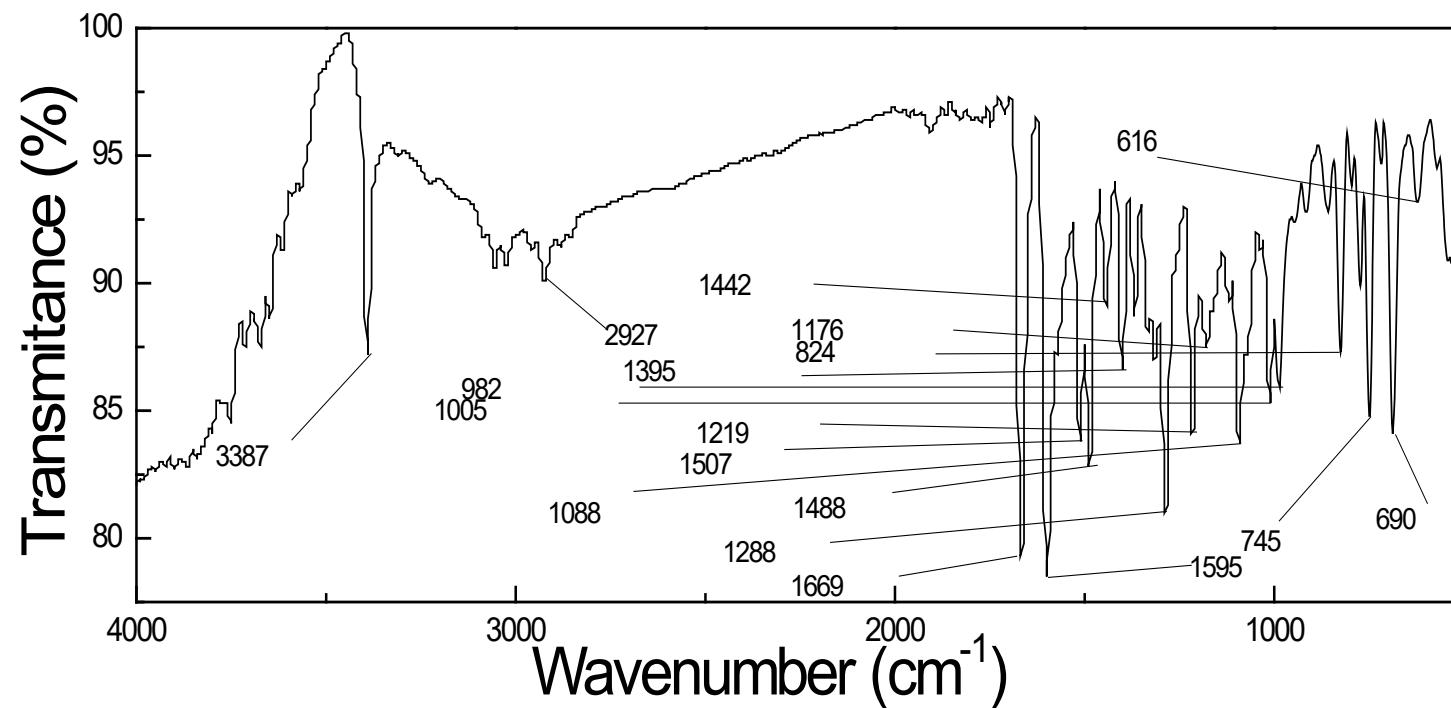
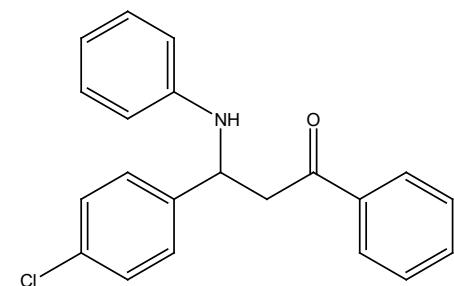
Compound 6



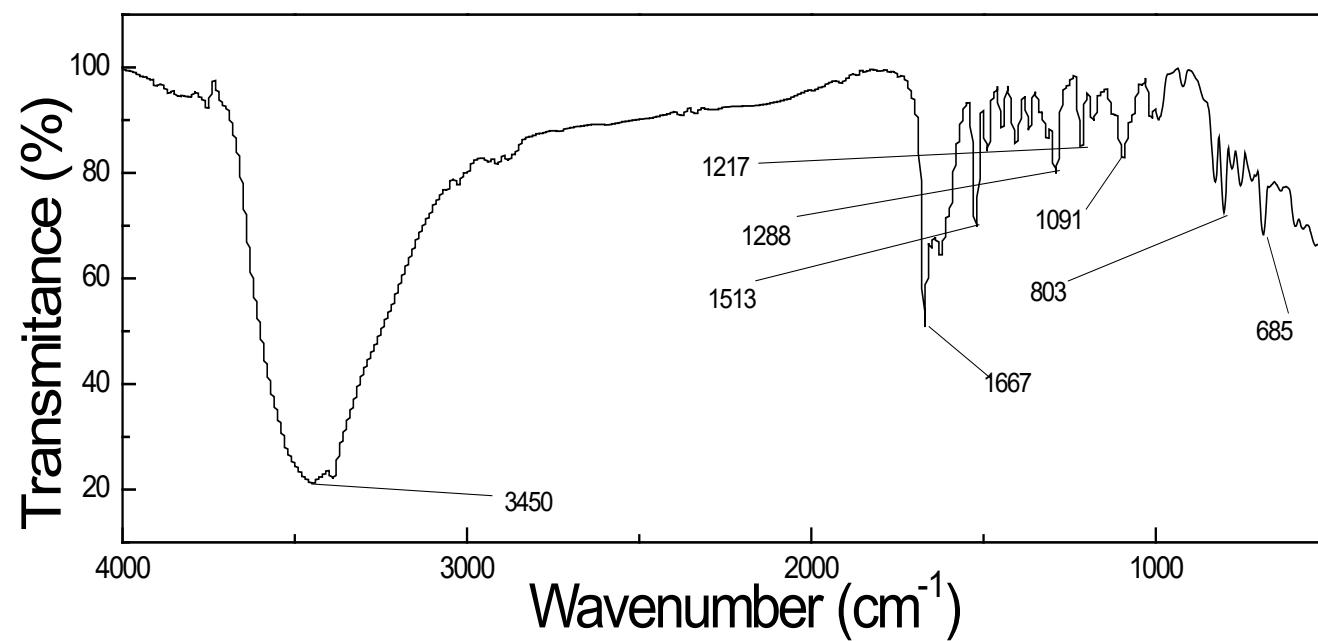
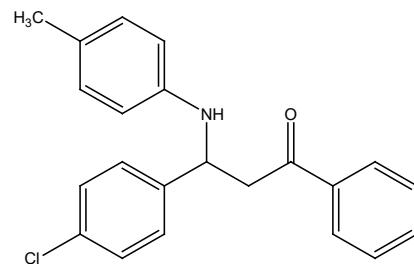
Compound 7



