Supporting Information

Practical method to obtain α-acetoxyketones promoted by (diacetoxyiodo)benzene and acetic acid

Osvaldo J. Quintana-Romero, Jorge Camacho-Ruiz, Armando Ariza-Castolo
Departamento de Química, Centro de Investigación y Estudios Avanzados del Instituto Politécnico Nacional, Av. IPN 2508, San Pedro Zacatenco, 07360, Ciudad de México.

Content:

Spectra determination

The NMR spectra were recorded using ECA-500 MHz JEOL spectrometers (^{1}H, 500.159 MHz; ^{13}C, 125.76 MHz). The unified scale[1] was used as a primary reference based on the ^{1}H resonance of TMS in a dilute solution (volume fraction $\varphi < 1\%$) in chloroform, $(CH_3)_4Si$ ($\delta\,^{1}$H, $\delta\,^{13}$C = 0).

The compounds were assigned using the pfg-COSY, pfg-HMBC and pfg-HSQC pulse sequences.[2]

The mass spectra of the compounds were determined in an Agilent 1200 Series SQ spectrometer coupled to HPLC with electrospray ionization.

NMR and MS data

1-Acetoxyacetophenone (2): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.93 (2H, d, J = 7.4 Hz), 7.62 (1H, t, J = 7.4 Hz), 7.50 (2H, t, J = 7.4 Hz), 5.35 (2H, s), 2.25 (3H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 192.27, 170.58, 134.29, 134.04, 128,99, 127.89, 66.15, 20.71. MS ES-API *m/z*: M^+ 179.1. The spectral data are in accordance with the previous reports.[3,4]

α-Tosylacetophenone (3): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.82 (2H, d, J = 8.5 Hz), 7.81 (2H, dd, J = 8.1, 1.1 Hz), 7.58 (1H, t,t, J = 7.4, 1.1 Hz), 7.44 (2H, dd, J = 8.1, 7.44 Hz), 7.31 (2H, d, J = 8.5 Hz), 5.25 (2H, s), 2.41 (3H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 190.44, 145.44, 134.31, 133.81, 132.68, 130.03, 129.01, 128.21, 128.06, 70.10, 21.76. Spectroscopic properties are consistence to report.[5]

2,4-Dioxopentan-3-yl Acetate (5): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.42 (1H, s), 2.22 (6H, s), 2.18 (3H, s); enol tautomer 14.4 (br, 1H), 2.20 (s, 3H), 2.19 (s, 6H). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 199.14, 169.32, 85.12, 27.33, 20.48; enol tautomer 184.78, 169.40, 128.20. ES-API *m/z*: M^+ 159.1. Spectroscopic properties are in agreement with the report.[6,7]

1-acetoxy-2,5-hexanodiona (7): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.66 (2H, s), 2.73 (2H, t, J= 6.7 Hz), 2.60 (2H, t, J= 6.7 Hz), 2.12 (3H, s), 2.10 (3H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 206.82, 202.29, 170.35, 68.06, 36.65, 32.29, 29.77, 20.49. ES-API *m/z*: M^+ 173.1. Previously reported.[8]

1-acetyloxy-2-butanone (10): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.43 (1H, s), 2.43 (2H, q, J = 7.4 Hz), 2.15 (3H, s), 1.08 (3H, t, J = 7.42 Hz). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 204.53, 170.40, 67.82, 32.14, 20.58, 7.22. ES-API m/z: M+ 131.0. Reported.[9]

3-acetyloxy-2-butanone (11): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.19 (1H, q, J = 7.1 Hz), 2.15 (3H, s), 2.12 (3H, s), 1.43 (3H, d, J = 7.1 Hz). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 205.73, 170.46, 74.98, 25.73, 20.81, 16.04. ES-API *m/z*: M^+ 131.0. This compound had spectral data which were the same as the given in the literature.[9]

1-acetoxy-4-methyl-2-pentanone (13): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.60 (2H, s), 2.26 (2H, d, J = 6.7 Hz) 2.14 (1H, m), 2.14 (3H, s), 0.92 (6H, d, J = 6.4 Hz). ^{13}C{^{1}H} NMR

($CDCl_3$, 125 MHz): δ 203.60, 170.38, 68.43, 47.66, 24.58, 22.60, 20.58. ES-API *m/z*: M^+ 159.1. The ^1H NMR is similar to reported.[10]

1-Acetoxy 2-propanone (15): ^1H NMR ($CDCl_3$, 500 MHz): δ 4.64 (2H, s), 2.16 (3H, s), 2.15 (3H, s). ^{13}C {^1H} NMR ($CDCl_3$, 125 MHz): δ 201.64, 170.18, 68.39, 26.15, 20.57. ES-API *m/z*: M^+ 117.5. The spectral data were consistent with those previously reported.[11]

2-acetoxycyclopetanone (17): ^1H NMR ($CDCl_3$, 500 MHz): δ 5.04 (1H, dt, J = 1.4, 10.2 Hz), 2.40 (1H, m), 2.33 (1H, m) 2.23 (1H, quint, J = 9.5), 2.11 (3H, s), 2.08 (1H, m), 1.83 (2H, m). ^{13}C {^1H} NMR ($CDCl_3$, 125 MHz): δ 212.62, 170.31, 75.80, 34.92, 28.47, 20.85, 17.18. ES-API *m/z*: . Lit.[9,12–15]

2-acetoxycyclohexanone (19): ^1H NMR ($CDCl_3$, 500 MHz): δ 5.09 (1H, dd, J = 6.3, 12.0 Hz), 2.43 (1H, ddt, J = 2.4, 4.5, 13.7 Hz), 2.33 (1H, dt, J = 6.0 13.7 Hz), 2.23 (1H, m), 2.08 (3H, s), 2.03 (1H, dquintd, J = 2.8, 6.0, 13.0 Hz), 1.91 (1H, m), 1.70 (2H, m), 1.56 (1H, tq , J = 3.8, 13.0 Hz). ^{13}C {^1H} NMR ($CDCl_3$, 125 MHz): δ 204.67, 170.06, 76.61, 40.73, 33.10, 27.21, 23.79, 20.78. ES-API *m/z*: M^+ 157.1. Spectral data are consistent with lit.[12,14–16]

2-acetoxycycloheptanone (21): ^1H NMR ($CDCl_3$, 500 MHz): δ 5.19 (1H, dd, J = 3.5, 9.8 Hz), 2.60 (1H, dt, J = 5.6, 16.6 Hz), 2.38 (1H, ddd, J = 4.5, 10.9, 15.8 Hz), 2.08 (3H, s), 1.95 (1H, dquint, J = 3.1, 13.7 Hz), 1.80 (3H, m), 1.70 (2H, m) 1.59 (1H, qd, J = 2.4, 10.6 Hz), 1.31 (1H, m). ^{13}C{^1H} NMR ($CDCl_3$, 125 MHz): δ 207.75, 170.30, 78.21, 40.74, 30.34 (2C), 28.52, 26.51, 23.06, 20.80. ES-API *m/z*: M^+ 171.1. Spectral data in agreement with the report.[15,17]

***Cis*-2-acetoxy-5-methylcyclohexanone (23a)**: ^1H NMR ($CDCl_3$, 500 MHz): δ 5.01 (1H, dd, J = 6.3, 10.9 Hz), 2.48 (1H, dd, J = 5.3, 12.7 Hz), 2.20 (1H, m), 2.17 (1H, m), 2.05 (3H, s), 1.95 (1H, m), 1.92 (1H, m), 1.67 (1H, m), 1.52 (1H, dd, J = 2.8, 13.4 Hz), 0.95 (3H, d, J = 6.3 Hz). ^{13}C {^1H} NMR ($CDCl_3$, 125 MHz): δ 204.97, 170.10, 76.46, 46.77, 34.33, 31.63, 24.42, 22.21, 20.73. ES-API *m/z*: M^+ 171.1

***Trans*-2-acetoxy-5-methylcyclohexanone (23b)**: ^1H NMR ($CDCl_3$, 500 MHz): δ 5.05 (1H, ddd, J = 1.0, 6.7, 12.7 Hz), 2.35 (1H, ddd, J = 2.8, 3.8, 13.4 Hz), 2.16 (1H, m), 2.05, (3H, s), 2.05 (1H, m), 1.84 (1H, m), 1.77 (1H, m), 1.66 (1H, qd, J = 3.5, 13.4 Hz), 1.42 (1H, qd, J = 3.5, 13.4 Hz), 0.95 (3H, d, J = 6.3 Hz). ^{13}C {^1H} NMR ($CDCl_3$, 125 MHz): δ 203.99, 170.00, 76.26, 48.61, 35.03, 32.16, 31.63, 22.11, 20.73. ES-API *m/z*: M^+ 171.1

***Cis*-2-acetoxy-6-methylcyclohexanone (26b)**: ^1H NMR ($CDCl_3$, 500 MHz): δ 5.12 (1H, ddd, J = 1.0, 6.3, 12.3 Hz), 2.48, (1H, sepd, J = 1.06, 6.36 Hz), 2.28 (1H, m), 2.13 (3H, s), 2.11 (1H, m), 2.05, (1H, m), 1.82 (1H, m), 1.72 (1H, m), 1.32 (1H, ddd, J = 3.8, 13.0 Hz) 1.03 (3H, d, J = 6.3 Hz). ^{13}C{^1H} NMR ($CDCl_3$, 125 MHz): δ 206.13, 170.05, 76.79, 44.34, 36.13, 33.49, 28.00, 20.82, 13.94. ES-API *m/z*: M^+ 171.1. Previously reported.[18]

***Cis* -2-acetoxy-4-*t*-butylcyclohexanone (29a)**: ^1H NMR ($CDCl_3$, 500 MHz): δ 5.17 (1H, ddd, , J = 1.0, 6.3, 13.0 Hz), 2.45 (1H, dq, , J = 2.4, 4.2, 13.7 Hz), 2.37 (1H, ddd, , J = 1.0, 6.0, 13.7 Hz), 2.27 (1H, dquin, , J = 2.18, 12.01 Hz) 2.11 (3H, s), 2.07 (2H, m), 1.66 (1H, tt, , J = 2.8, 12.3 Hz) 1.53 (1H, q, J = 12.3 Hz), 1.39 (1H, qd, J = 4.5, 13.4 Hz), 0.89 (9H, s). ^{13}C{^1H} NMR ($CDCl_3$, 125 MHz): δ 205.01, 170.08, 76.22, 45.83, 39.56, 34.25, 32.51, 27.65, 20.82. ES-API *m/z*: M^+ 213.1. Lit.[19]

1-acetoxy-4-methyl-3-penten-2-ona (31): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 6.00 (1H, septet, J = 1.0 Hz), 4.61 (2H, s), 2.13 (3H, d, J = 1.0 Hz), 2.12 (3H, s), 1.88 (3H, d, J = 1.0 Hz). ^{13}C {^{1}H} NMR ($CDCl_3$, 125 MHz): δ 203.60, 170.38, 159.41, 118.93, 68.79, 23.01, 21.31, 20.62. ES-API *m/z*: M^+ 157.1. The ^{1}H NMR and MS are similar to reported.[20]

1-acetoxy-2-indanona (33): δ 7.36 (4H, m), 5.97 (1H, s), 3.60 (1H, d, J = 22.0 Hz), 3.67 (1H, d, J = 22.0 Hz), 2.17 (3H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 209.56, 170.26, 137.18, 136.94, 129.69, 128.08, 125.31, 125.27, 75,37, 41.38, 20.67. ES-API *m/z*: M^+ 192.1. The ^{1}H NMR is similar to reported.[21]

7-oxo-6,7-dihydro-5H-cyclopenta[b]pyridin-6-yl acetate (35): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.81 (1H, d, J = 4.2 Hz), 7.85 (1H, d, J = 7.7 Hz), 7.50 (1H, dd, J = 4.5, 8,1 Hz), 5.41 (1H, dd, J = 4.9, 8.1 Hz), 3.68 (1H, dd, J = 8.12, 17.3 Hz), 3.08 (1H, dd, J = 4.9, 17.31 Hz), 2.18 (3H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 199.55, 170.38, 151.80, 151.64, 146.05, 135.36, 128.56, 72.77, 31.65, 20.75. ES-API *m/z*: M^+ 192.0

(*E*)-1,1'-(diazene-1,2-diylbis(2,1-phenylene))bis(ethan-1-one) (39): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.72 (2H, dd, J = 0.7, 8.4 Hz), 7.88 (2H, dd, J = 1.7, 8.1 Hz), 7.54 (2H, dt, J = 1.4, 8.4 Hz), 7.10 (2H, dt, J = 1.4, 8.4 Hz), 2.66 (6H, s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 202.98, 141.10, 135.27. 131.70, 122.39, 121.70, 120.79, 28.75. ES-API *m/z*: M^+ 267.1. First reported.[22]

Cholesta-1,4-dien-3-one (41): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.05 (1H, d, J = 10.0 Hz), 6.22 (1H, dd, J = 2.0, 10.0 Hz), 6.06 (1H, t, J = 2.0 Hz), 1.24 (3H, s), 0.90 (3H, d, J = 3.1 Hz), 0.85 (6H, d, J = 3.3 Hz), 0.72 (3H, s). ^{13}C {^{1}H} NMR ($CDCl_3$, 125 MHz): δ 186.36, 169.96, 156.36, 127.35, 123.75, 56.13, 55.53, 52.46, 42.74, 39.57, 39.55, 36.14, 35.81, 35.75, 35.60, 33.78, 33.05, 28.21, 28.09, 24.47, 23.89, 22.96, 22.91, 22.64, 18.77, 18.68, 12.13. ES-API *m/z*: M^+ 383.4. Previously reported.[23,24]

1,4-Benzenediol (43): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 6.64 (s). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 149.75, 116.19.

Reference:

(1) Harris, R. K.; Becker, E. D.; Cabral De Menezes, S. M.; Goodfellow, R.; Granger, P. NMR Nomenclature: Nuclear Spin Properties and Conventions for Chemical Shifts (IUPAC Recommendations 2001). *Concepts Magn. Reson. Part A Bridg. Educ. Res.* **2002**, *14* (5), 326–346.

(2) Berger, S.; Braun, S. *200 and More NMR Experiments, A Practical Course*; Wiley-VCH, 2004.

(3) Sabot, C.; Kumar, K. A.; Antheaume, C.; Mioskowski, C. Triazabicyclodecene: An Effective Isotope Exchange Catalyst in CDCl 3. *J. Org. Chem.* **2007**, *72* (13), 5001–5004.

(4) Srinivas, B. T. V.; Supriya, P.; Rohithrao, V.; Naidu, N. V. S.; Sreedhar, B. Magnetic CuFe2O4 and Fe3O4 Nanoparticles Catalyzed Diacetoxylation of Alkenes and 1,2-Oxyacetoxylation of Terminal Alkynes Using PhI(OAc)2 as Oxidant. *ChemistrySelect* **2017**, *2* (8), 2600–2604.

(5) Lex, T. R.; Swasy, M. I.; Whitehead, D. C. Relative Rate Profiles of Functionalized Iodoarene Catalysts for Iodine(III) Oxidations. *J. Org. Chem.* **2015**, *80* (24), 12234–12243.

(6) Bityukov, O. V.; Matveeva, O. K.; Vil, V. A.; Kokorekin, V. A.; Nikishin, G. I.; Terent'Ev, A. O. Electrochemically Induced Intermolecular Cross-Dehydrogenative C-O Coupling of β-Diketones and β-Ketoesters with Carboxylic Acids. *J. Org. Chem.* **2019**, *84* (3), 1448–1460.

(7) Matveenko, M.; Cichero, E.; Fossa, P.; Becker, C. F. W. Impaired Chaperone Activity of Human Heat Shock Protein Hsp27 Sitespecifically Modified with Argpyrimidine. *Angew. Chem., Int. Ed.* **2016**, *55*, 11397–11402.

(8) Stetter, H.; Mohrmann, K.; Schlenker, W. Herstellung Und Reaktionen von Alkoxy- Und Acetoxy-2,s-Diketonen. *Chem. Ber.* **1981**, *596*, 581–596.

(9) Singh, M.; Murray, R. W. Chemistry of Dioxiranes. 21. Thermal Reactions of Dioxiranes. *J. Org. Chem.* **1992**, *57* (15), 4263–4270.

(10) Hoffman, J.; Sarafidis, A.; Voss, J. ACETOXYLATION OF KETONES BY ELECTROGENERATED LEAD TETRAACETATE. *Electrochim. Acta* **1991**, *36* (7), 1165–1169.

(11) Griesbaum, K.; Greinert, R. Ozonolysen von Olefinen Mit Vinylständigen Chlorsubstituenten Auf Polyethylen. *Chem. Ber.* **1990**, *123* (2), 391–397.

(12) Tanyeli, C.; Turkut, E.; Akhmedov, I. M. Chemoenzymatic Synthesis of A′- And α-Acetoxylated Cyclic Ketones. *Tetrahedron Asymmetry* **2004**, *15* (11), 1729–1733.

(13) Rubottom, G. M.; Mott, R. C.; Juve, H. D. Reaction of Enol Silyl Ethers with Silver Carboxylate-Iodine. Synthesis of α-Acyloxy Carbonyl Compounds. *J. Org. Chem.* **1981**, *46* (13), 2717–2721.

(14) May, G.; Pinhey, J. The Chemistry of Aryllead(IV) Tricarboxylates. Reaction with β-Diketones. *Aust. J. Chem.* **1982**, *35* (7), 1859–1871.

(15) Müller, C. E.; Zell, D.; Schreiner, P. R. One-Pot Desymmetrization of Meso-l,2-Hydrocarbon Diols through Acylation and Oxidation. *Chem. - A Eur. J.* **2009**, *15* (38), 9647–9650.

(16) Lauktien, G.; Volk, F. J.; Frahm, A. W. Diastereo- and Enantioselective Synthesis of Cis-2- Hydroxycyclohexanamine and Corresponding Ethers by Asymmetric Reductive Amination. *Tetrahedron Asymmetry* **1997**, *8* (20), 3457–3466.

(17) Dave, V.; Warnhoff, E. W. Regiospecific Homologation of Unsymmetrical Ketones. *J. Org. Chem.* **1983**, *48* (15), 2590–2598.

(18) House, H. O.; Richey, F. A. Use of Ketoxime Derivatives to Prepare α-Acetoxy Ketones. *J. Org. Chem.* **1969**, *34* (5), 1430–1439.

(19) Kuehne, M. E.; Giacobbe, T. J. The Formation of α-Acetoxy Ketones by Oxidation of Enamines with Thallium Triacetate. *J. Org. Chem.* **1968**, *33* (9), 3359–3369.

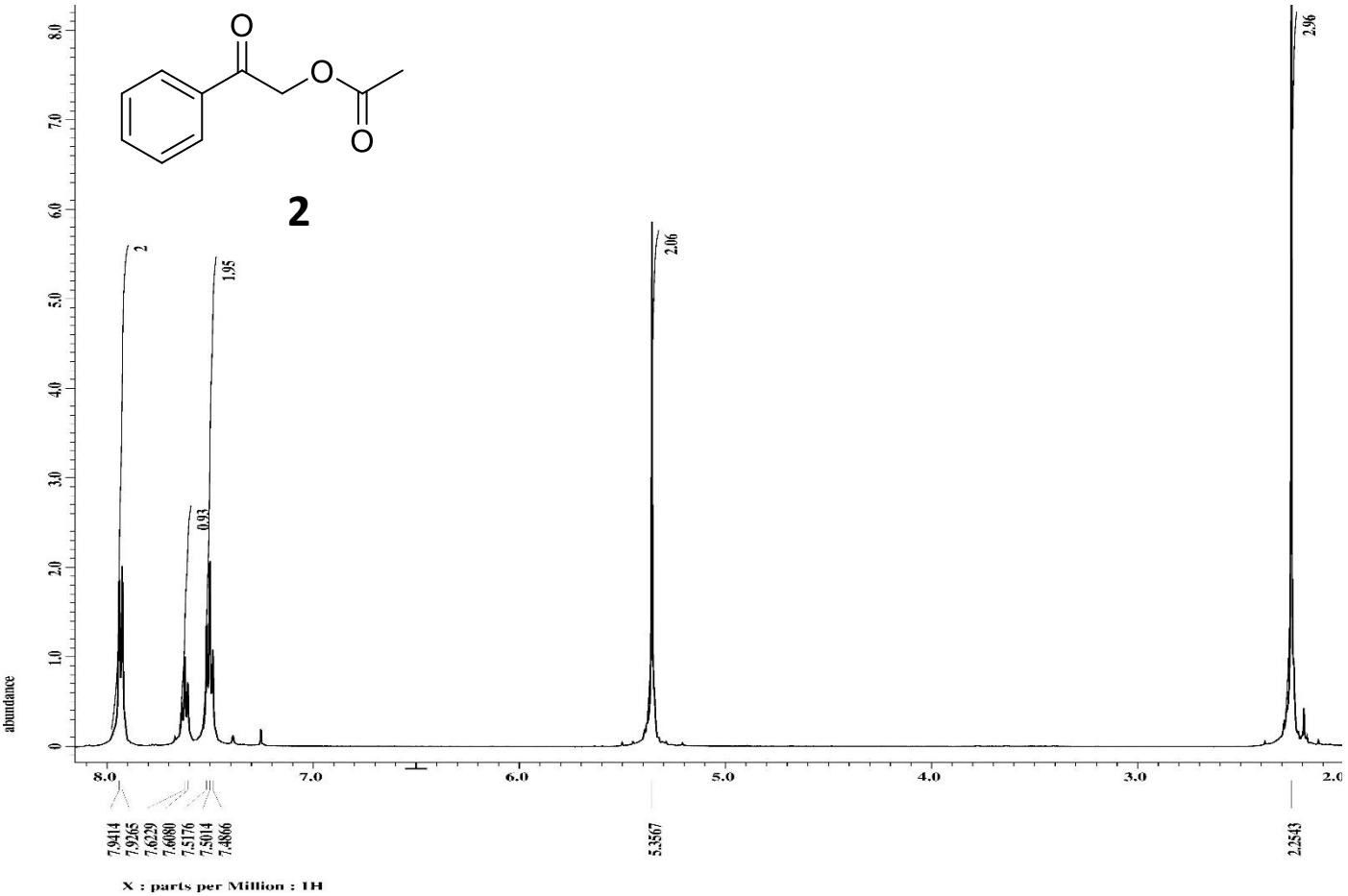
(20) Fristad, W. E.; Peterson, J. R. Manganese(III)-Mediated γ-Lactone Annulation. *J. Org. Chem.* **1985**, *50* (1), 10–18.