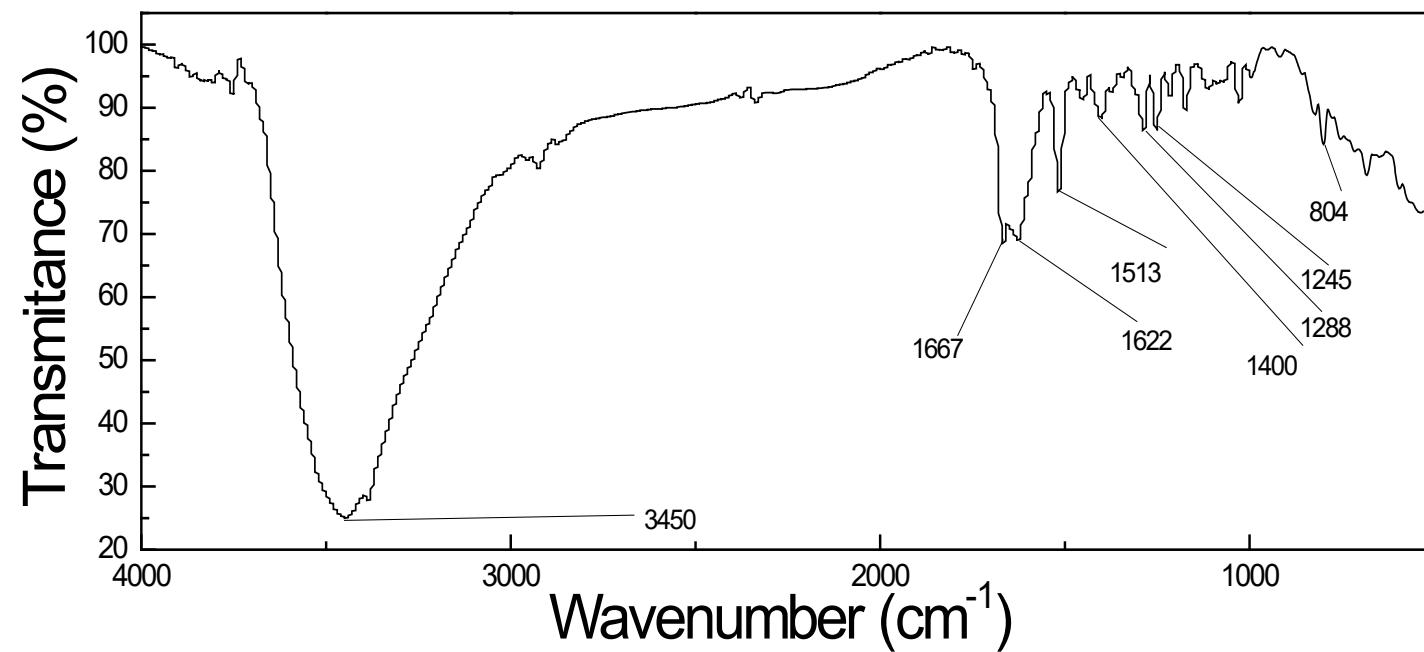
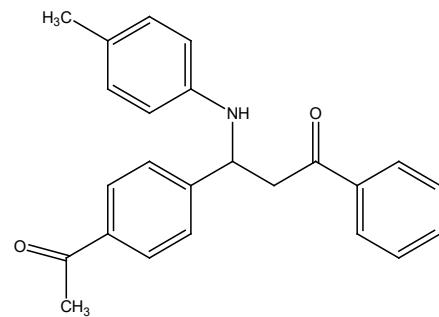
Compound 8



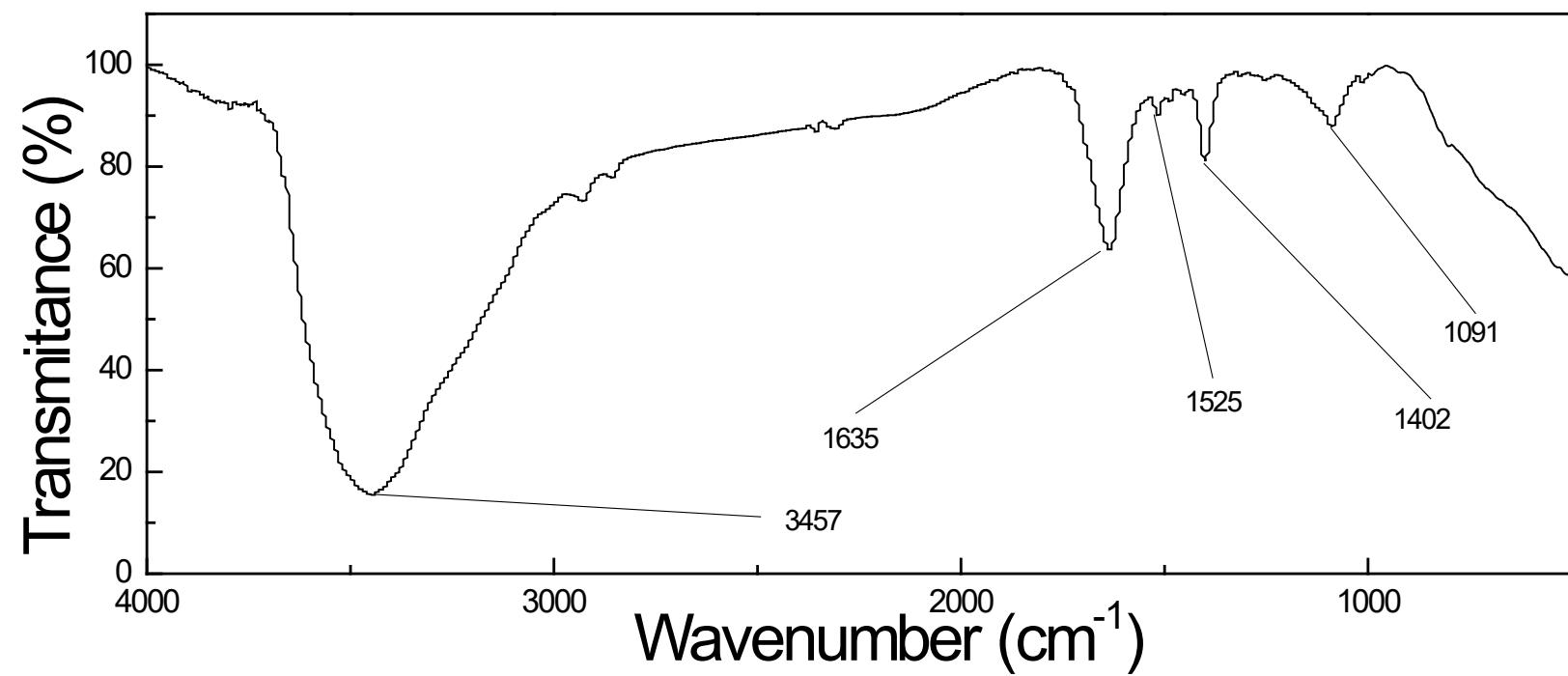
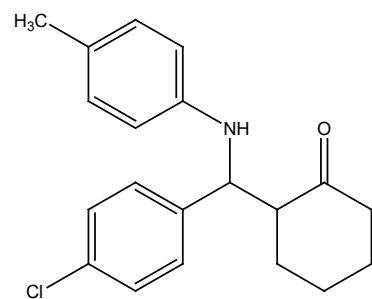
Compound 9