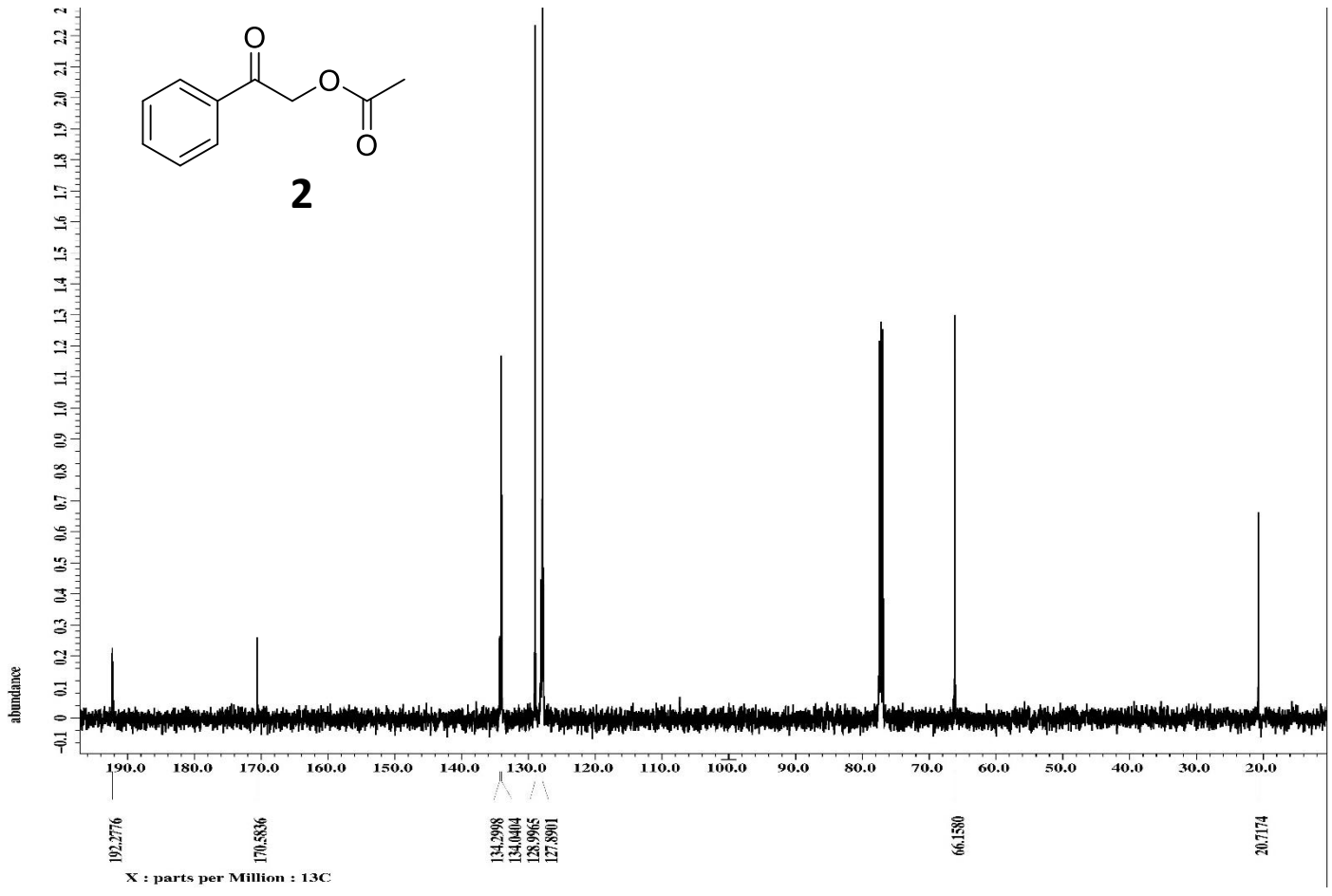
(21) Dayan, S.; Bareket, Y.; Rozen, S. An Efficient α-Hydroxylation of Carbonyls Using the HOF·CH3CN Complex. *Tetrahedron* **1999**, *55* (12), 3657–3664.

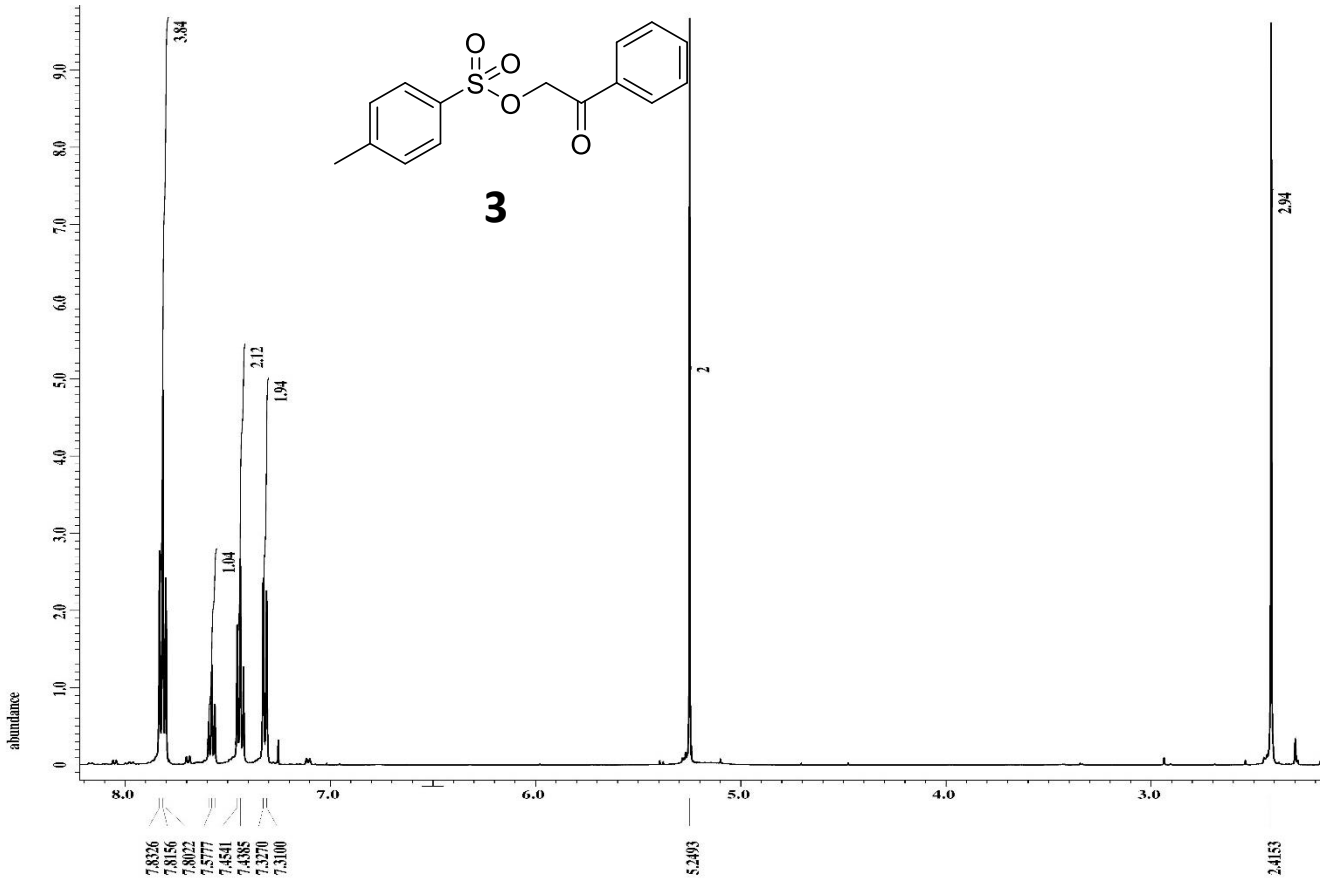
(22) Bamberger, E. Weiteres Über Anthranil Und Methyl-Anthranil. (XX. Mitteilung Über Anthranil.). *Berichte der Dtsch. Chem. Gesellschaft* **1915**, *48*, 537–582.

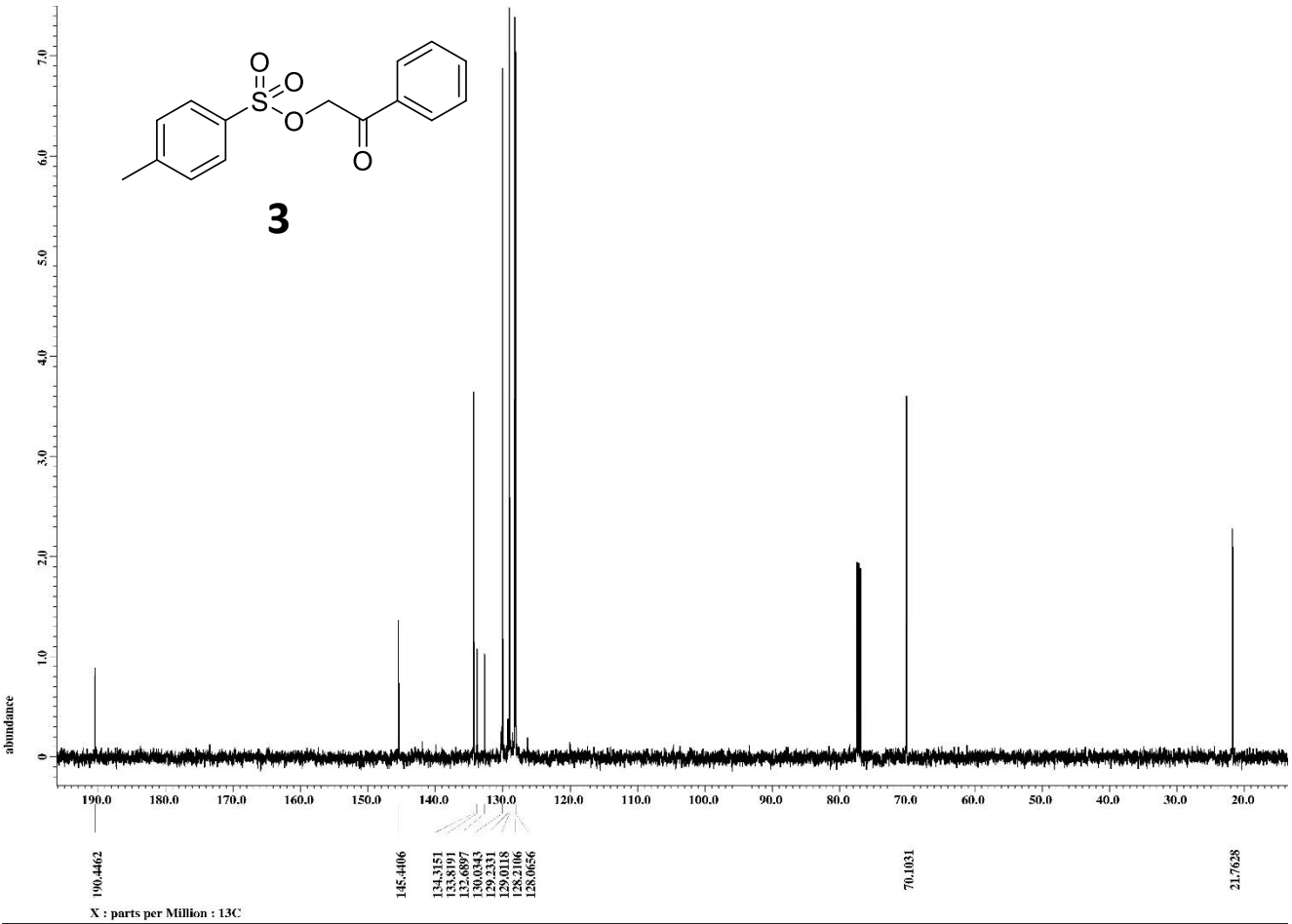
(23) Kubota, T.; Yoshida, K.; Hayashi, F.; Takeda, K. Studies on A-Norsteroids, I. Synthesis of A-Nor- -1,2-Dioxo Steroids. *Chem. Pharm. Bull.* **1965**, *13* (1), 50–57.

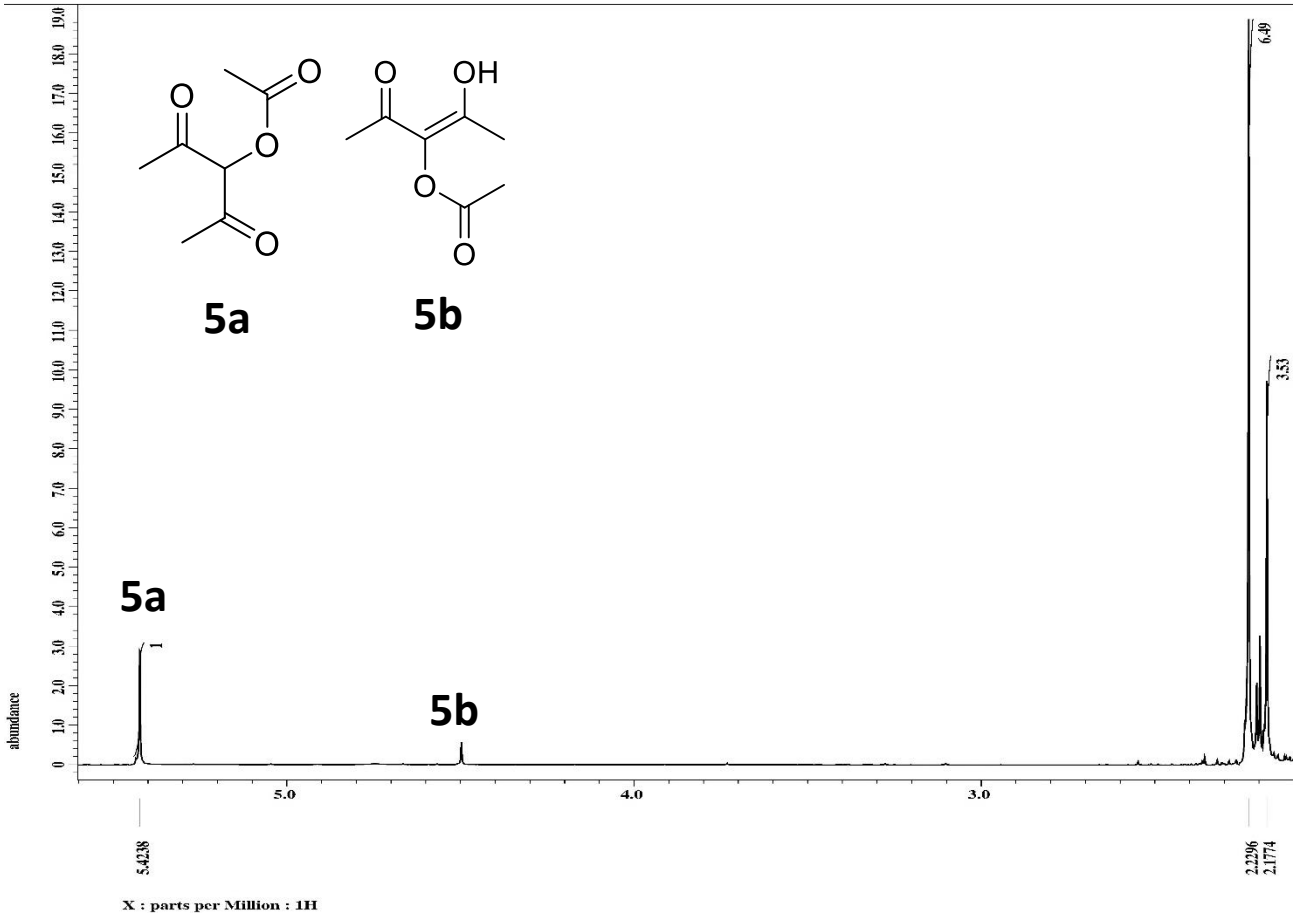
(24) Iida, T.; Shinohara, T.; Goto, J.; Nambara, T.; Chang, F. C. A Facile One-Step Synthesis of Delta 1,4-3-Keto Bile Acid Esters by Iodoxybenzene and Benzeneselenic Anhydride. *J. Lipid Res.* **1988**, *29* (8), 1097–1101.

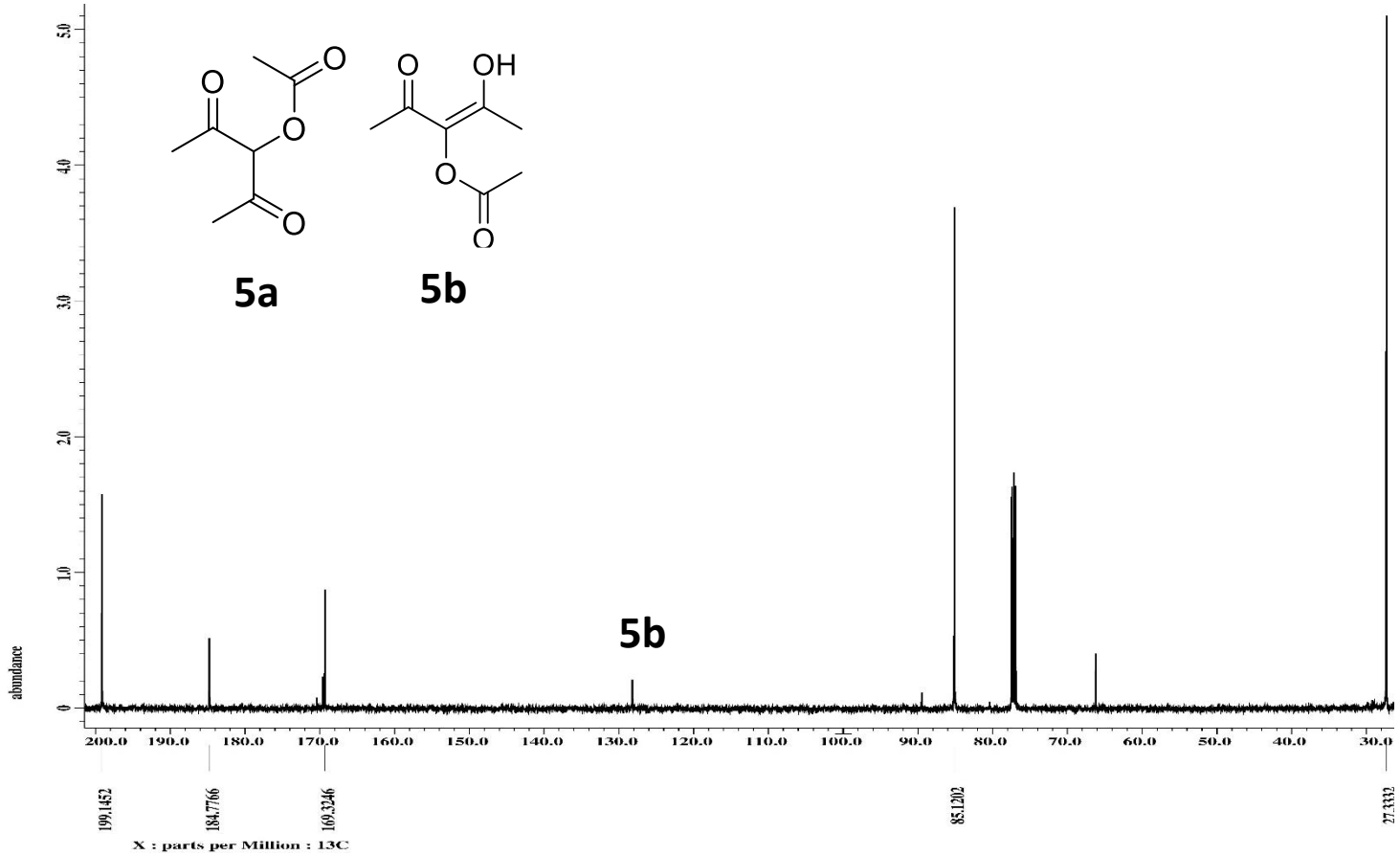

2
abundance
2.06
1.95
0.93
2.96
7.9414
7.9265
7.6229
7.6080
7.5176
7.5014
7.4866
5.3567
2.2543
X : parts per Million : 1H

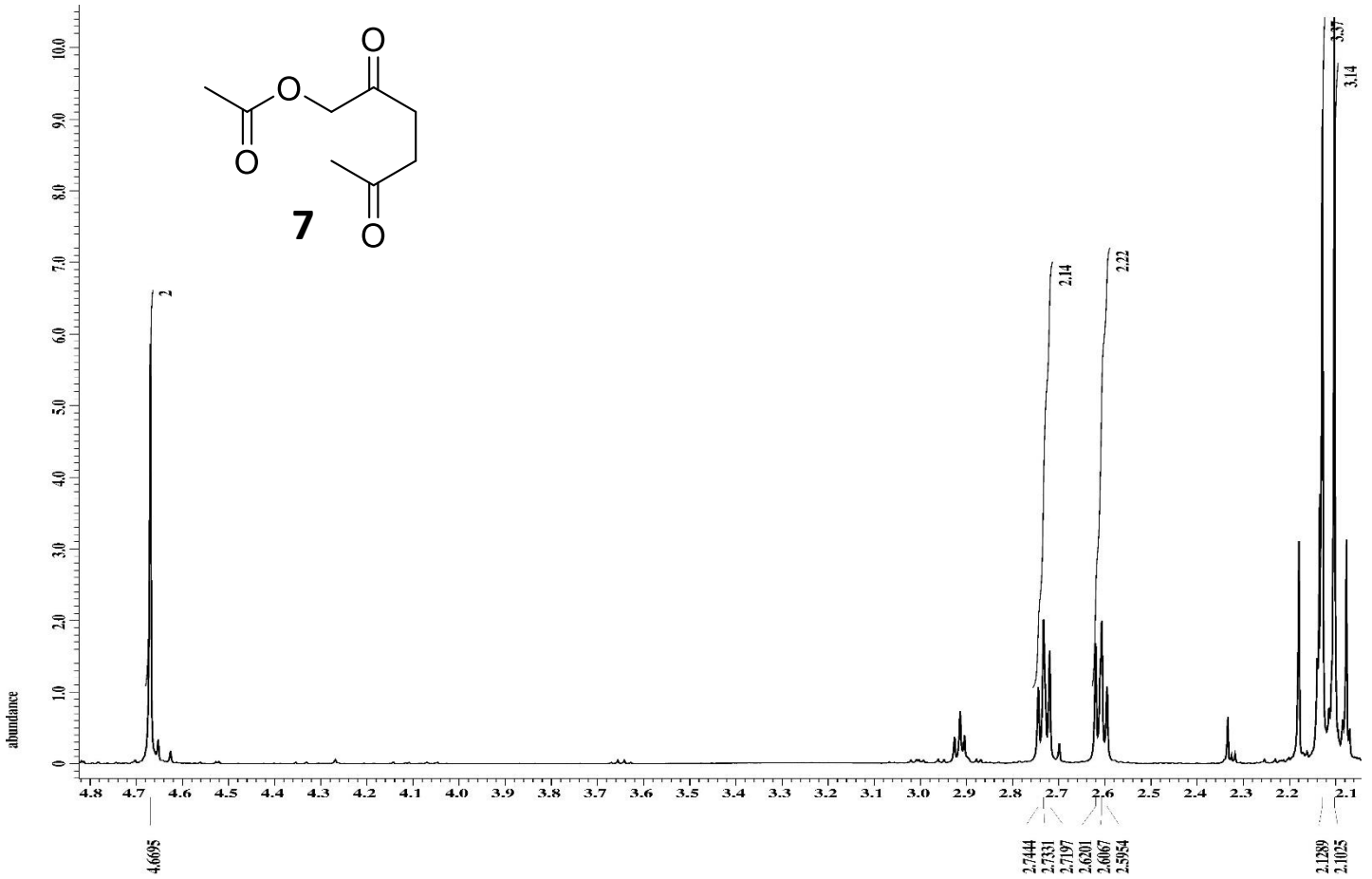

2
abundance
192.2776
170.5836
134.2998
134.0404
128.9965
127.8901
66.1580
20.7174
X : parts per Million : 13C

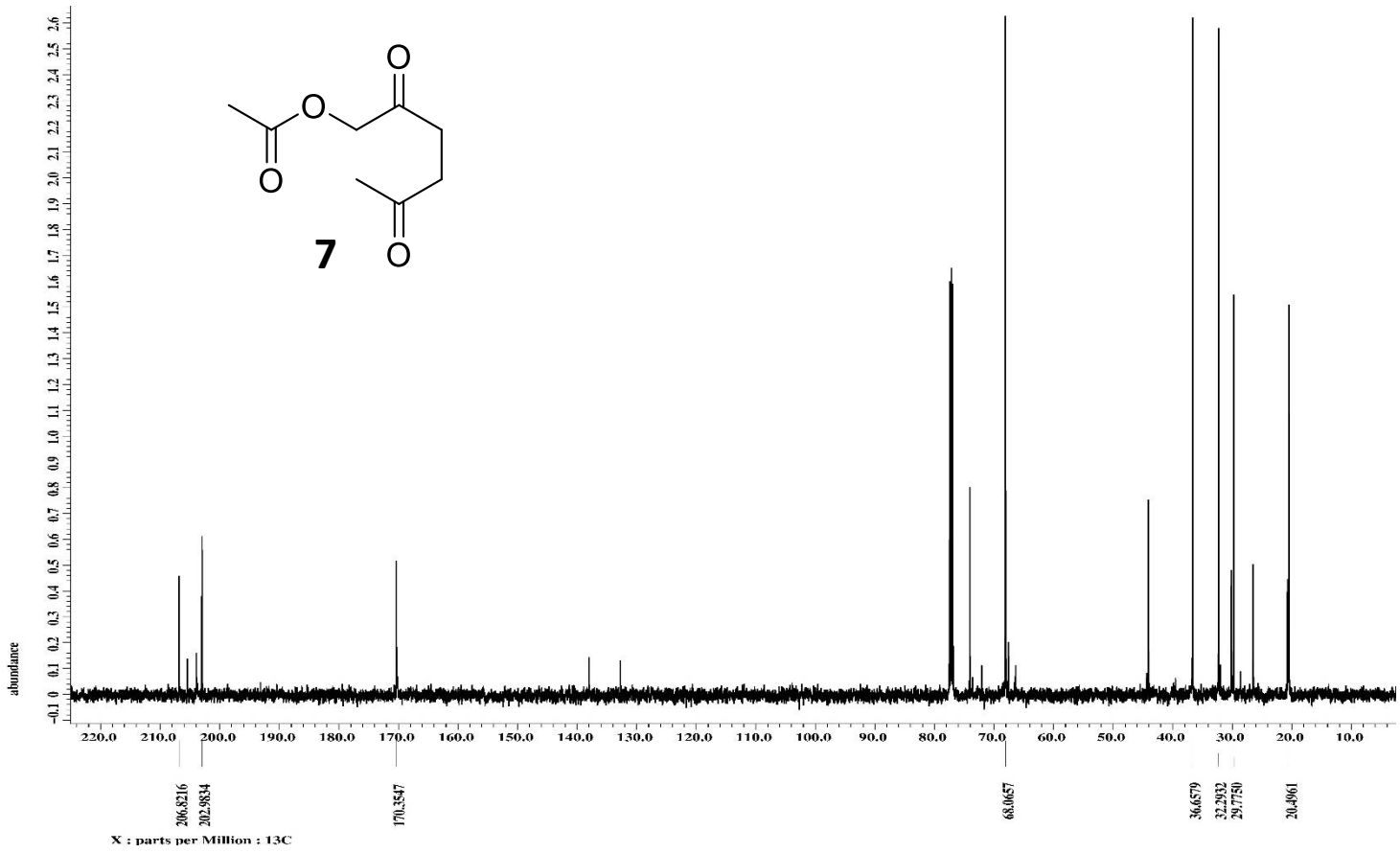

3.84
2.12
1.94
1.04
2
2.94
abundance
7.8326
7.8156
7.8022
7.5777
7.4541
7.4385
7.3270
7.3100
5.2493
2.4153
X : parts per Million : 1H

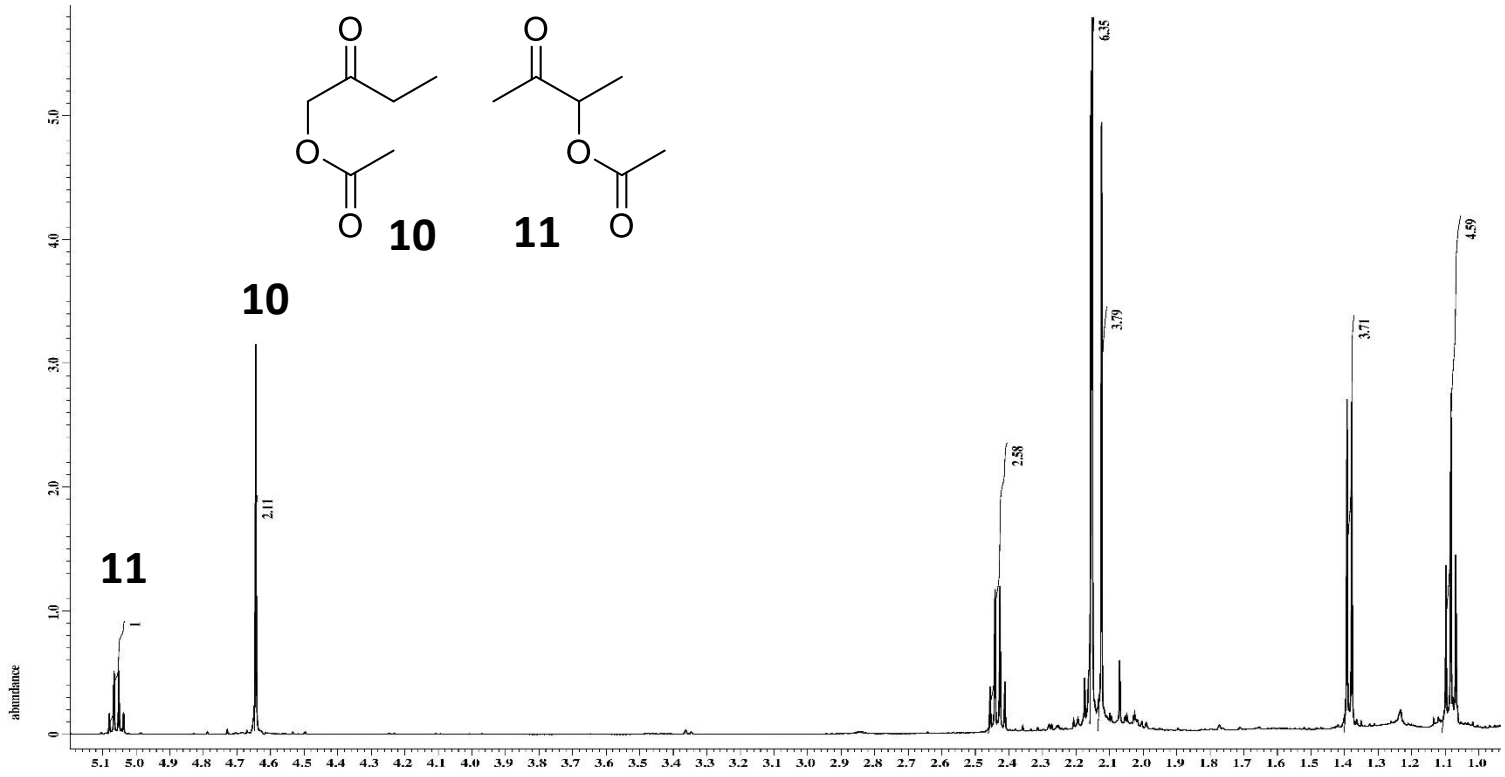

abundance
190.4462
145.4406
134.3151
133.8191
132.6897
130.0343
129.2331
129.0118
128.2106
128.0656
70.1031
21.7628
X : parts per Million : 13C

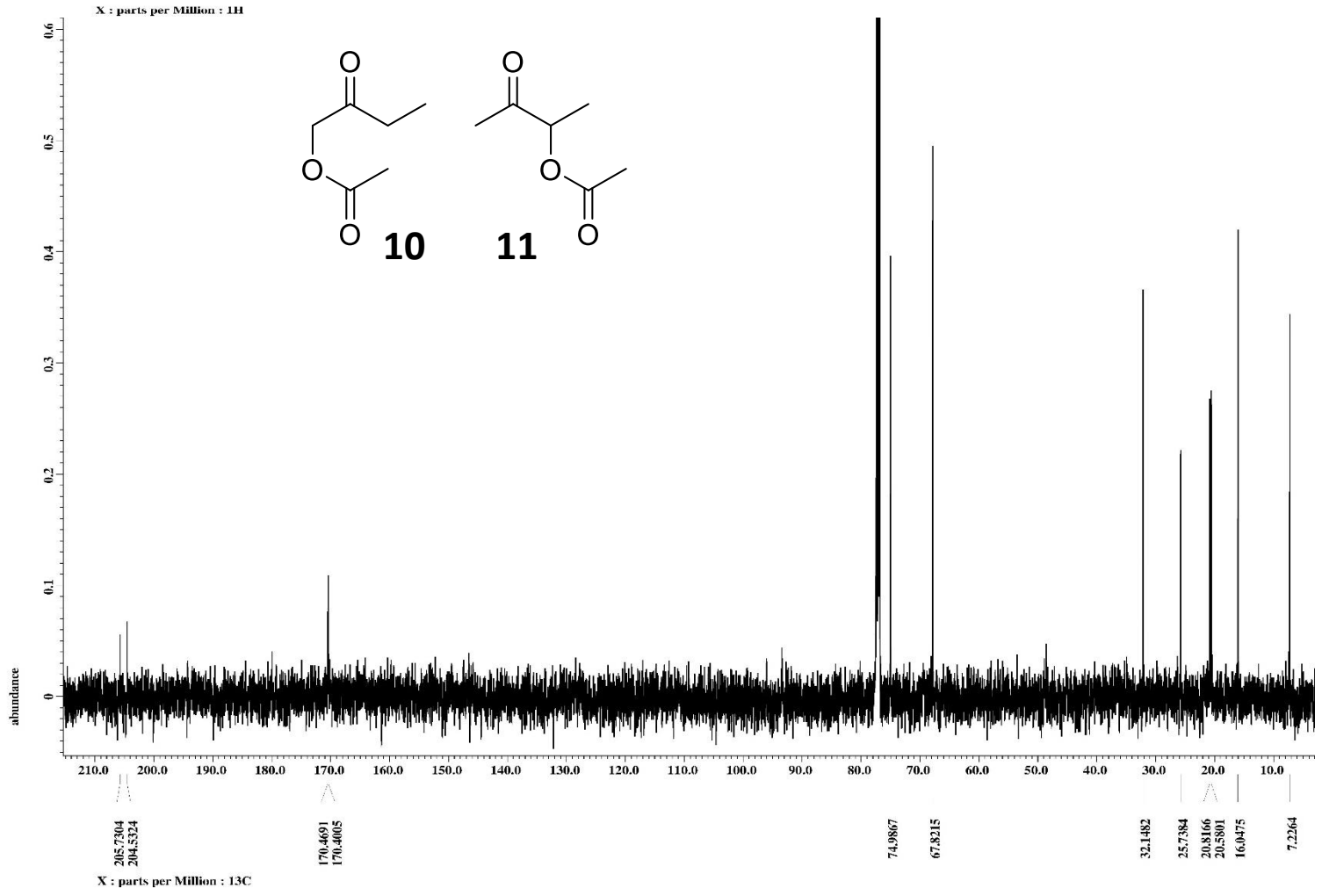

5a
5b
5a
5b
6.49
3.53
abundance
5.0
4.0
3.0
5.4238
2.2296
2.1774
X : parts per Million : 1H

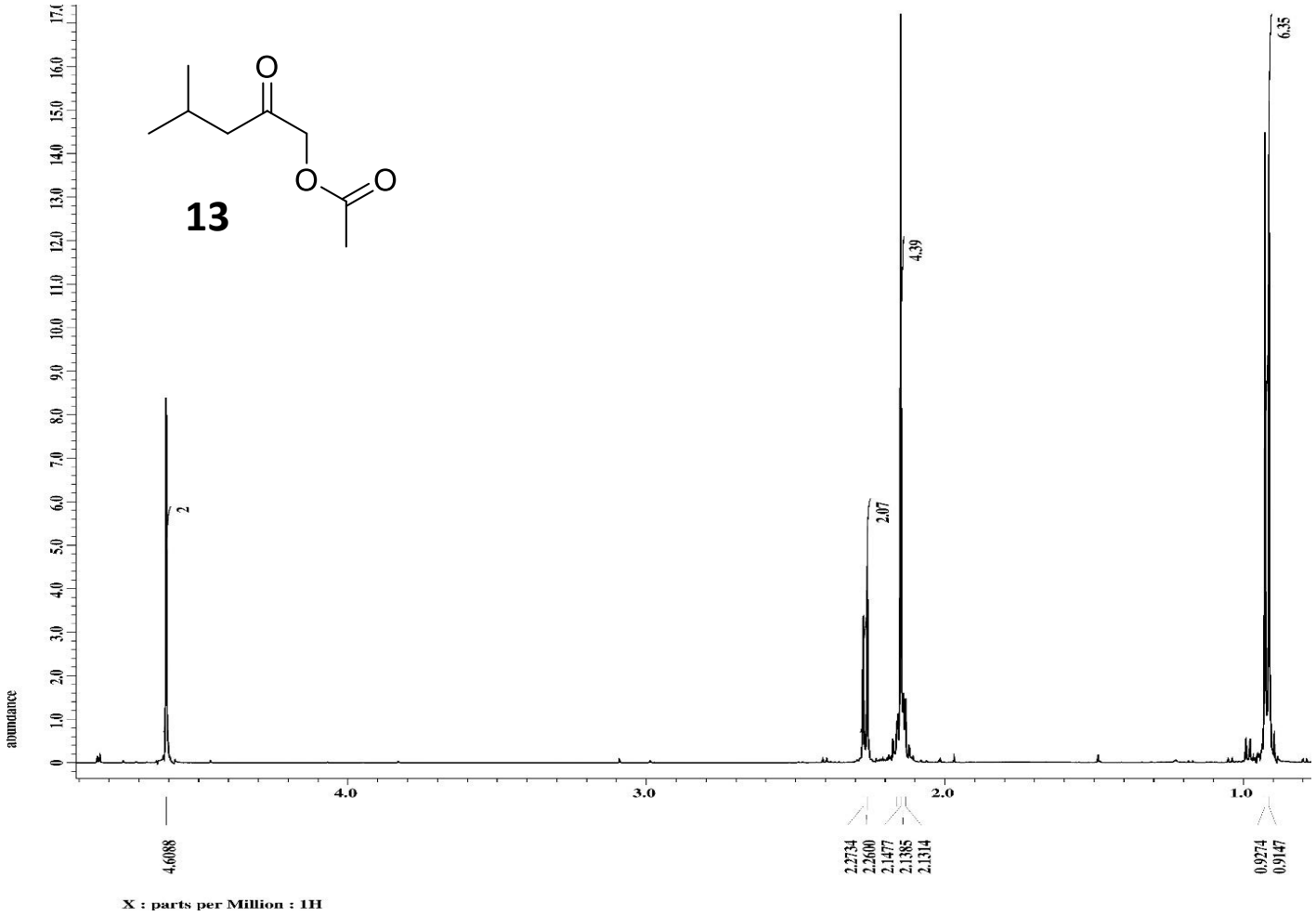

5a
5b
5b
abundance
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
190.1452
184.7766
169.3246
85.1202
27.3332
X : parts per Million : 13C

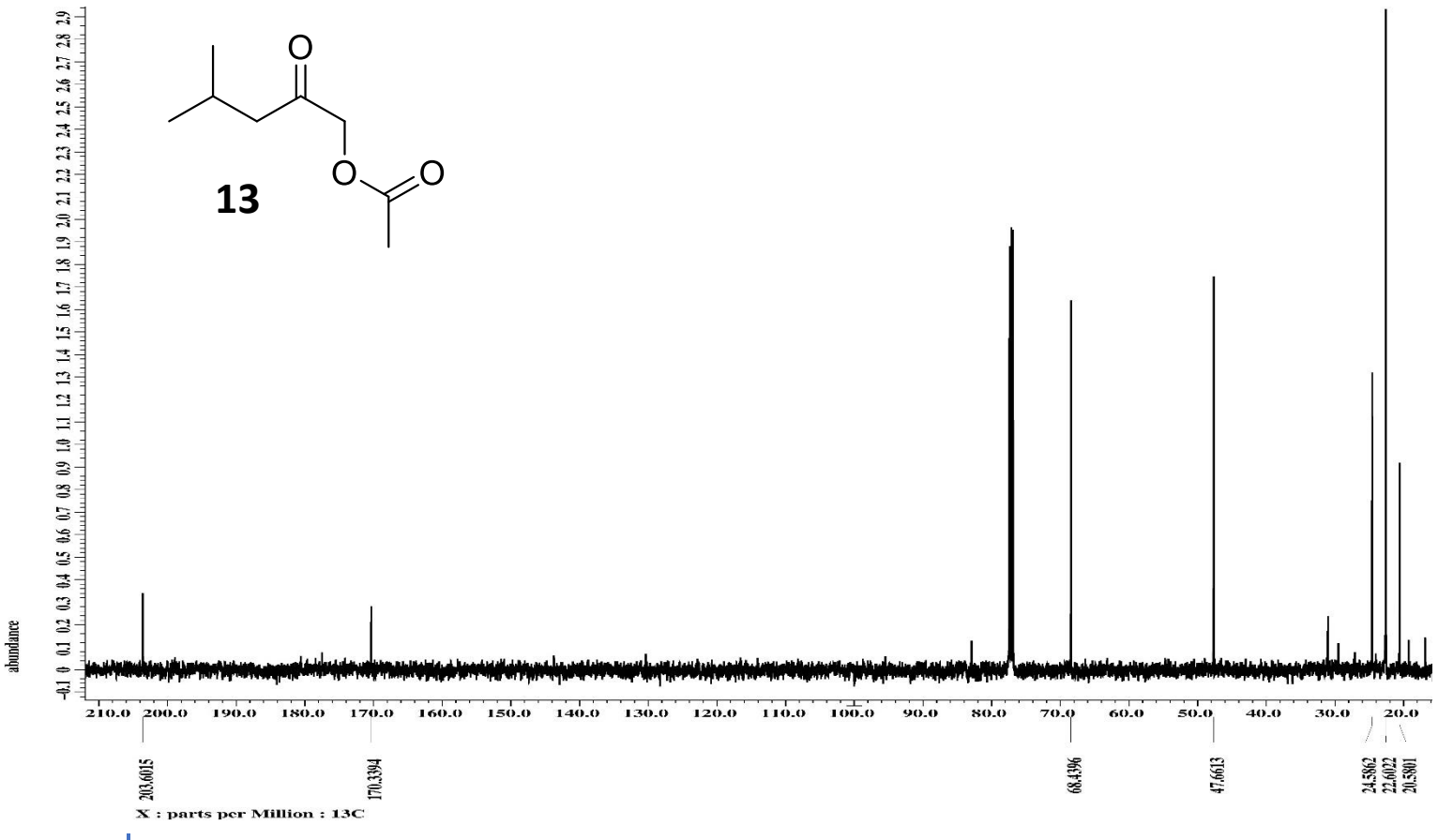

7
abundance
4.6695
2.7444
2.7331
2.7197
2.6201
2.6067
2.5954
2.1289
2.1025
X : parts per Million : 1H

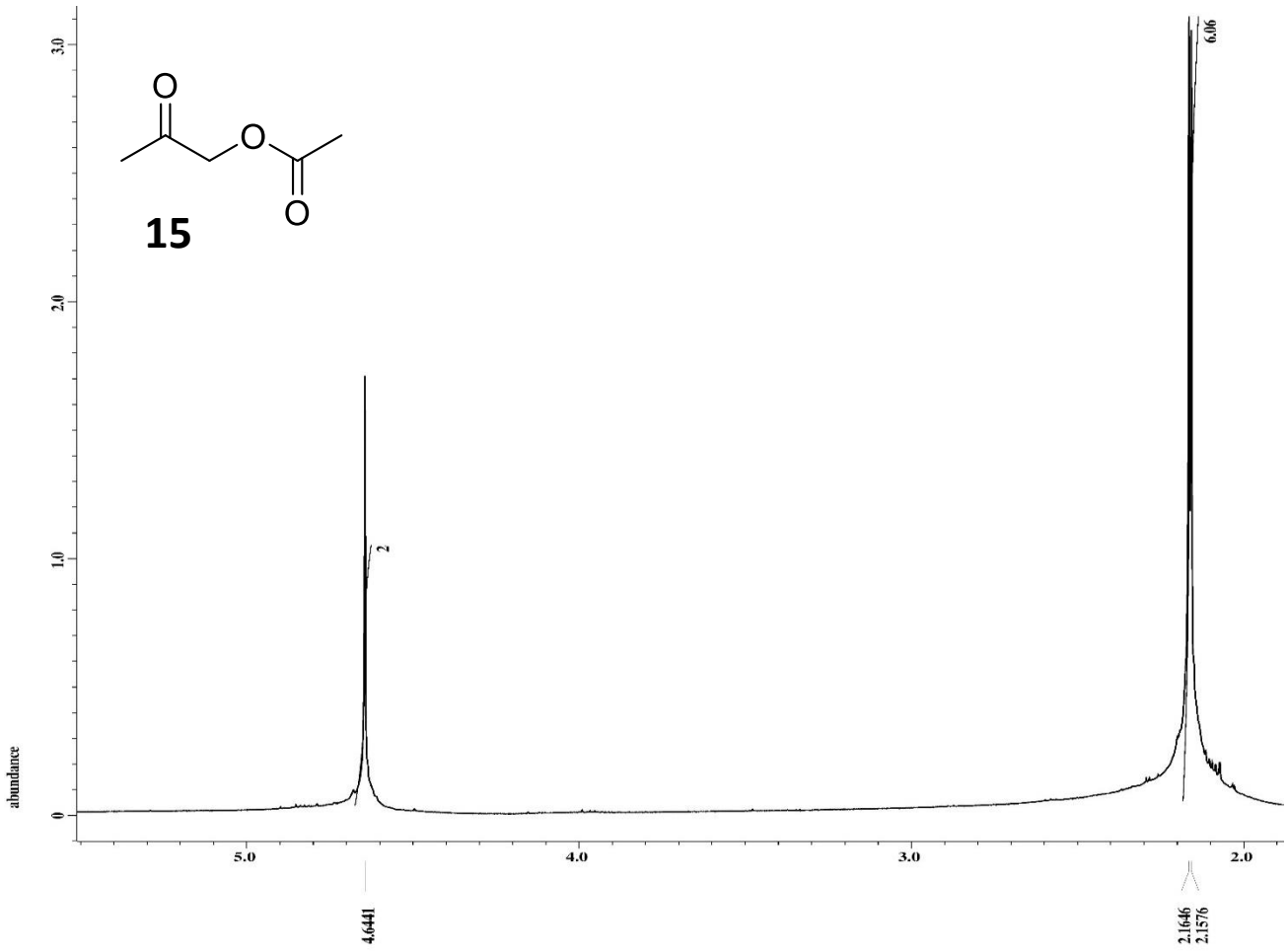

7
abundance
206.8216
202.9834
170.3547
68.0657
36.6579
32.2932
29.7750
20.4961
X : parts per Million : 13C