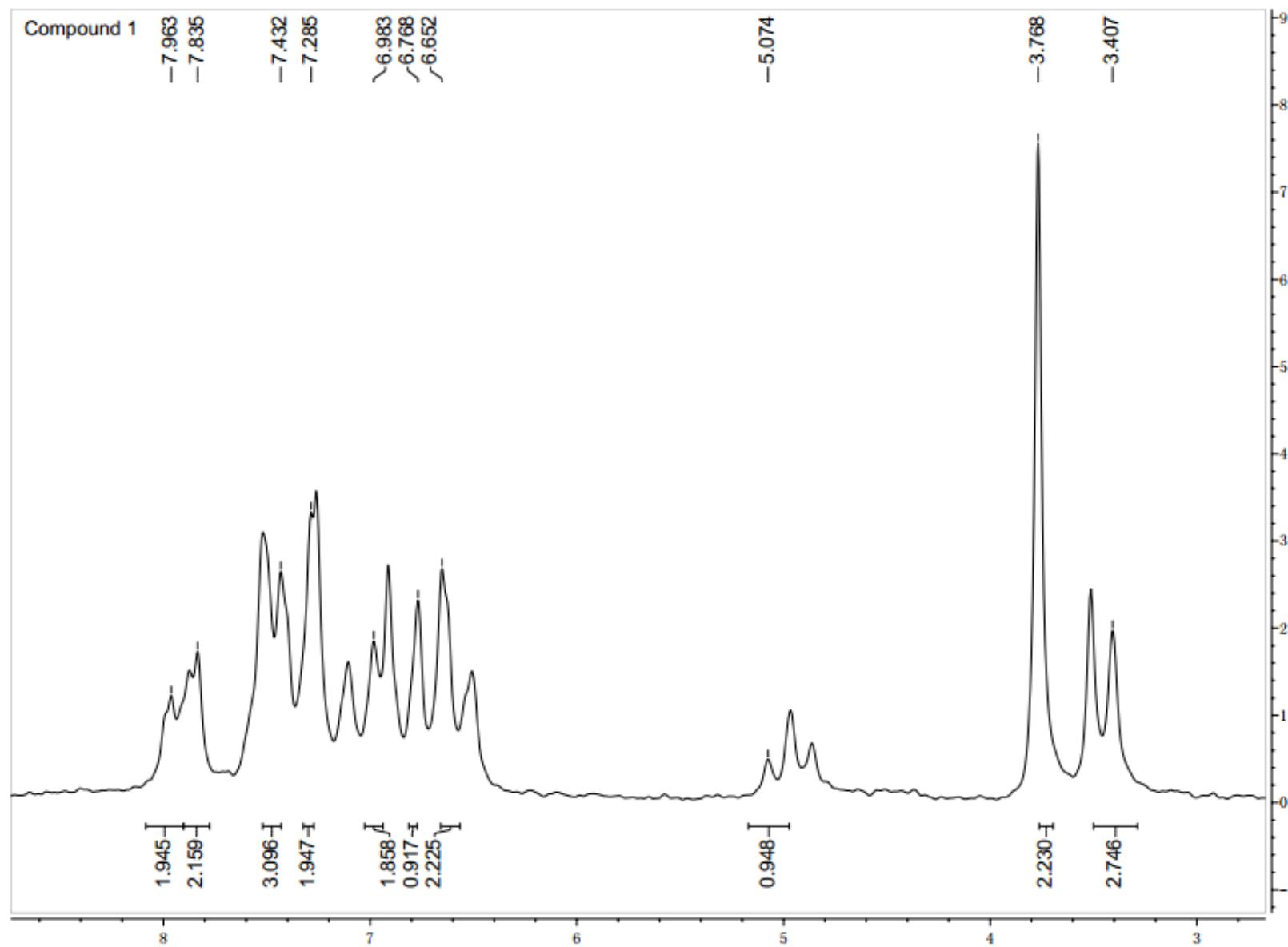


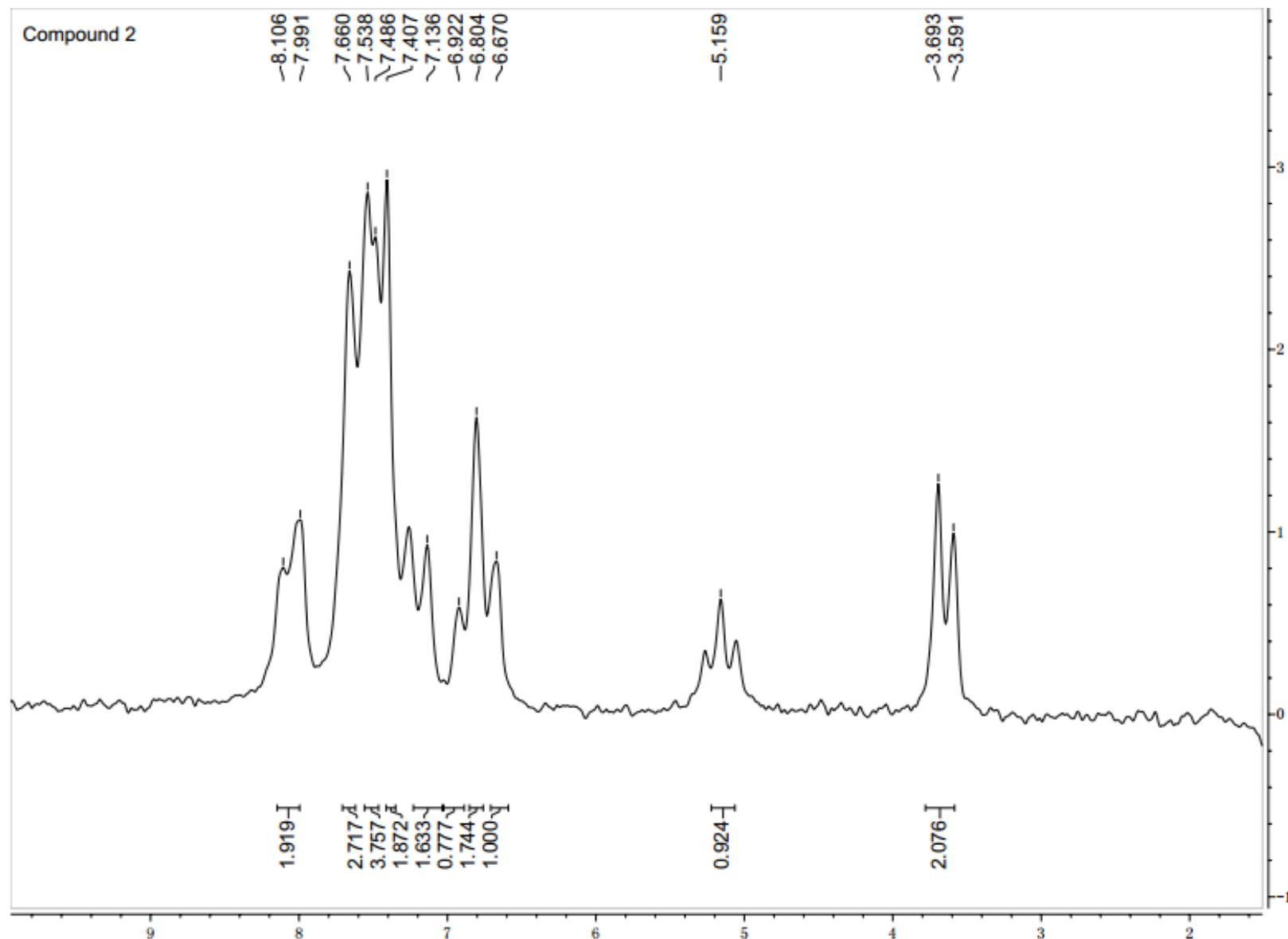
Compound 10

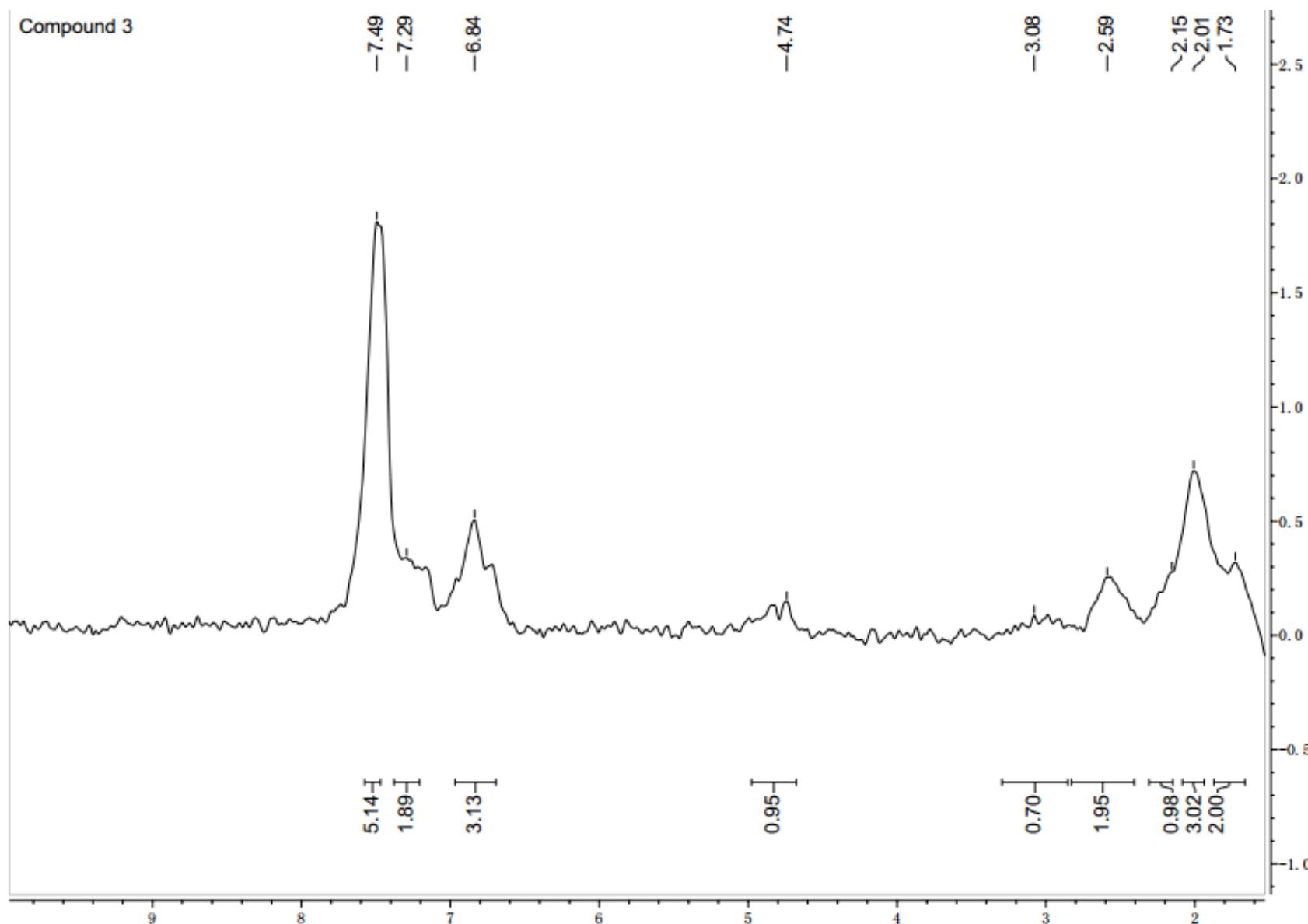


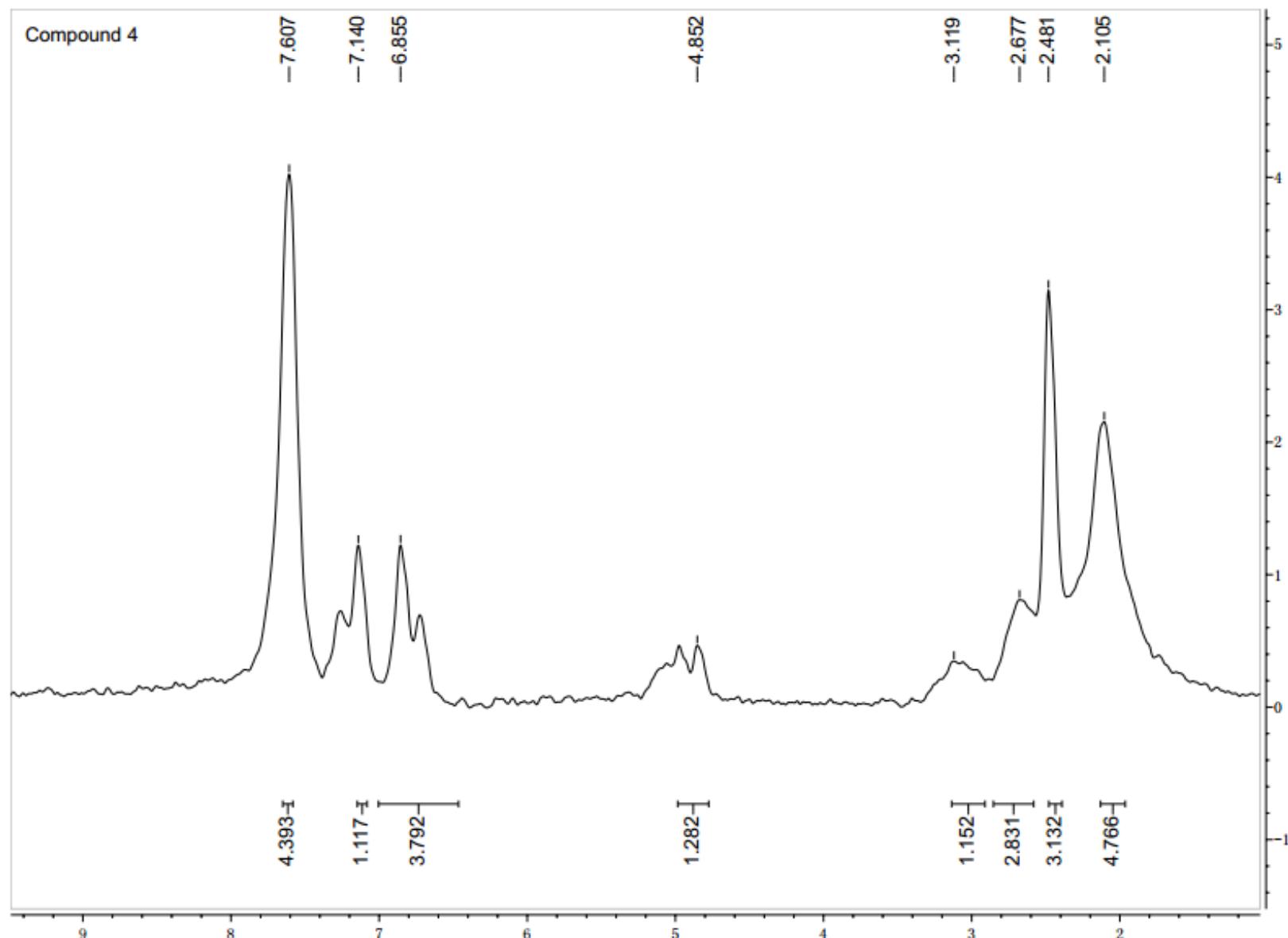
Compound 11

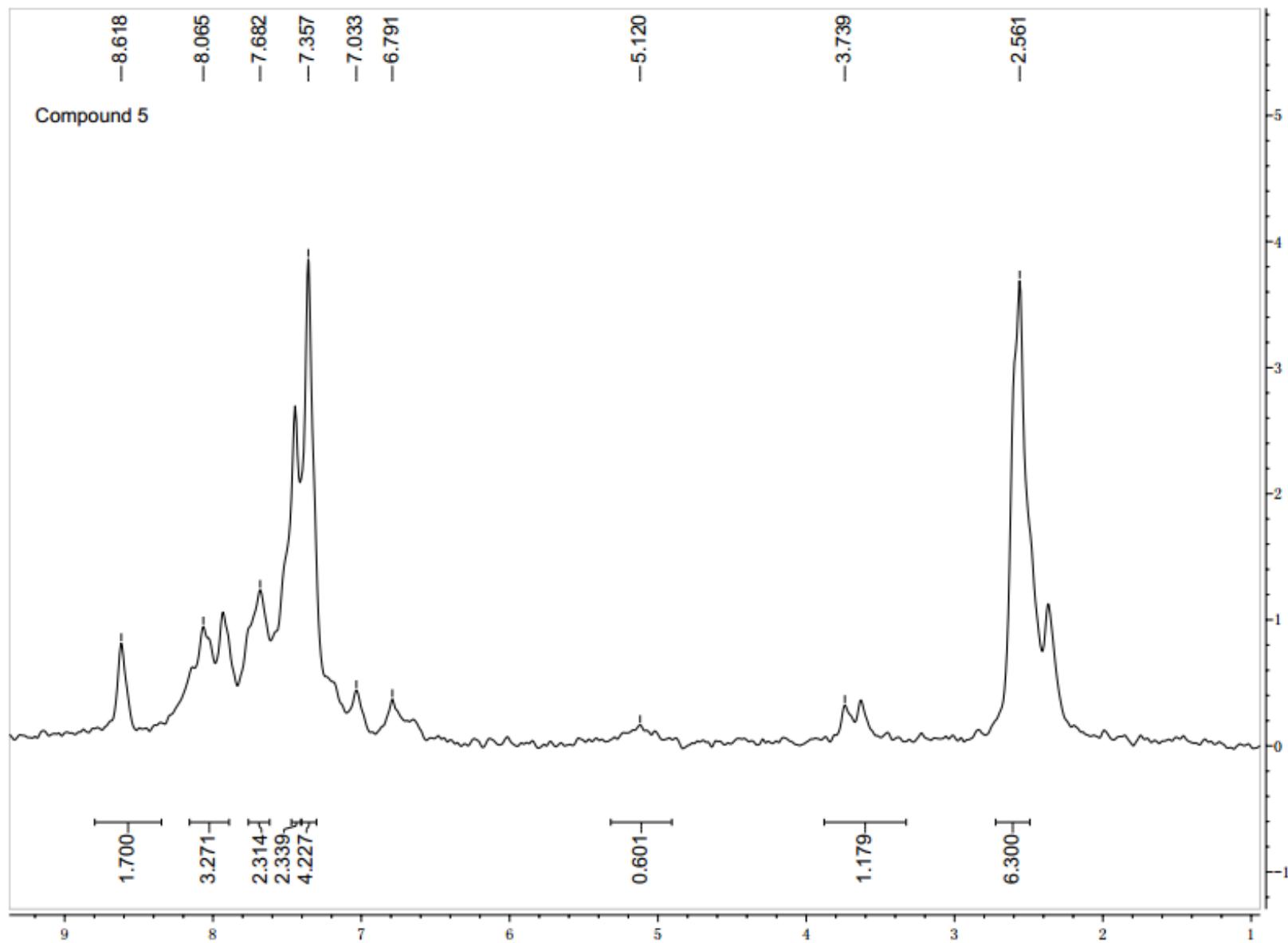


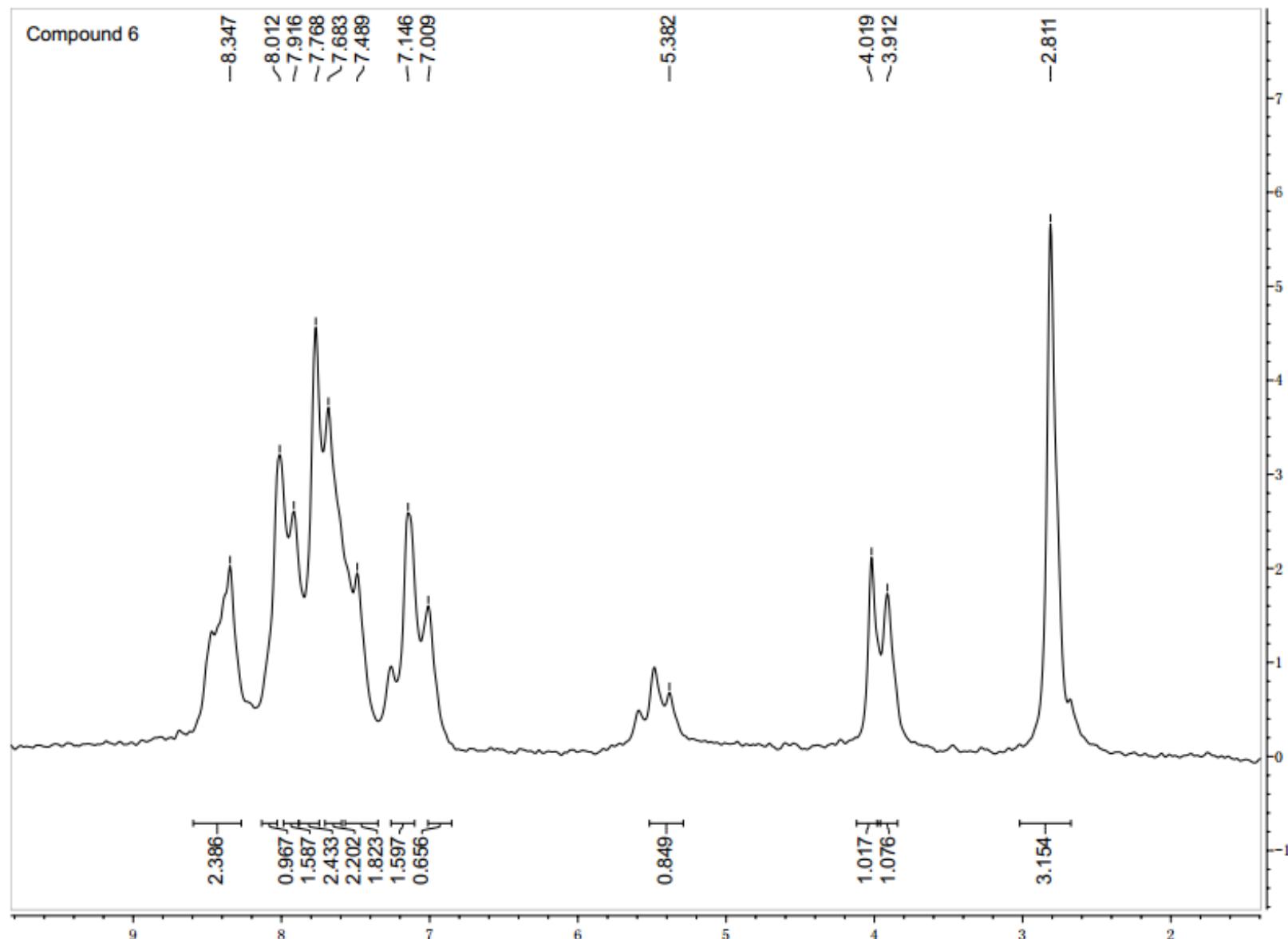