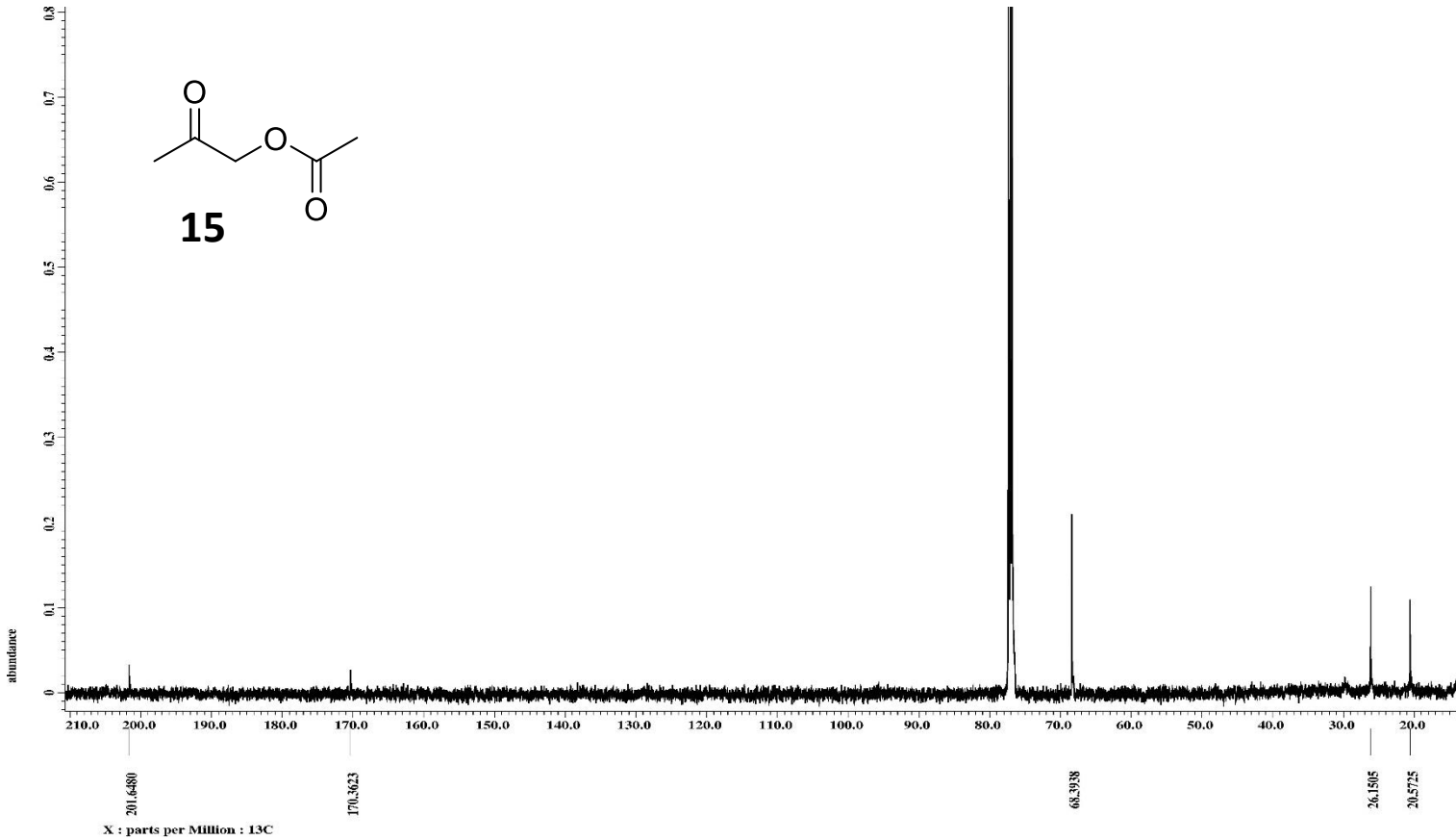

10
11
10
11
6.35
4.59
3.79
3.71
2.58
2.11
1
abundance

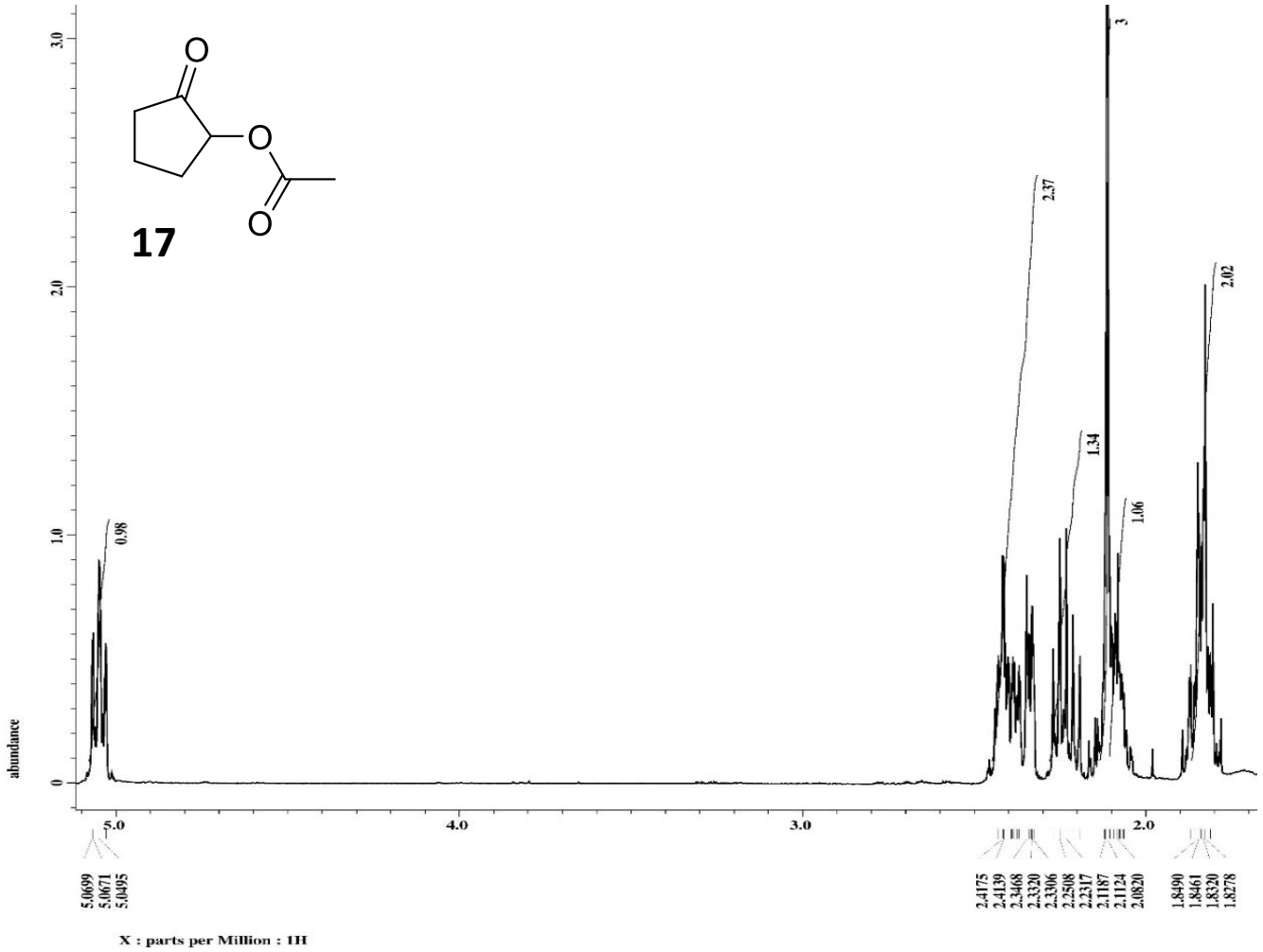

X : parts per Million : 1H
10
11
abundance
205.7304
204.5324
170.4691
170.4005
74.9867
67.8215
32.1482
25.7384
20.8166
20.5801
16.0475
7.2264
X : parts per Million : 13C

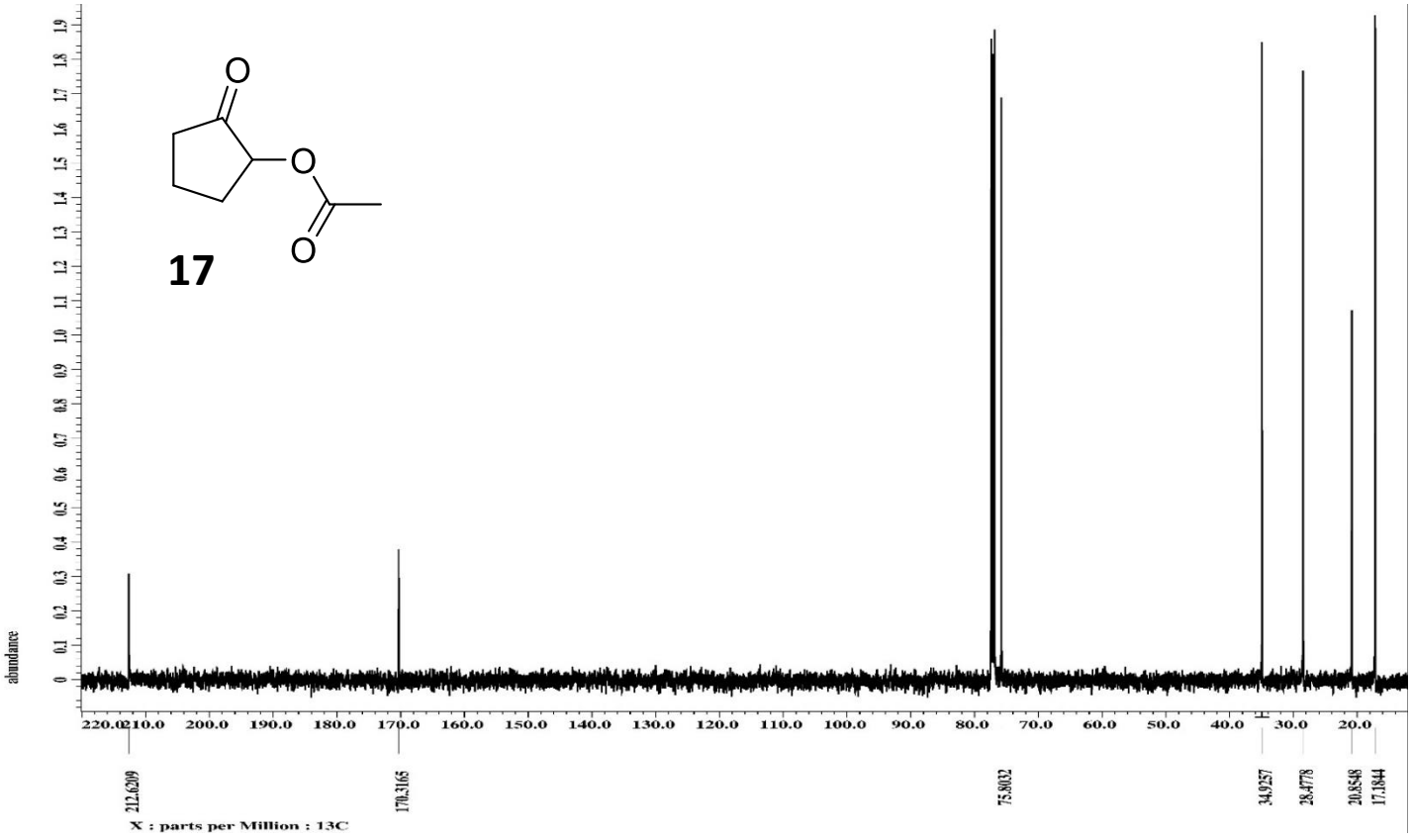

13
abundance
6.35
4.39
2.07
2
4.6088
2.2734
2.2600
2.1477
2.1385
2.1314
0.9274
0.9147
X : parts per Million : 1H

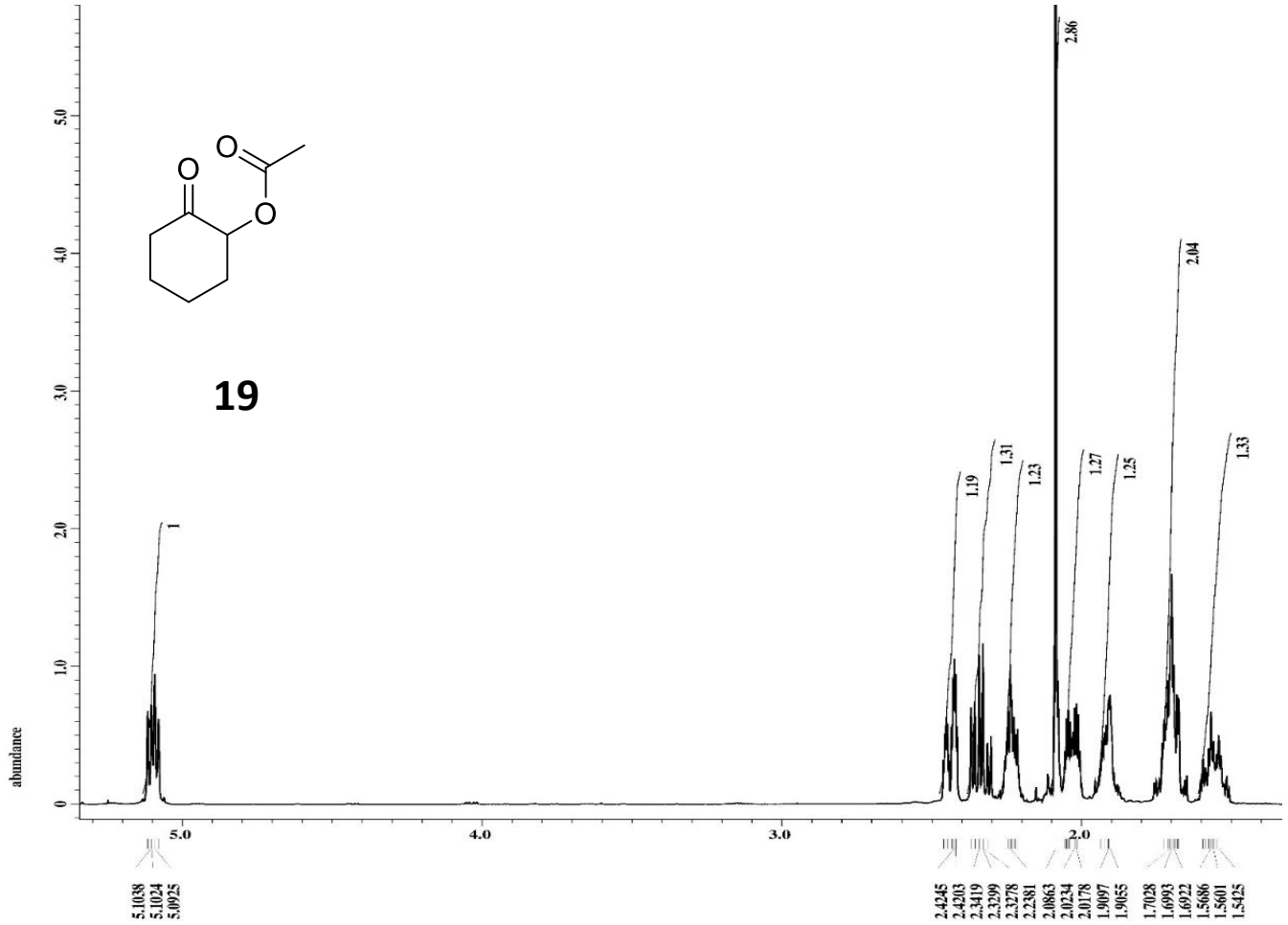

13
abundance
203.6015
170.3394
68.4396
47.6613
24.5862
22.6022
20.5801
X : parts per Million : 13C

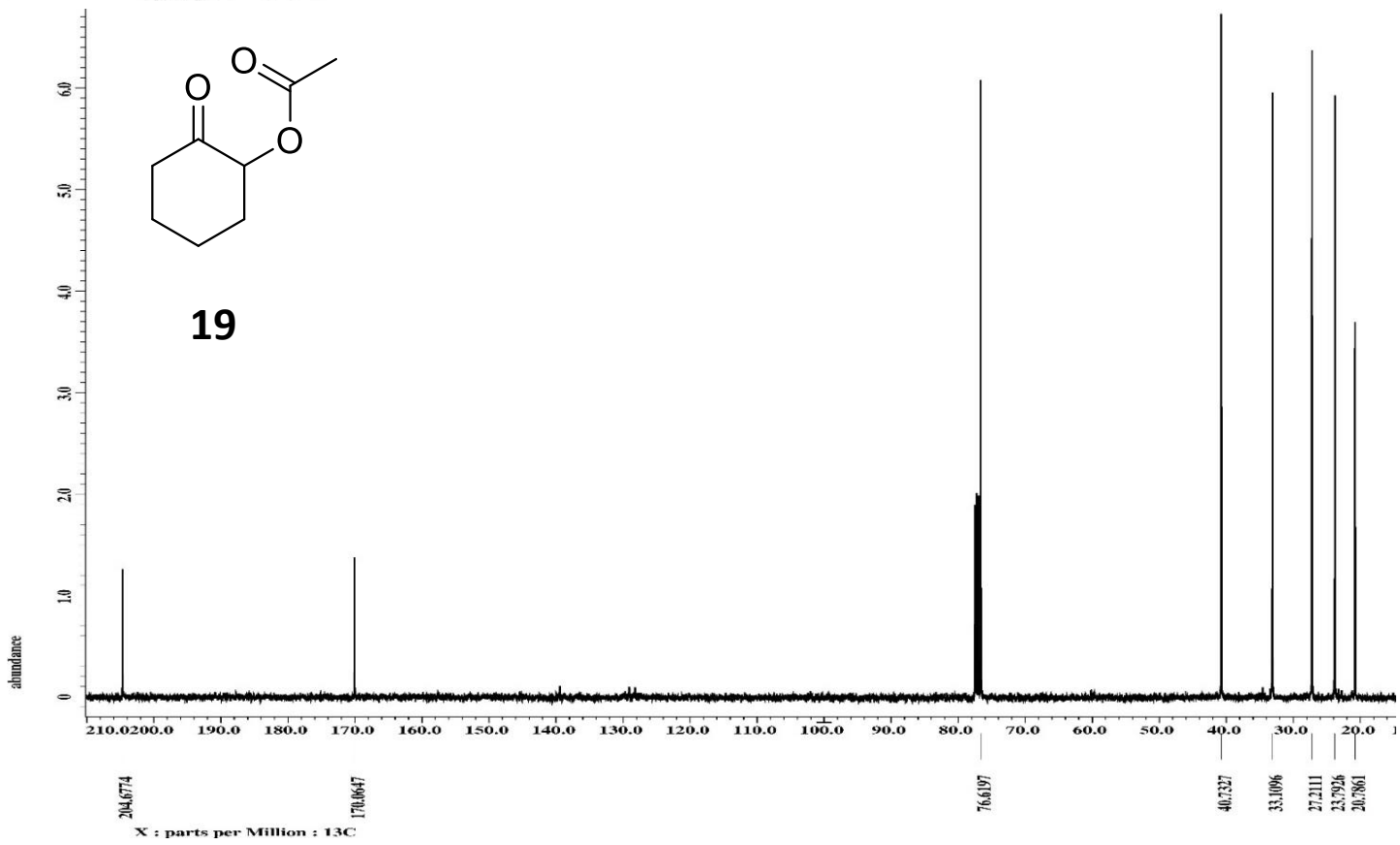

15
abundance
6.06
2
4.6441
2.1646
2.1576
X : parts per Million : 1H

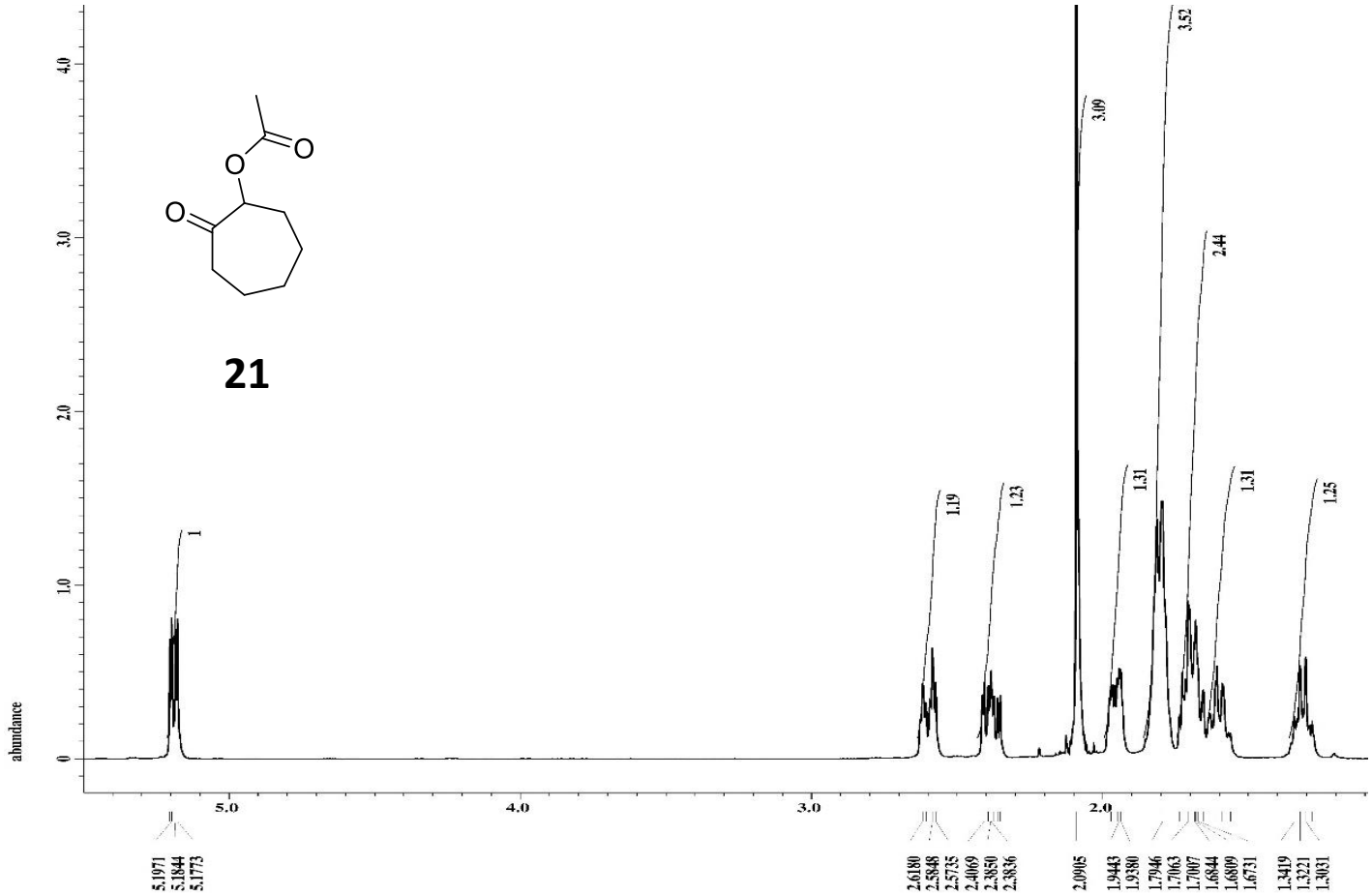

15
abundance
201.6480
170.3623
68.3938
26.1505
20.5725
X : parts per Million : 13C