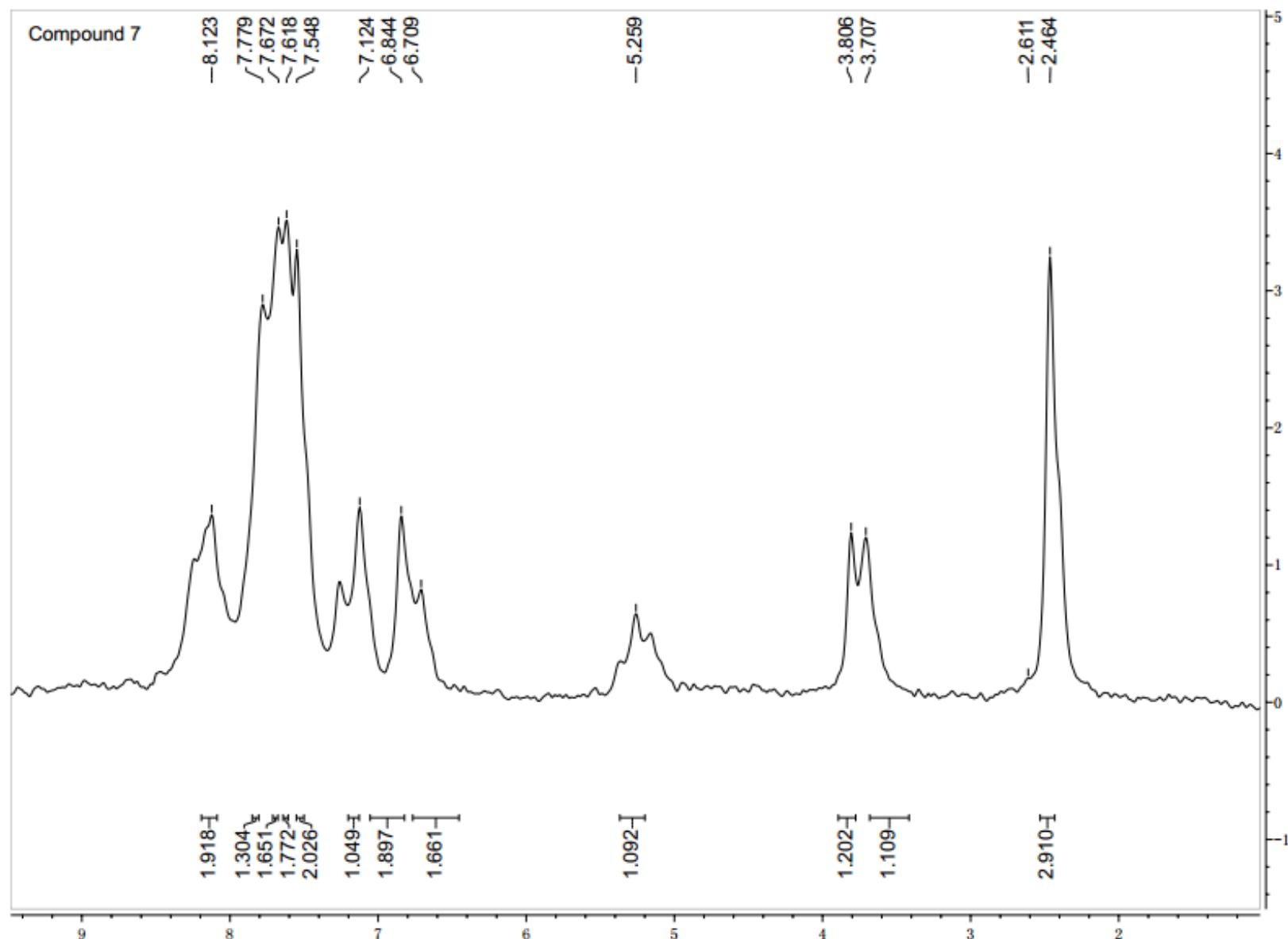


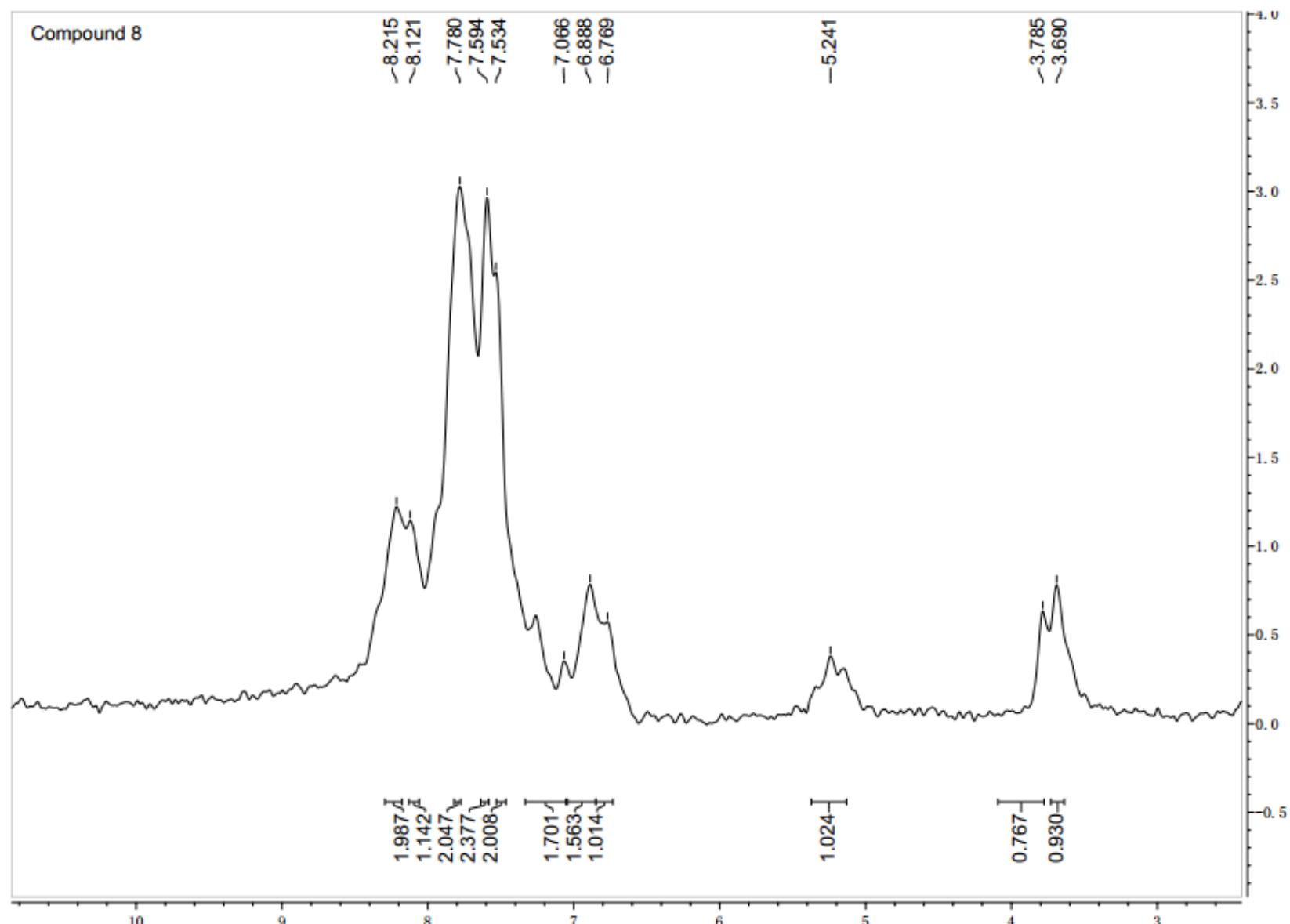


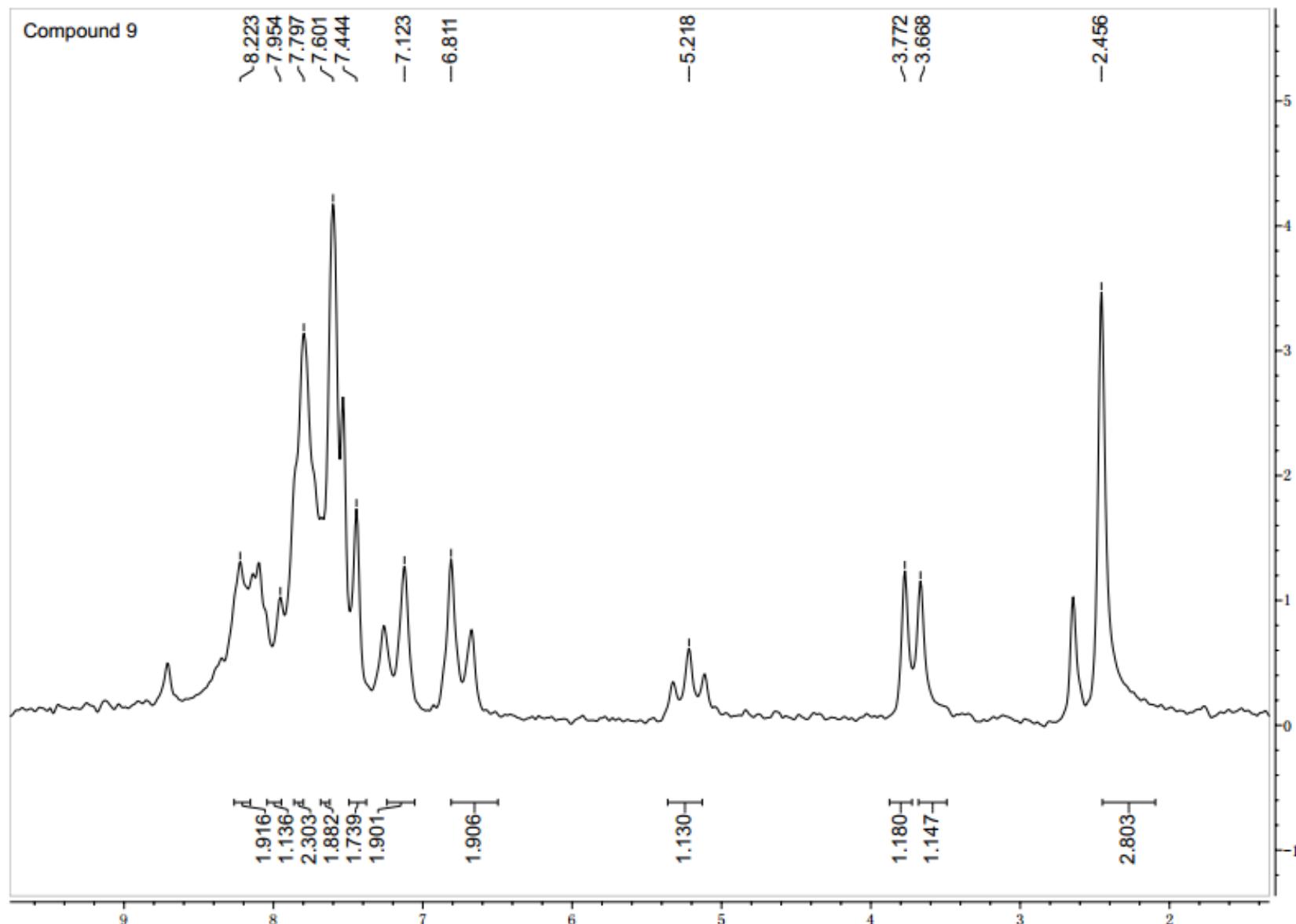