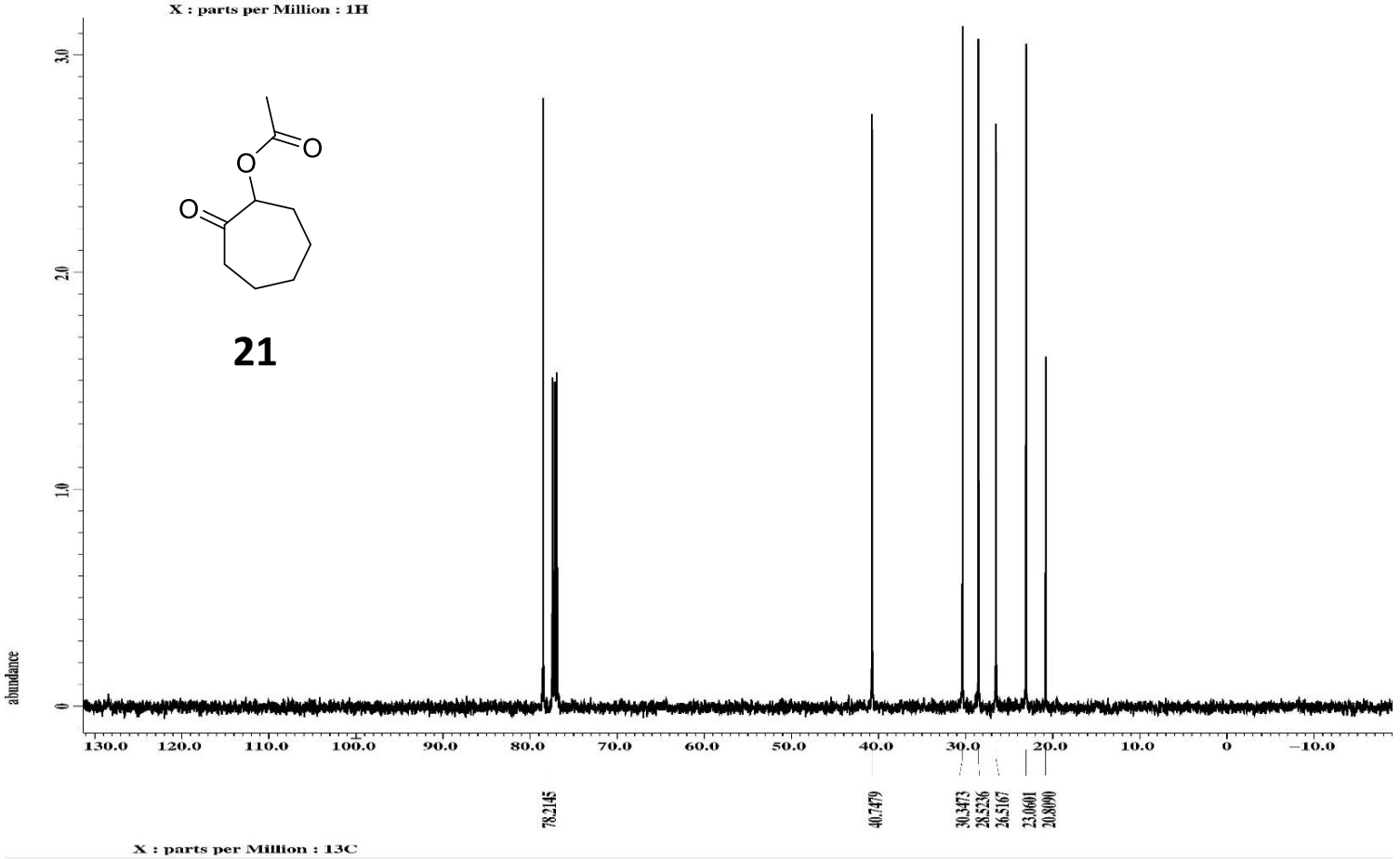

17
abundance
0.98
2.37
1.34
1.06
2.02
3
5.0699
5.0671
5.0495
2.4175
2.4139
2.3468
2.3320
2.3306
2.2508
2.2317
2.1187
2.1124
2.0820
1.8490
1.8461
1.8320
1.8278
X : parts per Million : 1H

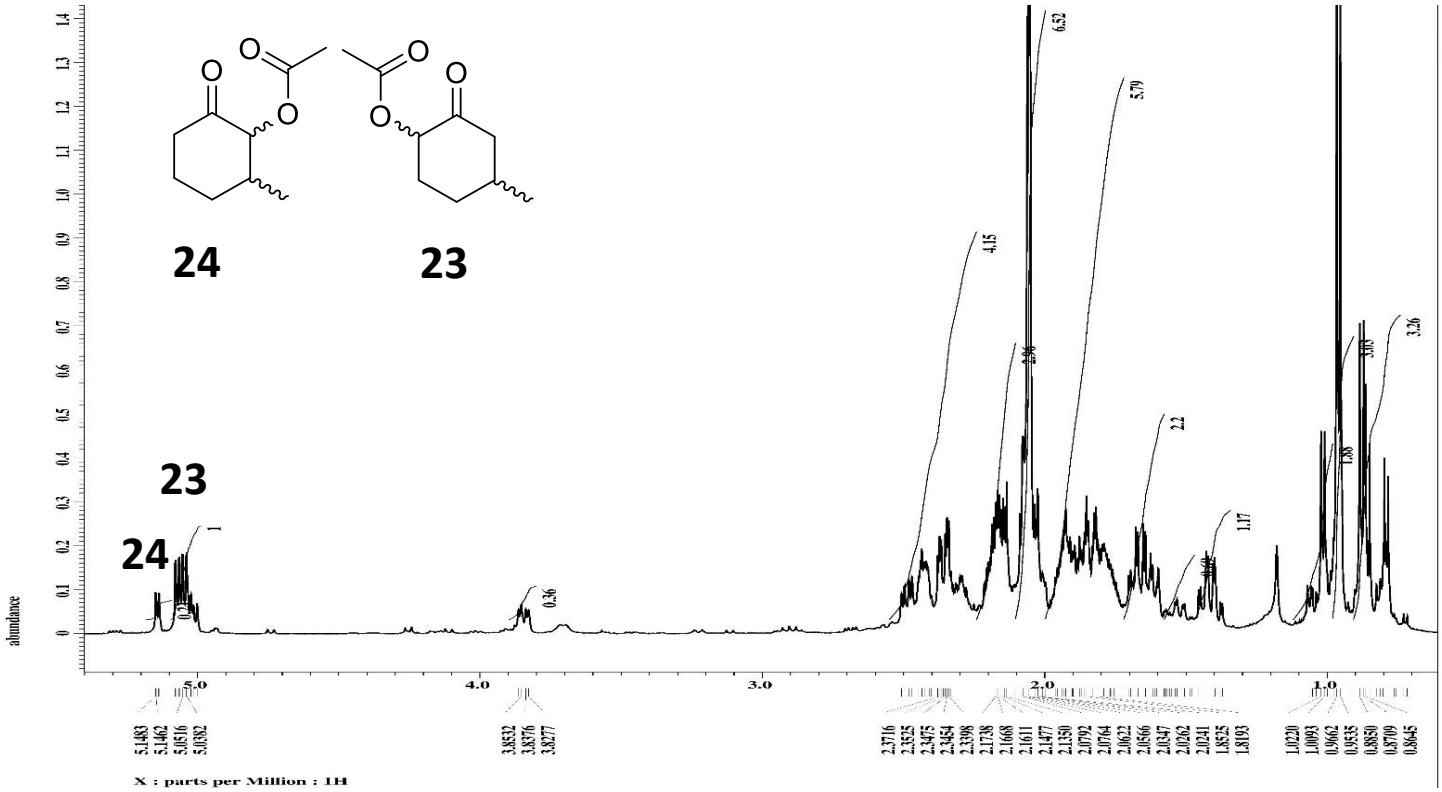

17
abundance
212.6209
170.3165
75.8032
34.9257
28.4778
20.8548
17.1844
X : parts per Million : 13C

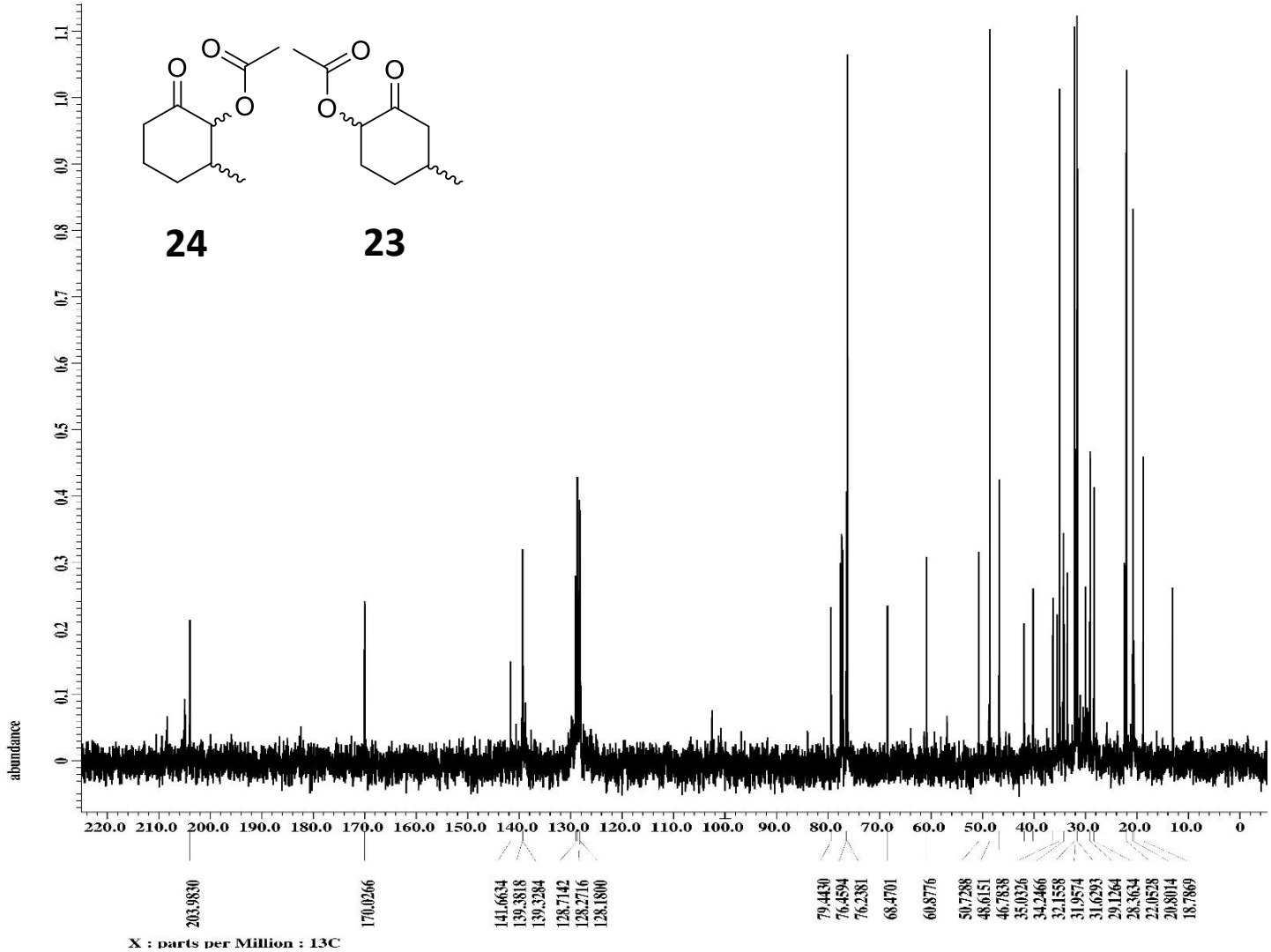

19
abundance
2.86
2.04
1.33
1.31
1.27
1.25
1.23
1.19
1
5.0
4.0
3.0
2.0
5.1038
5.1024
5.0925
2.4245
2.4203
2.3419
2.3299
2.3278
2.2381
2.0863
2.0234
2.0178
1.9097
1.9055
1.7028
1.6993
1.6922
1.5686
1.5601
1.5425
X : parts per Million : 1H

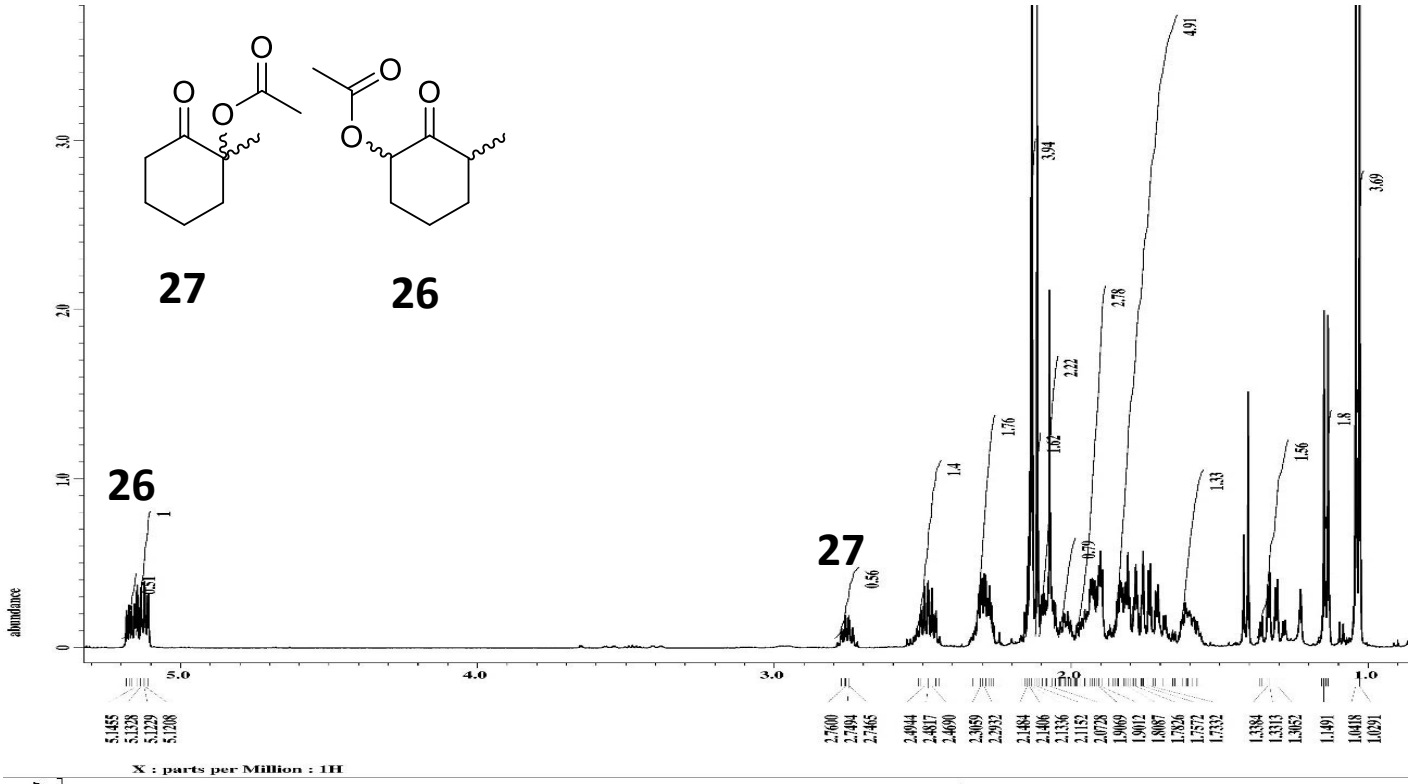

19
abundance
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
204.6774
170.0647
76.6197
40.7327
33.1096
27.2111
23.7926
20.7861
X : parts per Million : 13C

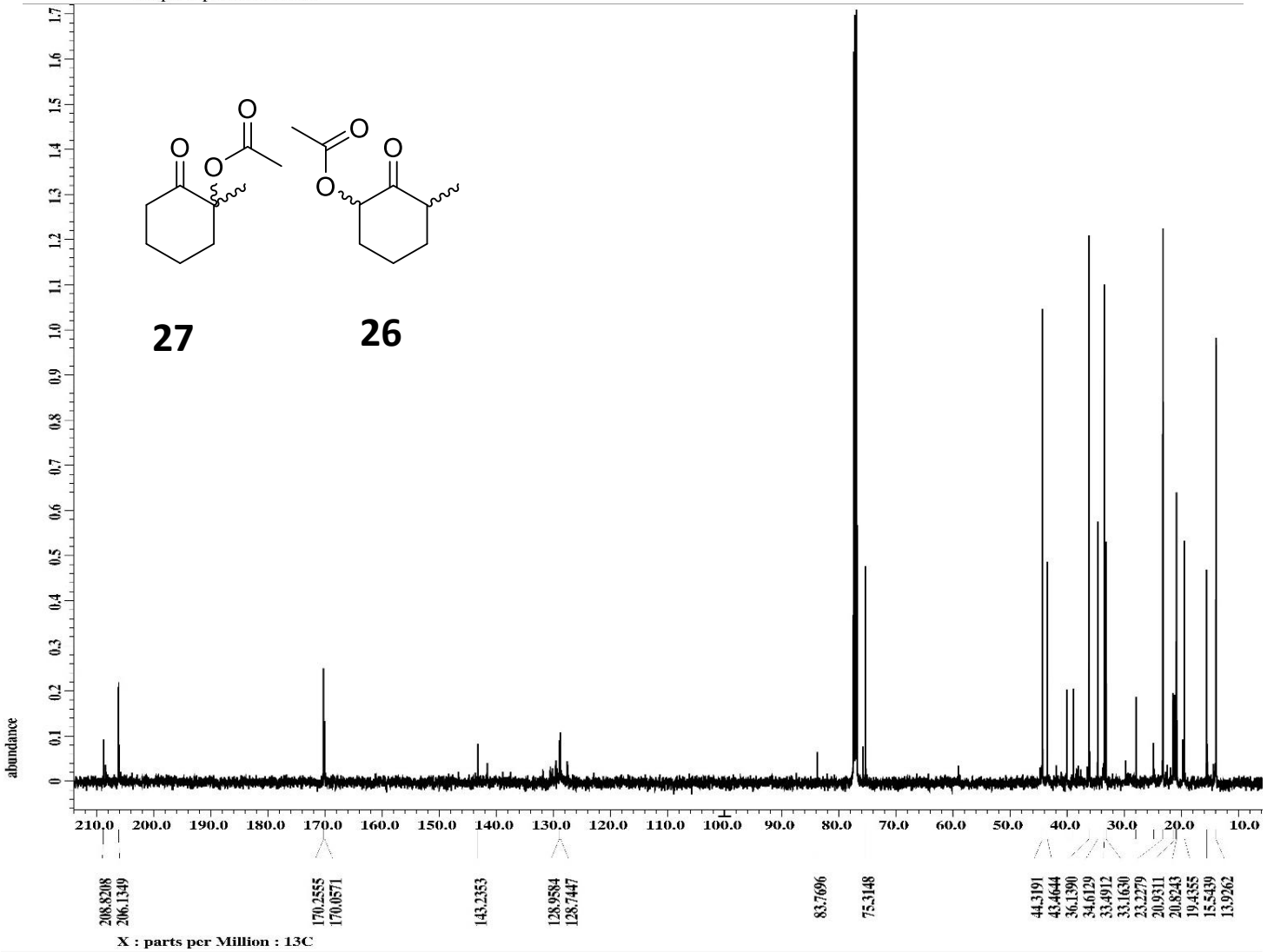

21
abundance
1
3.52
3.09
2.44
1.31
1.31
1.25
1.19
1.23
5.1971
5.1844
5.1773
2.6180
2.5848
2.5735
2.4069
2.3850
2.3836
2.0905
1.9443
1.9380
1.7946
1.7063
1.7007
1.6844
1.6809
1.6731
1.3419
1.3221
1.3031

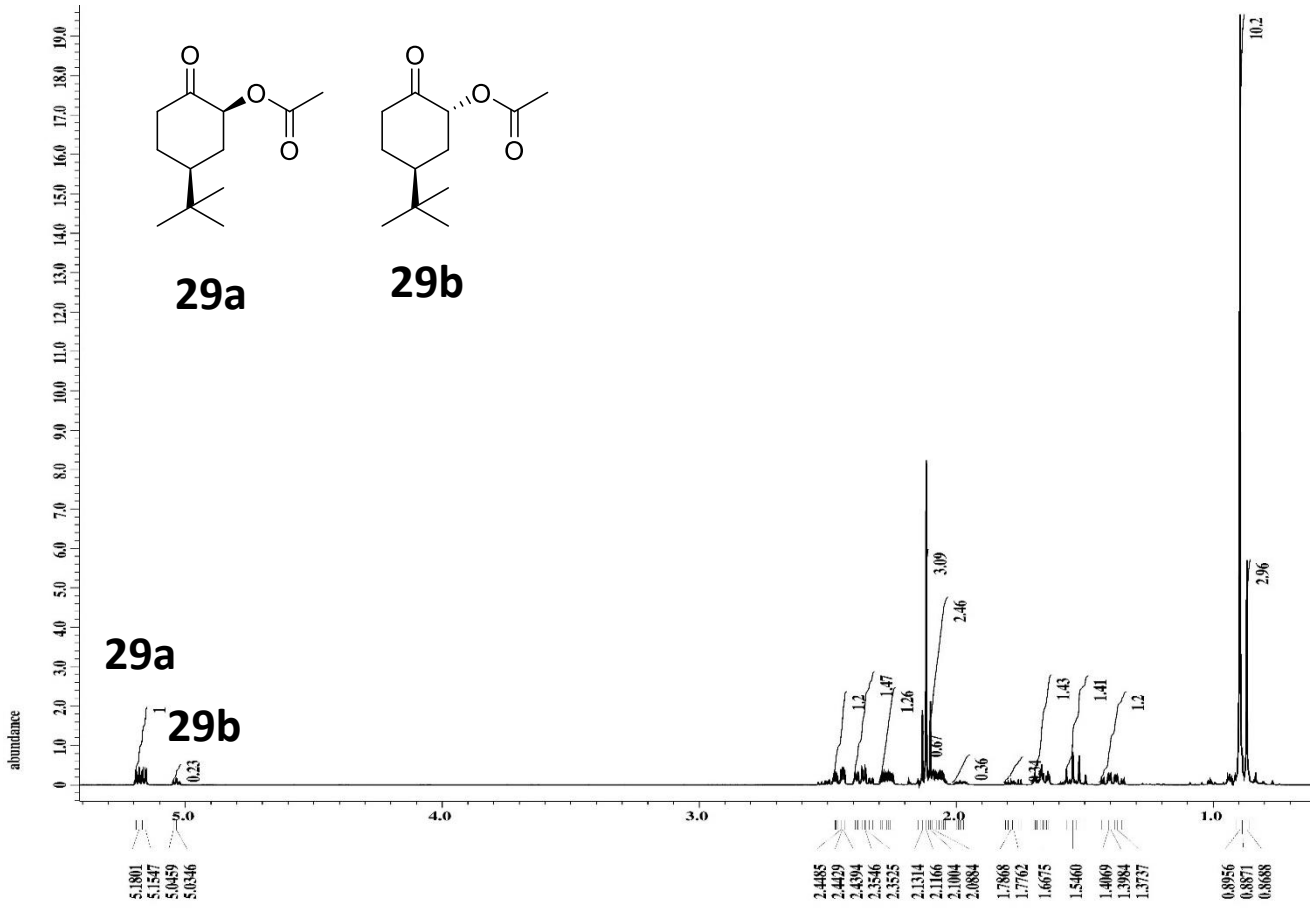

X : parts per Million : 1H
21
abundance
78.2145
40.7479
30.3473
28.5236
26.5167
23.0601
20.8090
X : parts per Million : 13C

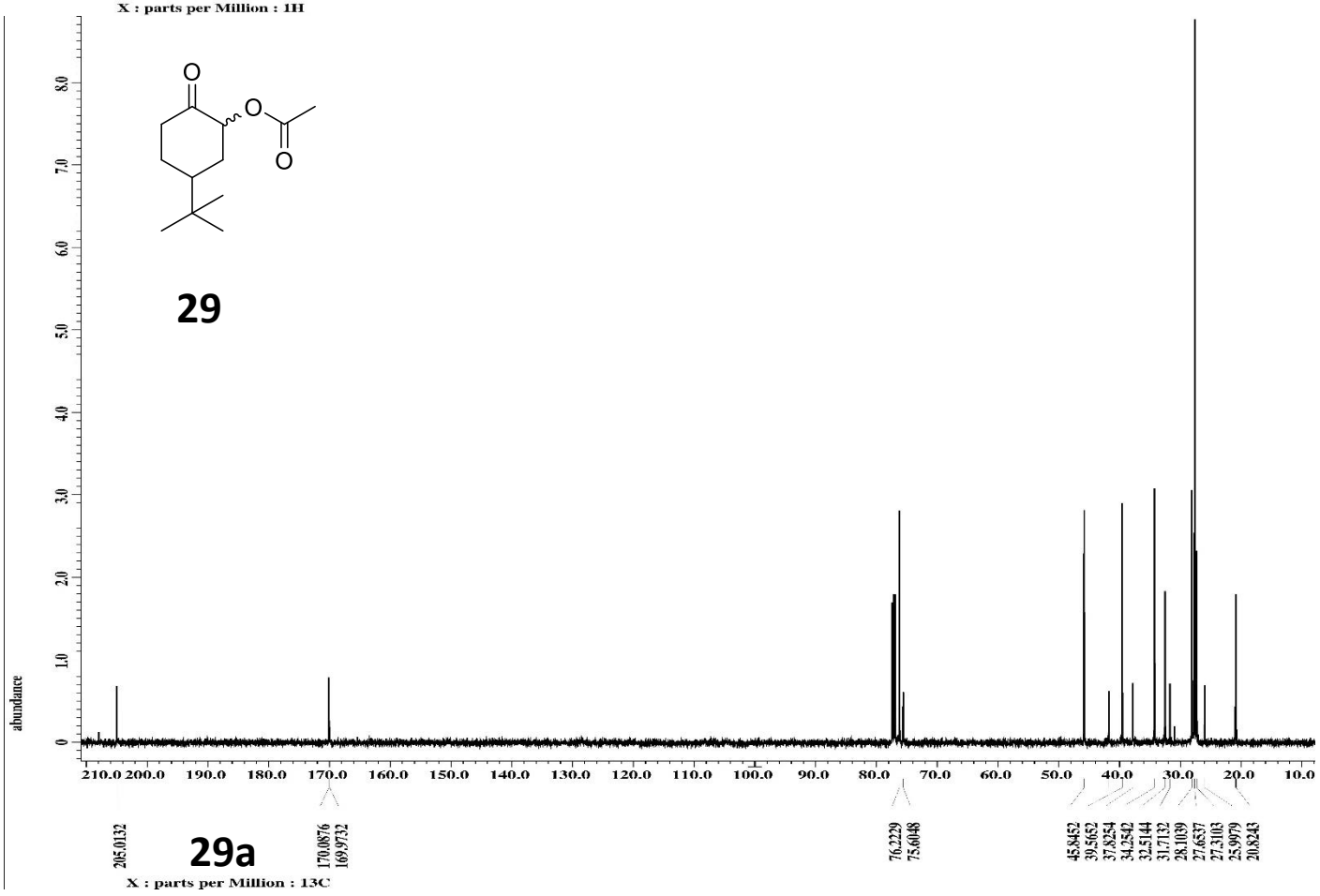

24
23
23
24
abundance
X : parts per Million : 1H

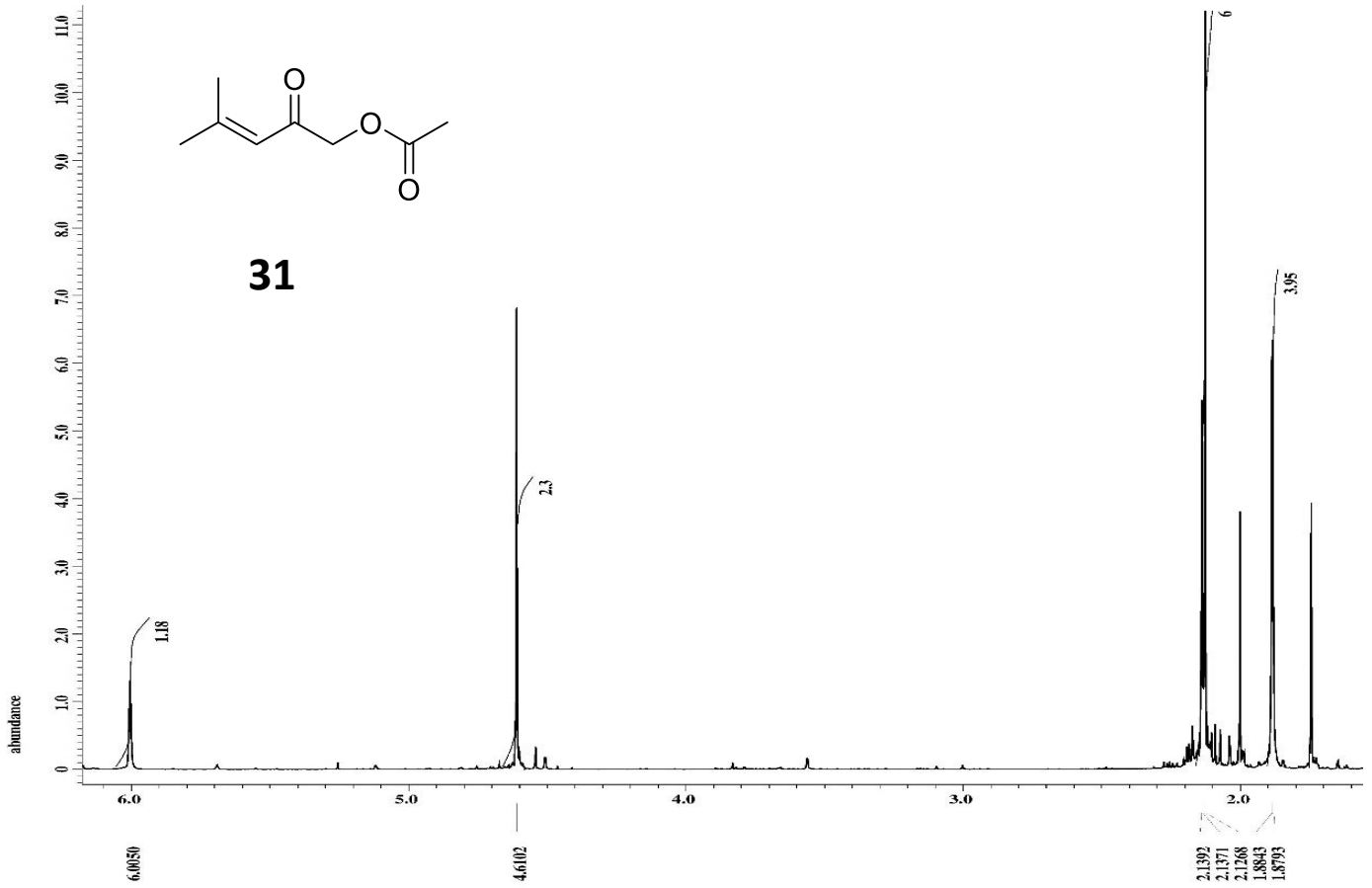

24
23
abundance
X : parts per Million : 13C

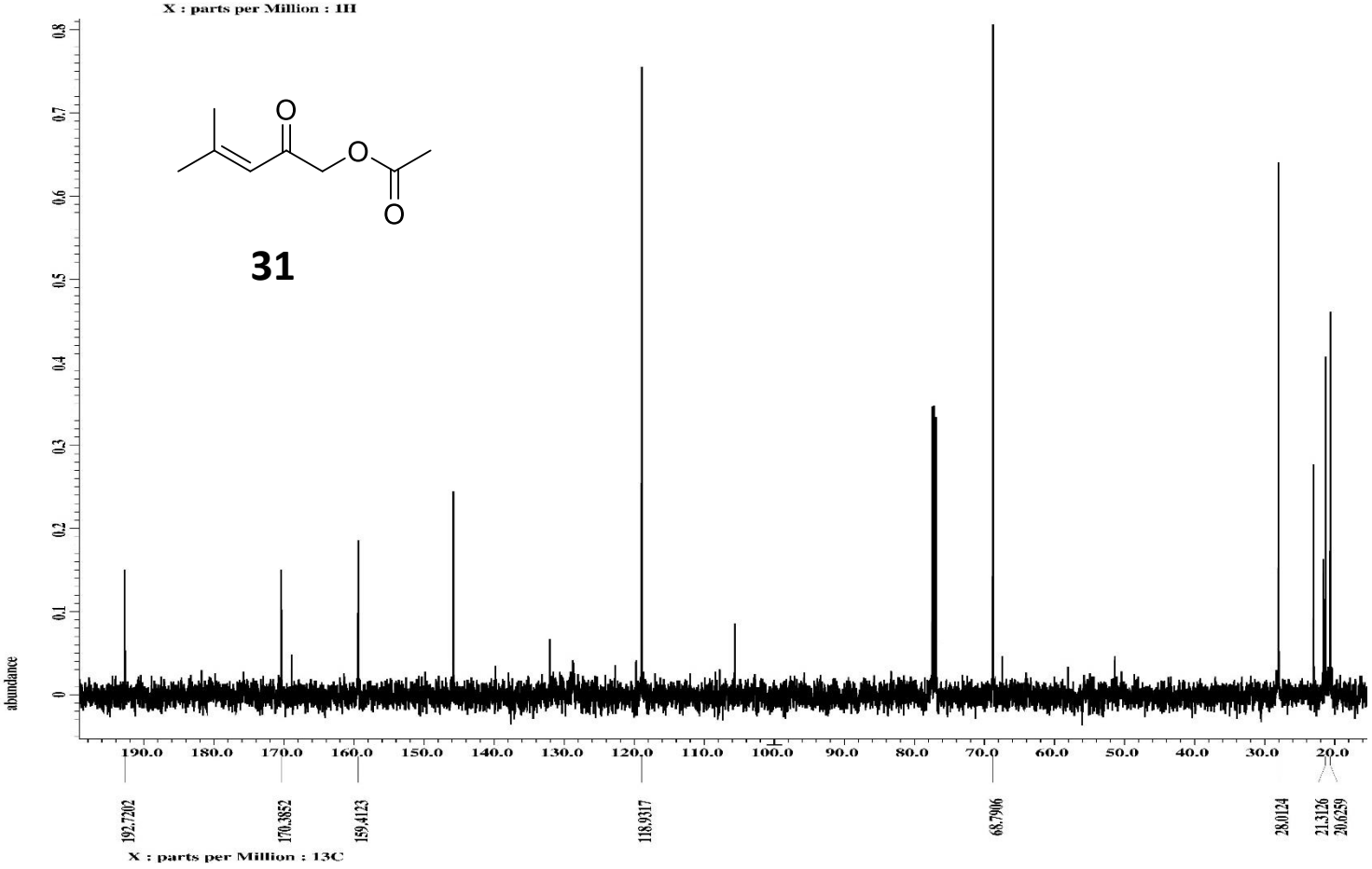

27
26
26
27
abundance
X : parts per Million : 1H

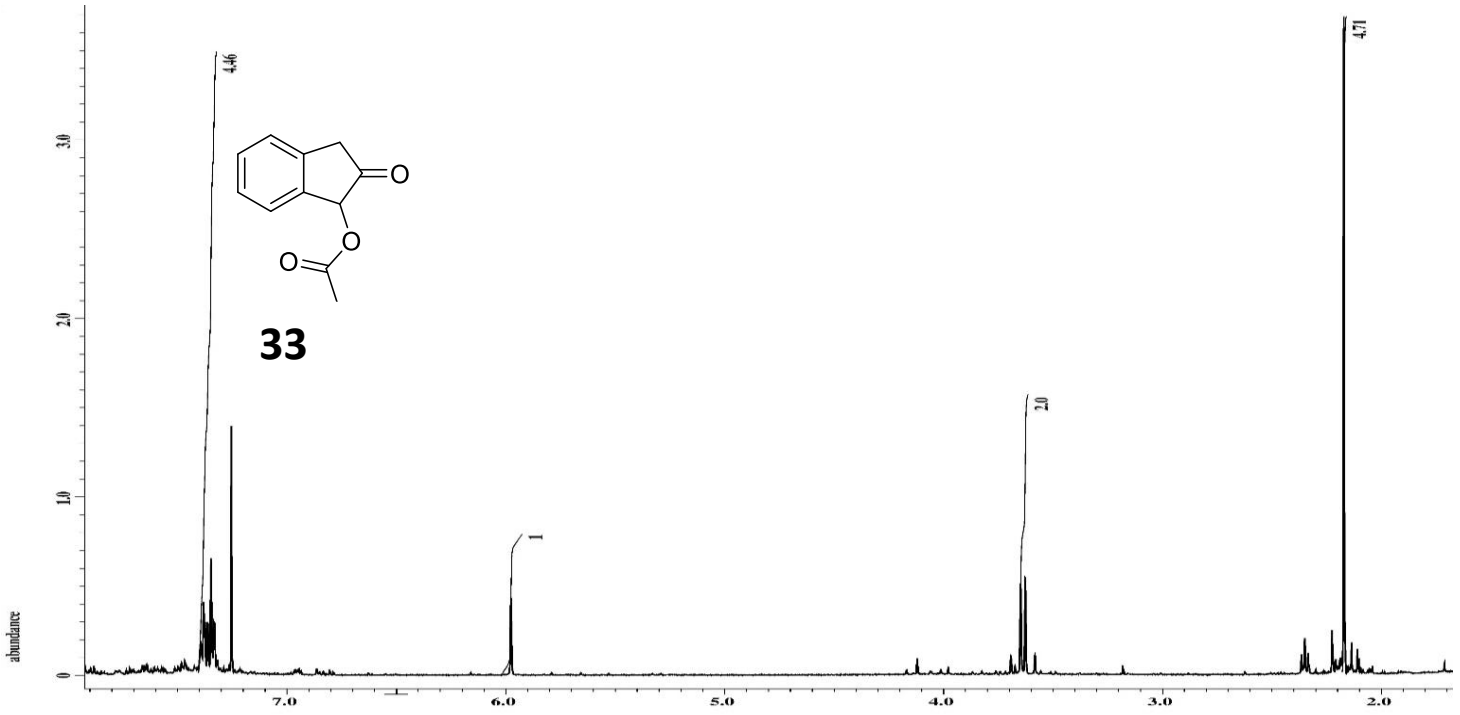

27
26
abundance
X : parts per Million : 13C


29a
29b
29a
29b
abundance
X : parts per Million : 1H

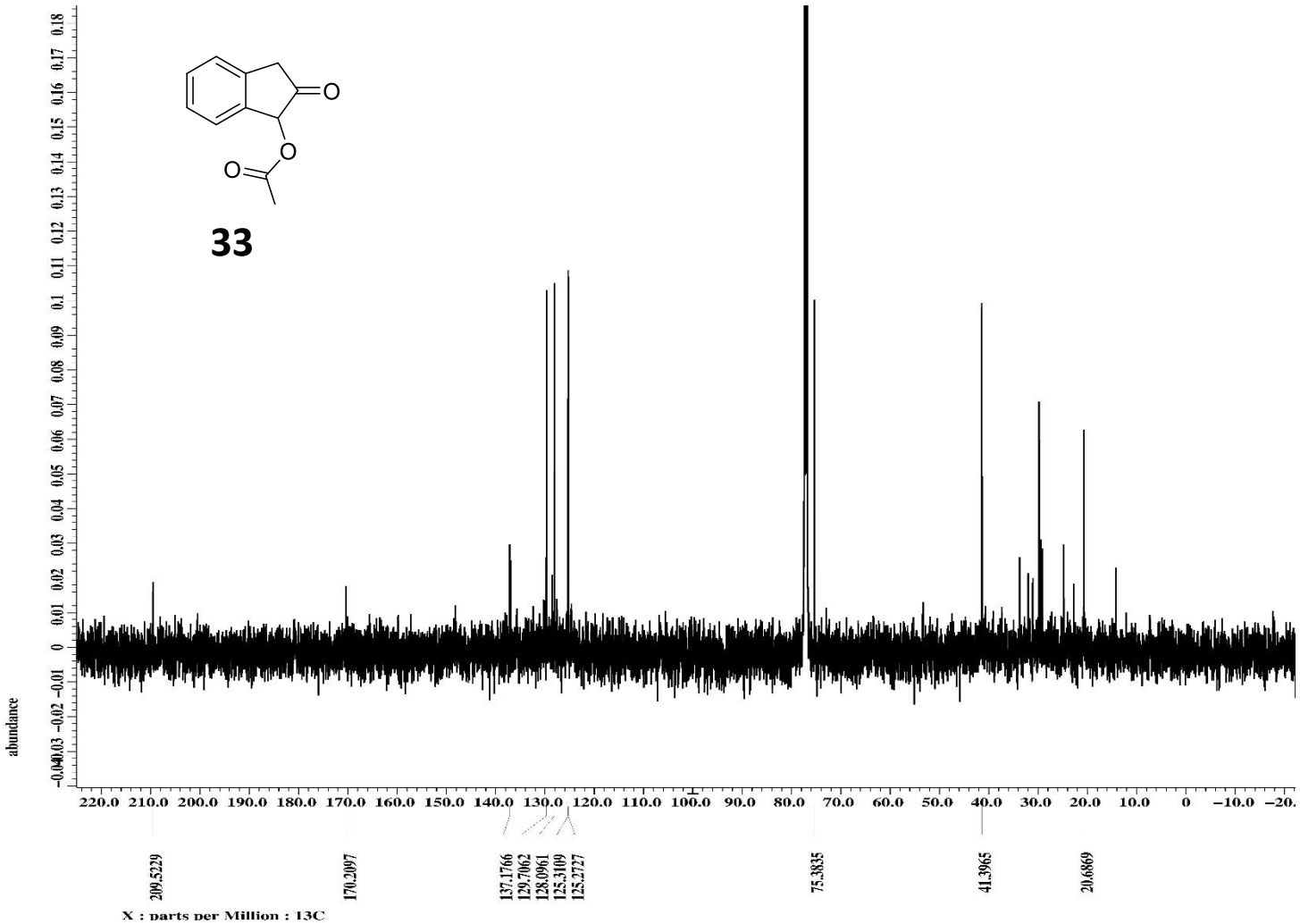

29
29a
abundance
X : parts per Million : 13C


31
abundance
6.0
5.0
4.0
3.0
2.0
1.18
2.3
3.95
6.00350
4.6102
2.1392
2.1371
2.1268
1.8843
1.8793
X : parts per Million : 1H