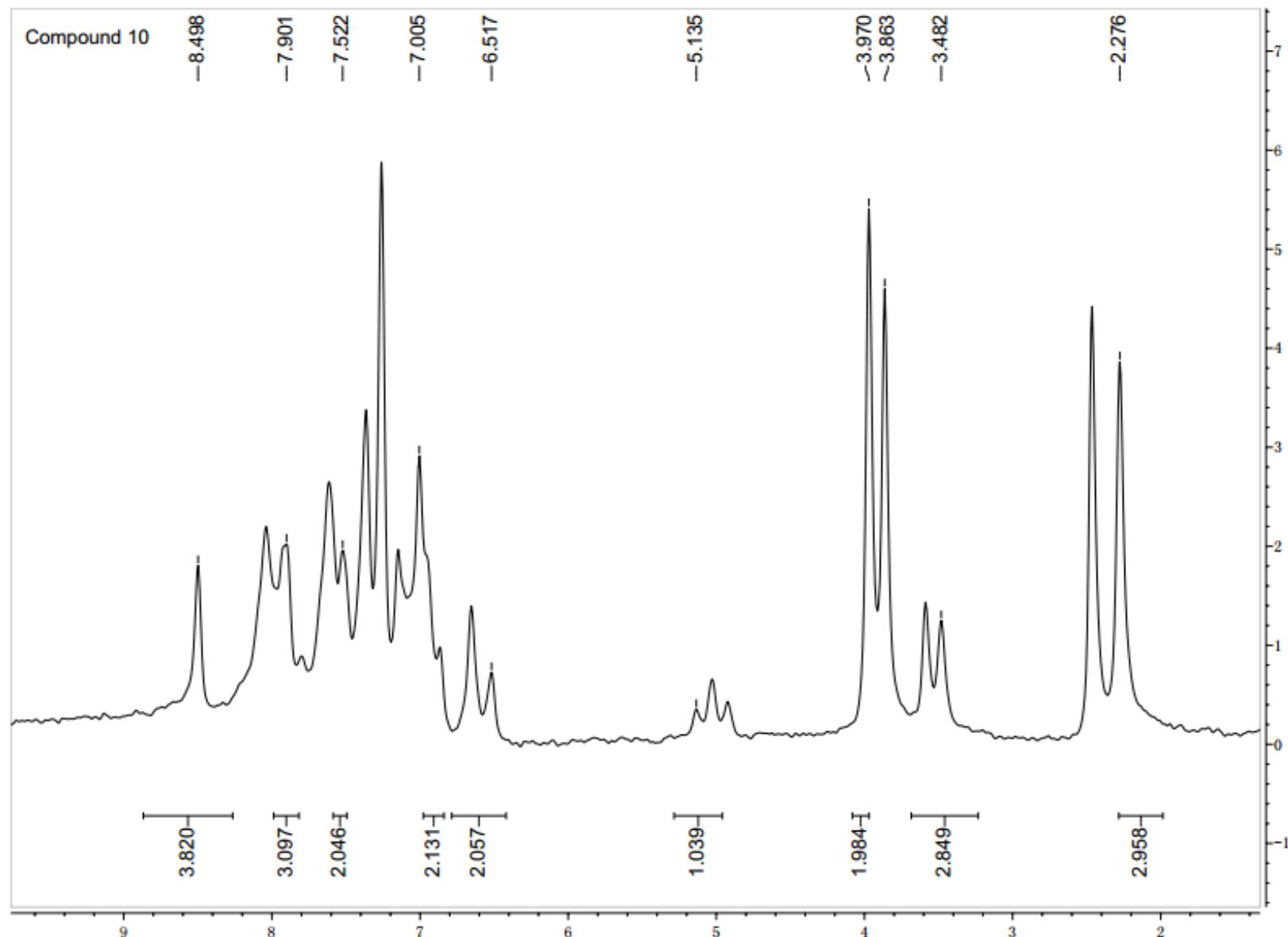


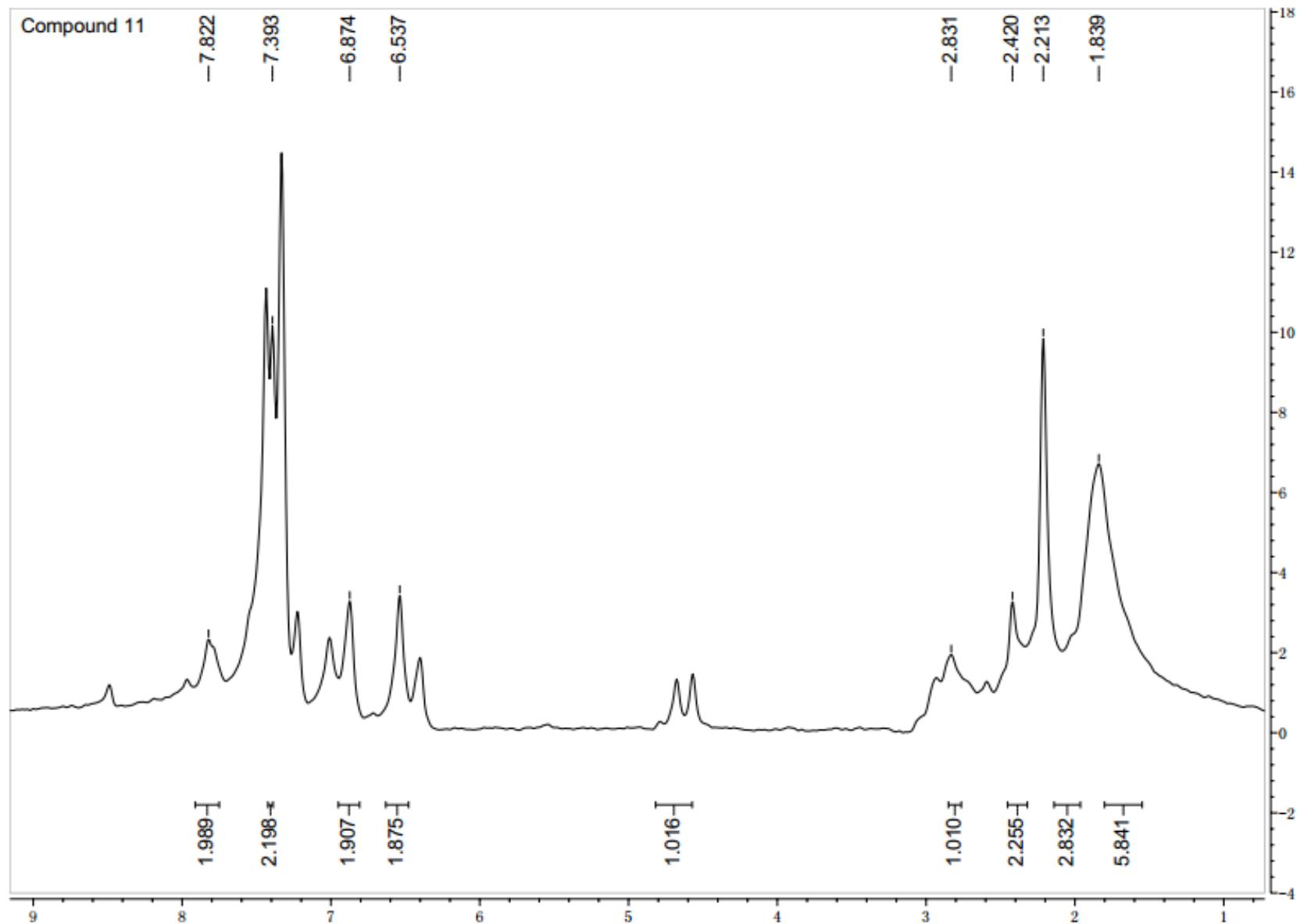












References

1. Rafiee, E.; Eavani, S. *Green Chem.* **2011**, *13*, 8.
2. Wang, R.; Li, B.G.; Huang, T.K.; L. Shi, L.; Lu, X.X. *Tetrahedron Lett.* **2007**, *48*, 12.
3. Najmodin Azizi, L.T.; Mohammad R. Saidi, *Org. Lett.* **2006**, 8.
4. Kooti, M.; Kooshki, F.; Nasiri, E.; Sede, A.N. *J. Iran. Chem. Soc.* **2018**, *15*, 4.