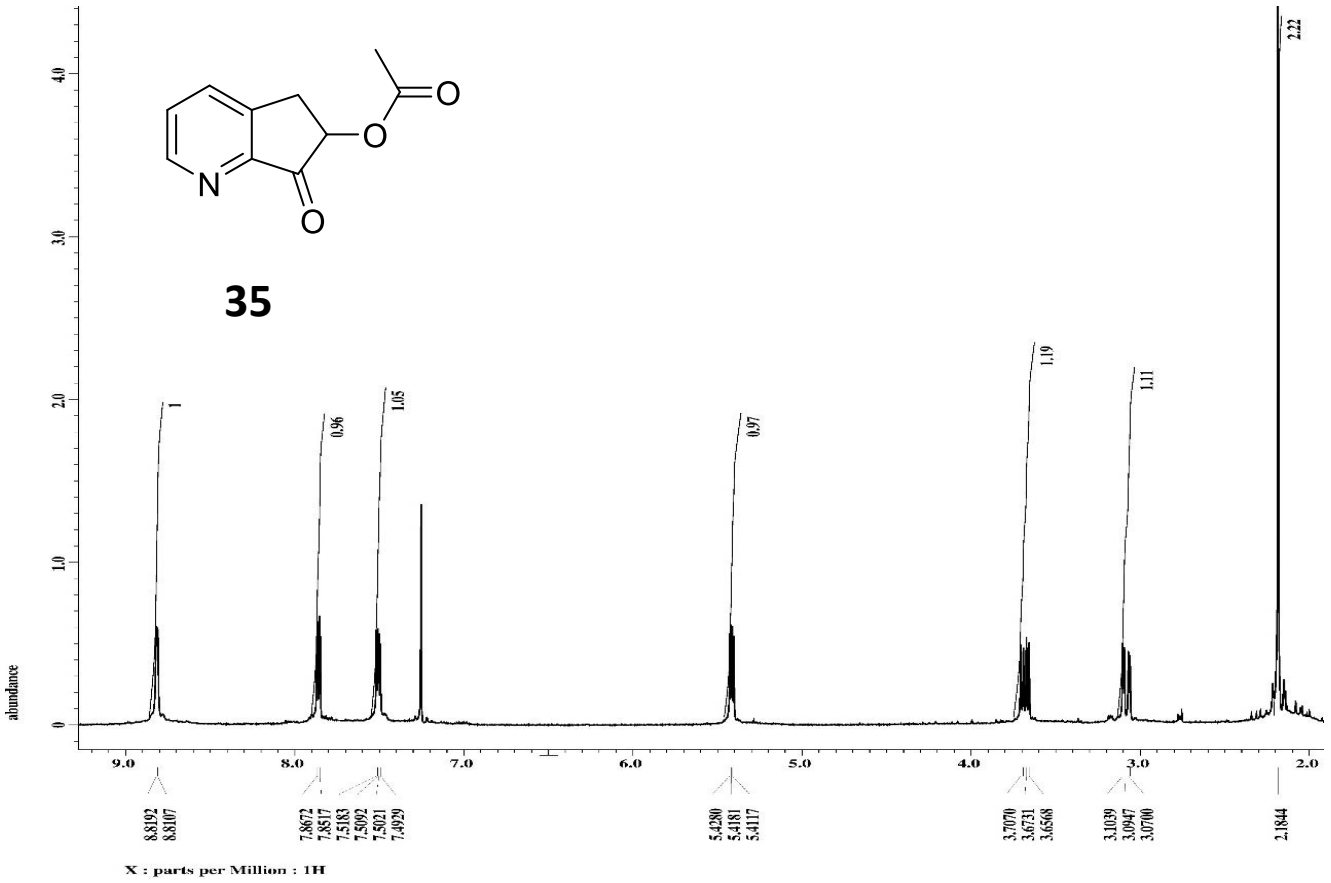

31
abundance
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
192.7202
170.3852
159.4123
118.9317
68.7906
28.0124
21.3126
20.6259
X : parts per Million : 13C

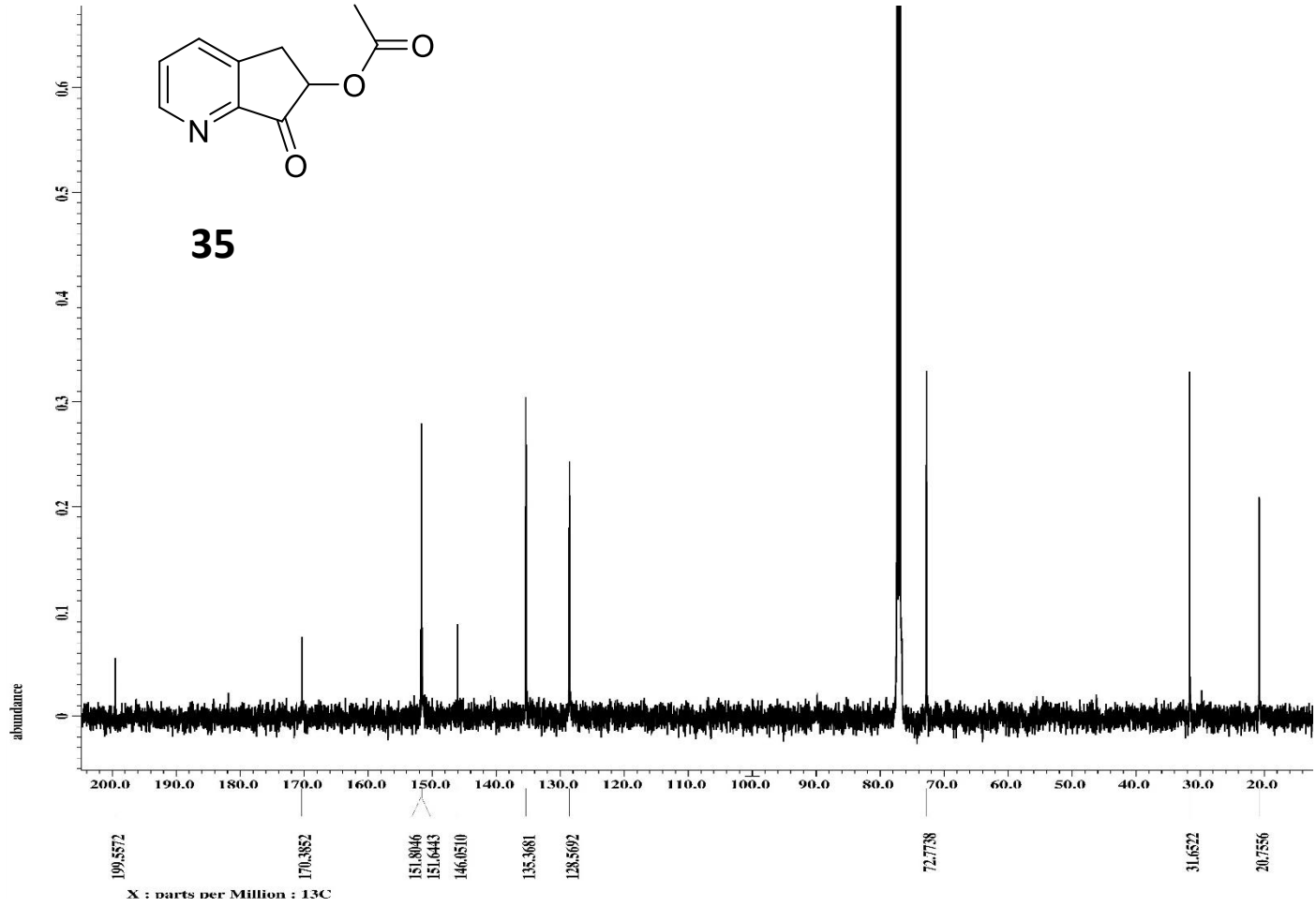

4.46
4.71
2.0
1
abundance
7.0
6.0
5.0
4.0
3.0
2.0
X : parts per Million : 1H

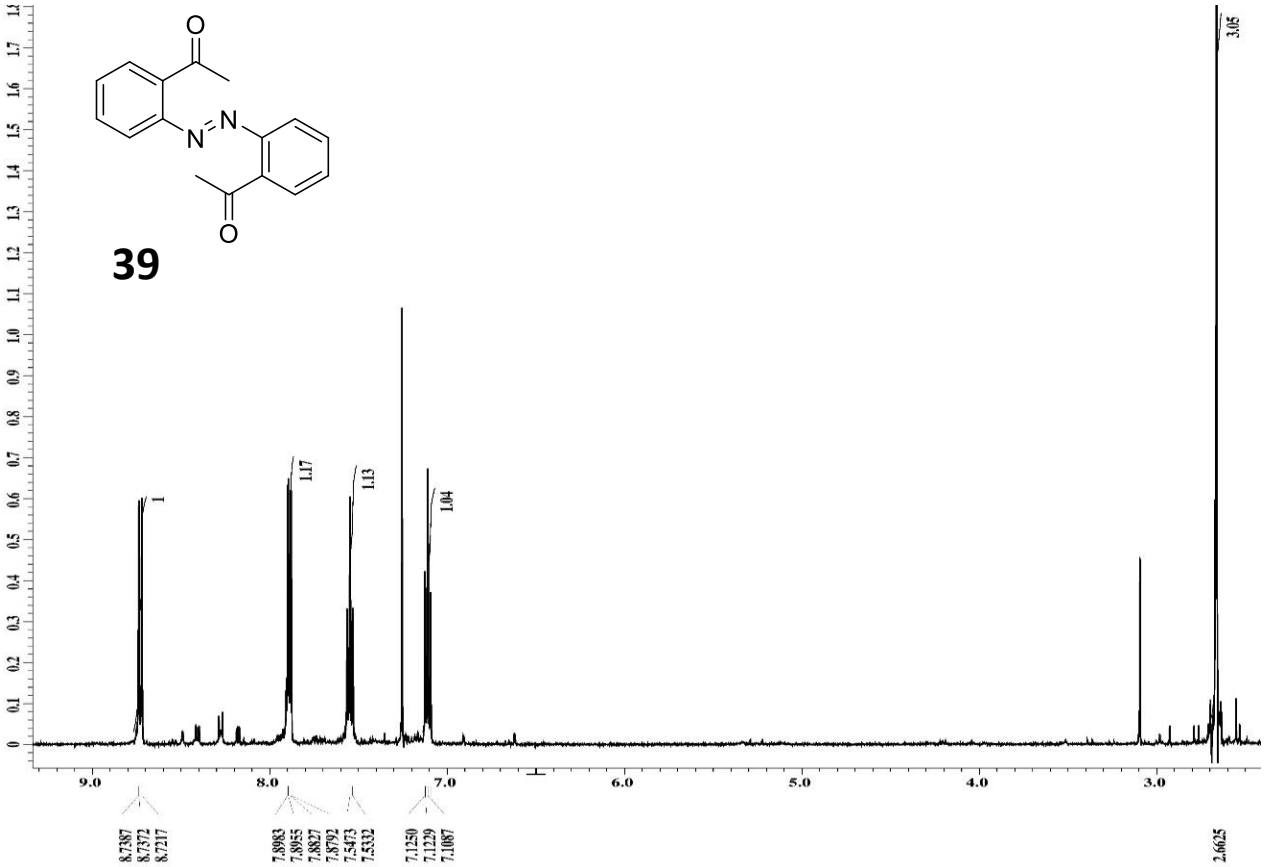
33

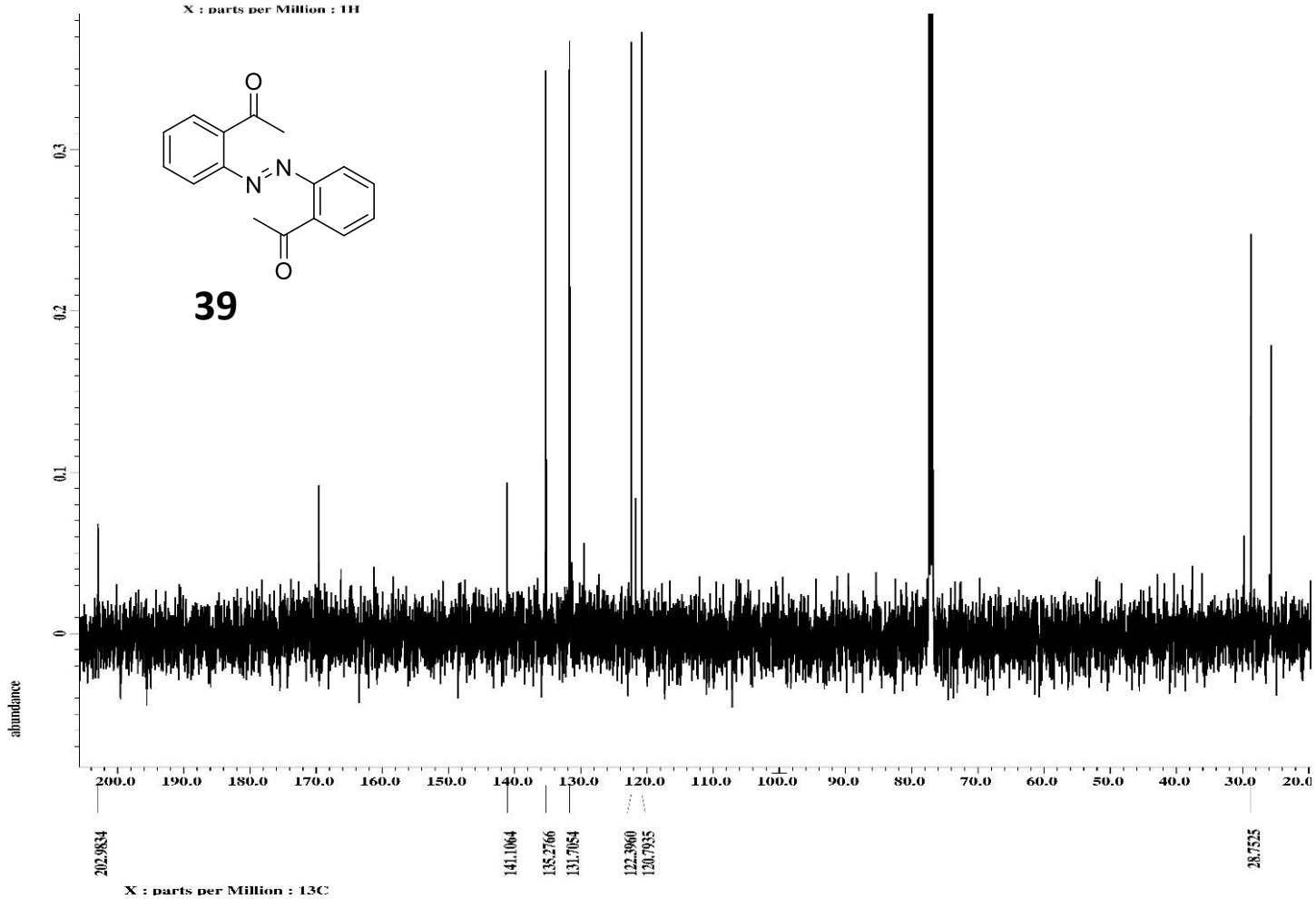

abundance
220.0
200.0
180.0
160.0
140.0
120.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
209.5229
170.2097
137.1766
129.7062
128.0961
125.3109
125.2727
75.3835
41.3965
20.6869
X : parts per Million : 13C

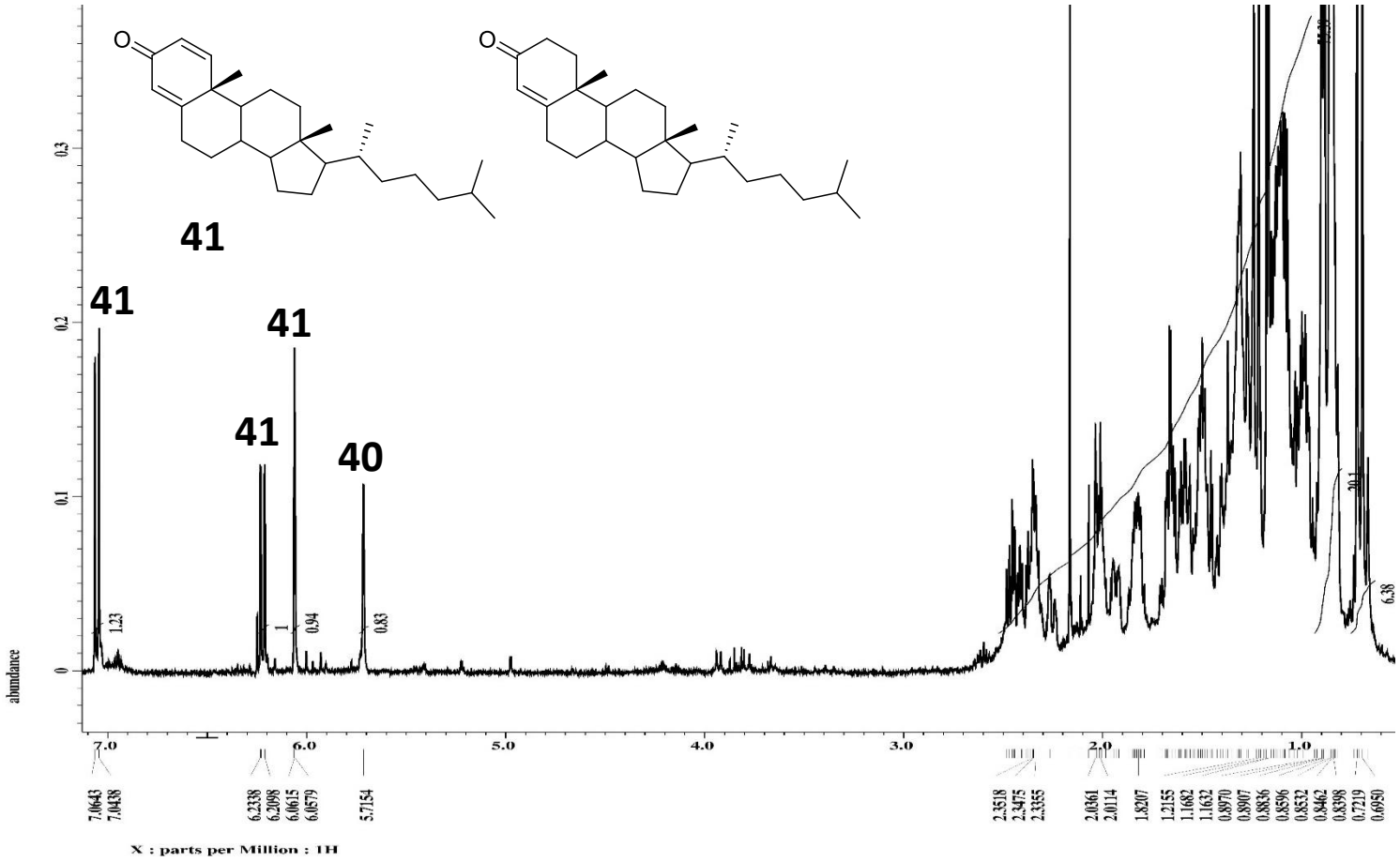
33

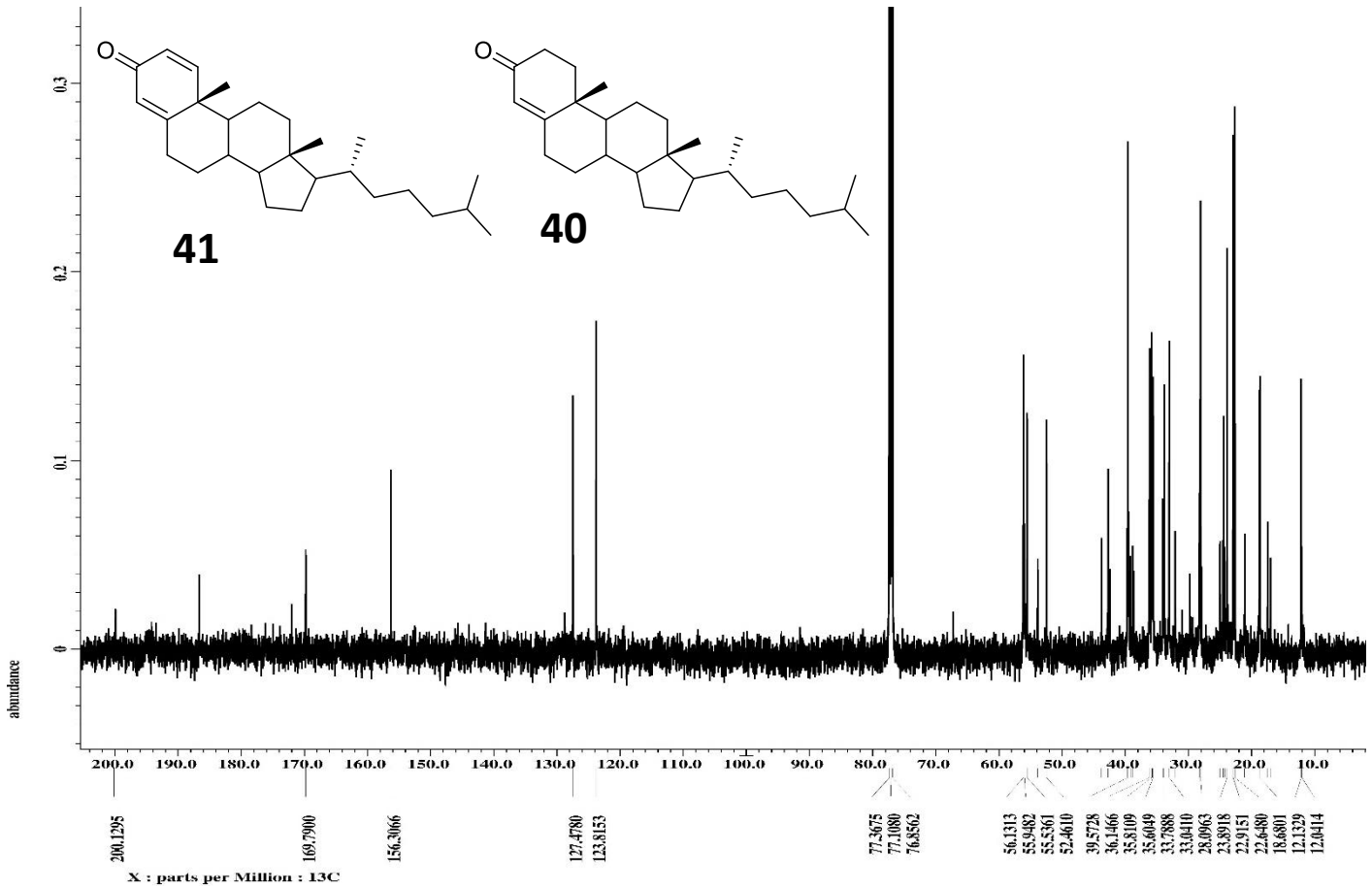

35
abundance
X : parts per Million : 1H

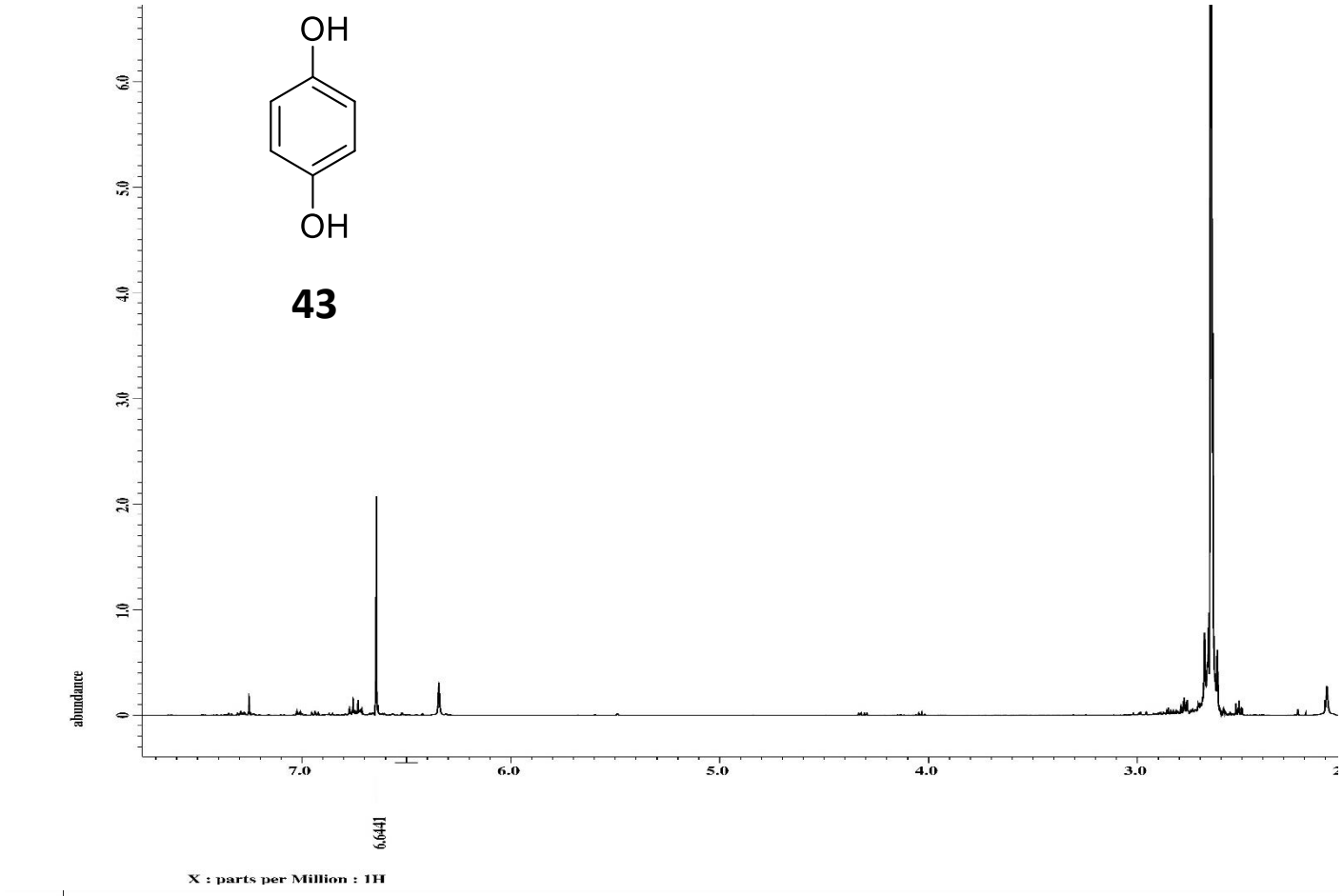

35
abundance
X : parts per Million : 13C

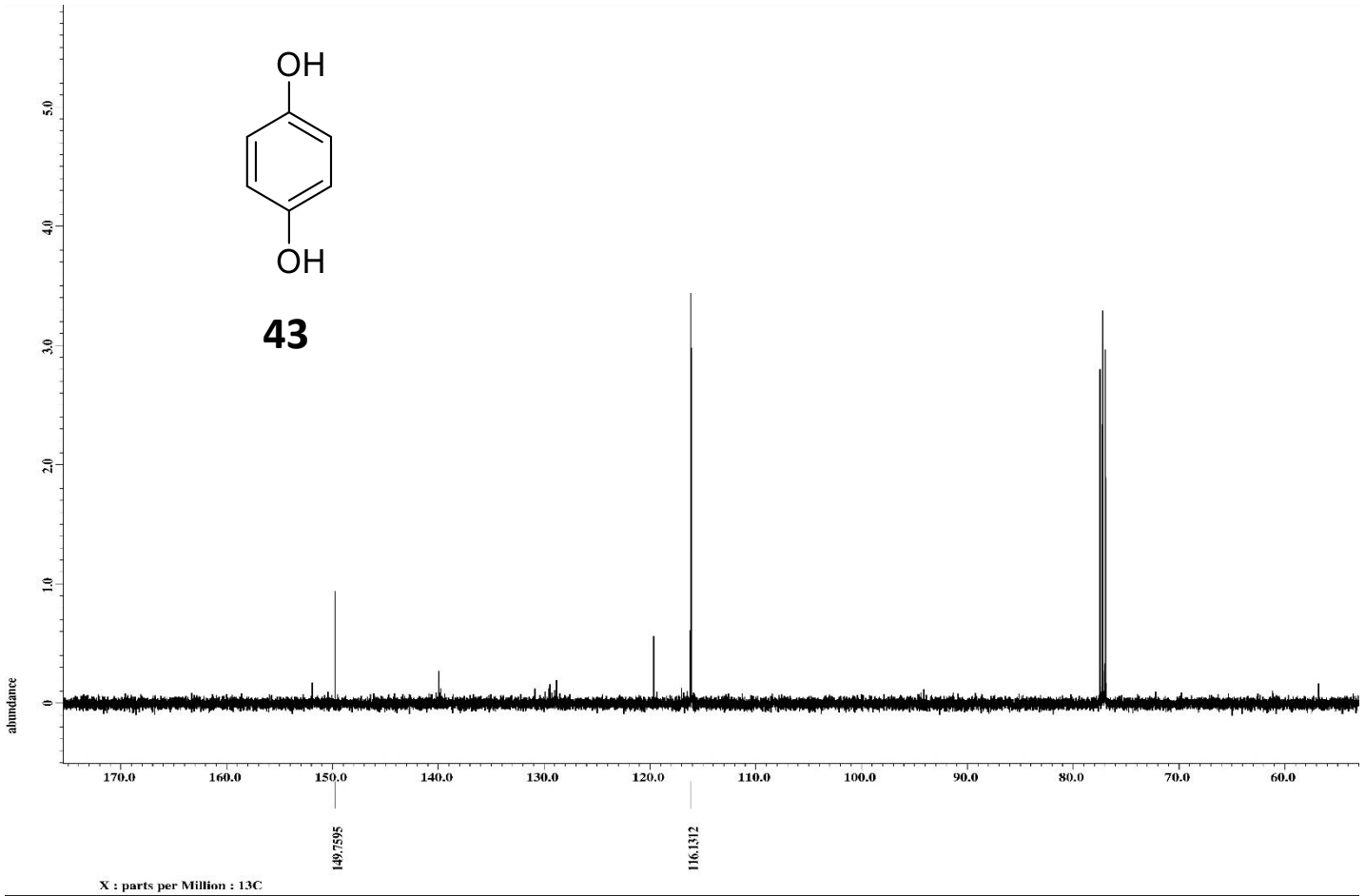

39
1
1.17
1.13
1.04
3.05
8.7387
8.7372
8.7217
7.8983
7.8955
7.8827
7.8792
7.5473
7.5332
7.1250
7.1229
7.1087
2.6625


X : parts per Million : 1H
39
abundance
202.9834
141.1064
135.2766
131.7054
122.3960
120.7935
28.7525
X : parts per Million : 13C

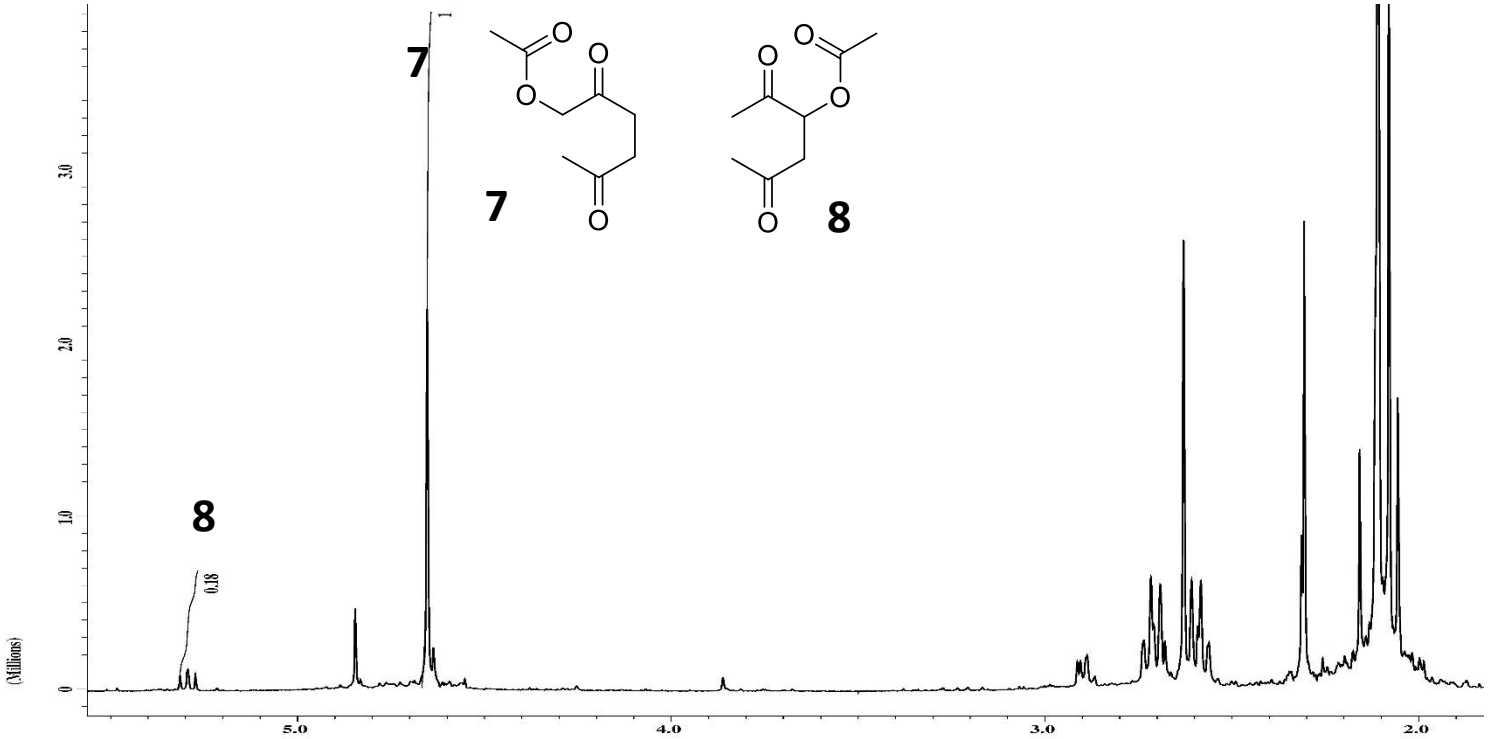

41
41
41
41
40
abundance
1.23
1
0.94
0.83
6.38
7.0643
7.0438
6.2338
6.2098
6.0615
6.0579
5.7154
2.3518
2.3475
2.3355
2.0361
2.0114
1.8207
1.2155
1.1682
1.1632
0.8970
0.8907
0.8836
0.8596
0.8532
0.8462
0.8398
0.7219
0.6950
X : parts per Million : 1H

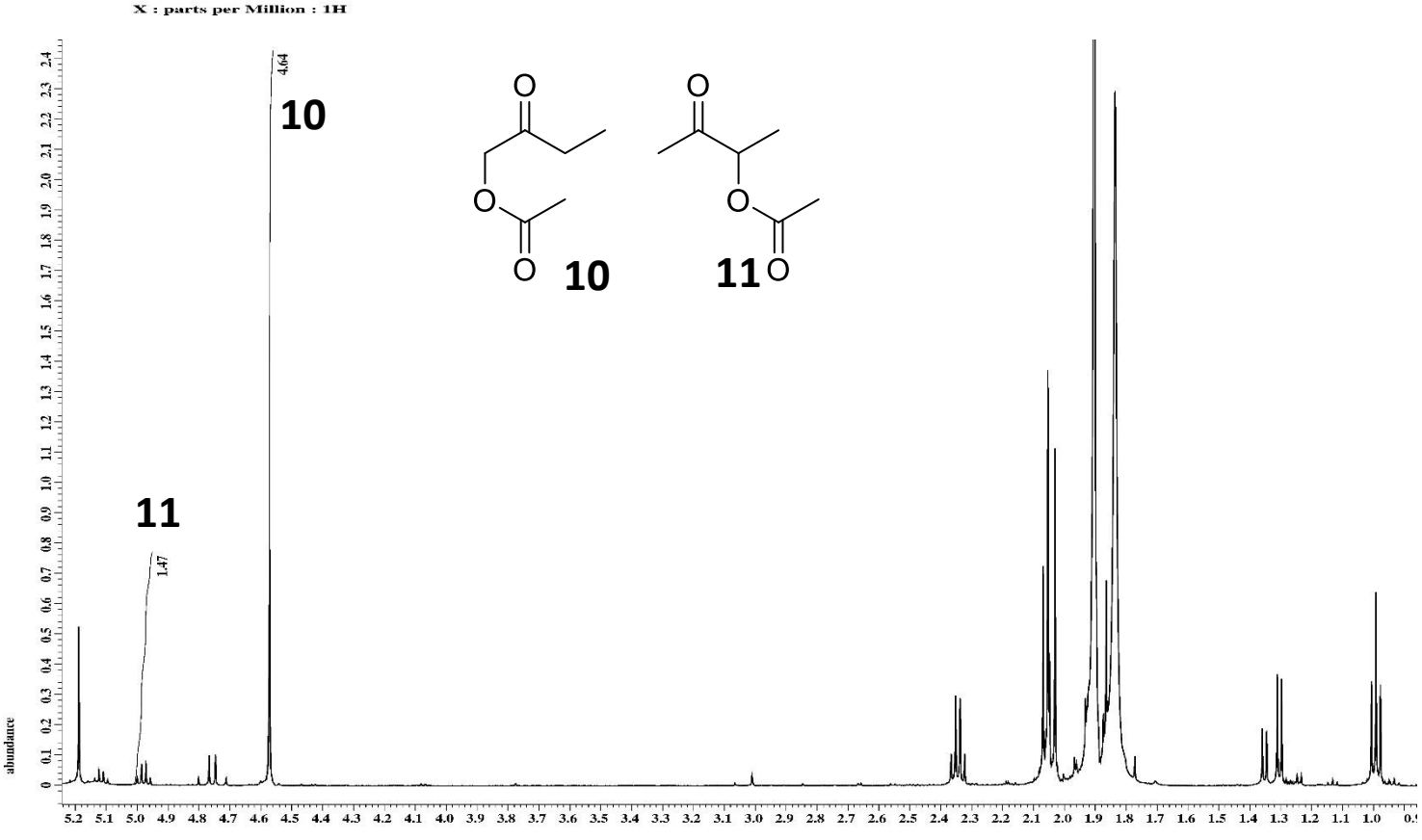

41
40
abundance
200.1295
169.7900
156.3066
127.4780
123.8153
77.3675
77.1080
76.8562
56.1313
55.9482
55.5361
52.4610
39.5728
36.1466
35.8109
35.6049
33.7888
33.0410
28.0963
23.8918
22.9151
22.6480
18.6801
12.1329
12.0414
X : parts per Million : 13C


OH
OH
43
abundance
6.0
5.0
4.0
3.0
2.0
1.0
0
7.0
6.0
5.0
4.0
3.0
6.6441
X : parts per Million : 1H

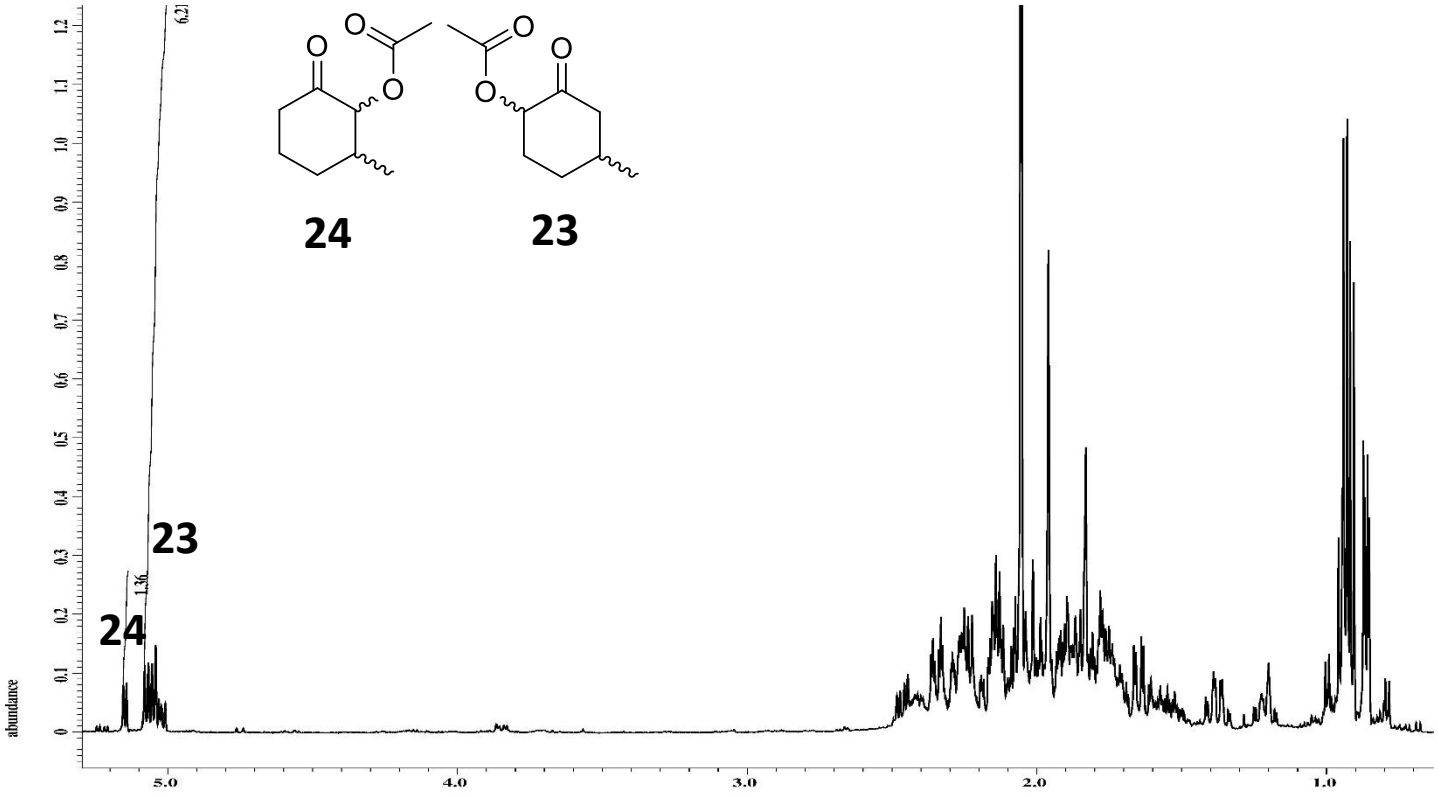

OH
OH
43
abundance
5.0
4.0
3.0
2.0
1.0
0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
149.7595
116.1312
X : parts per Million : 13C

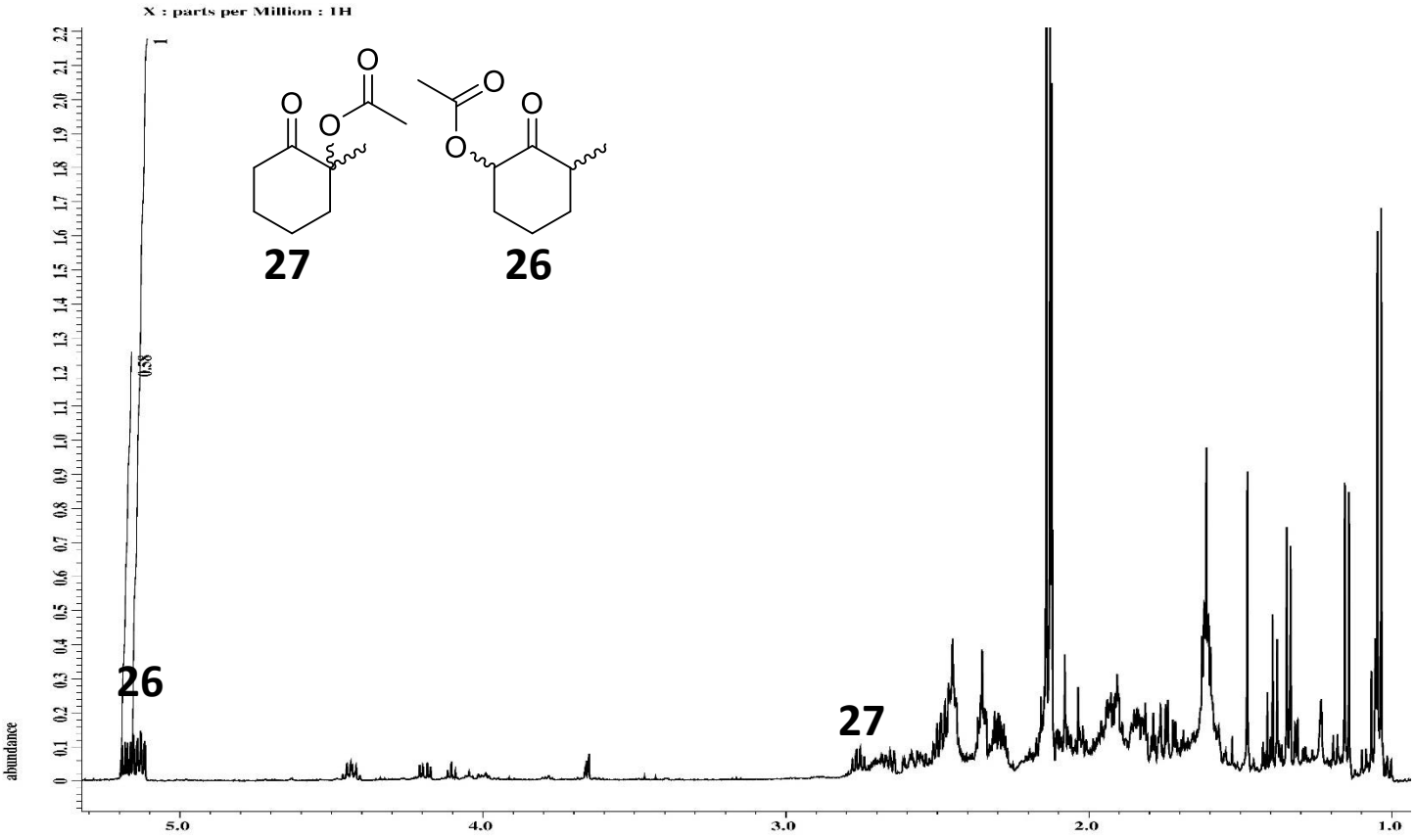
Crude reaction products



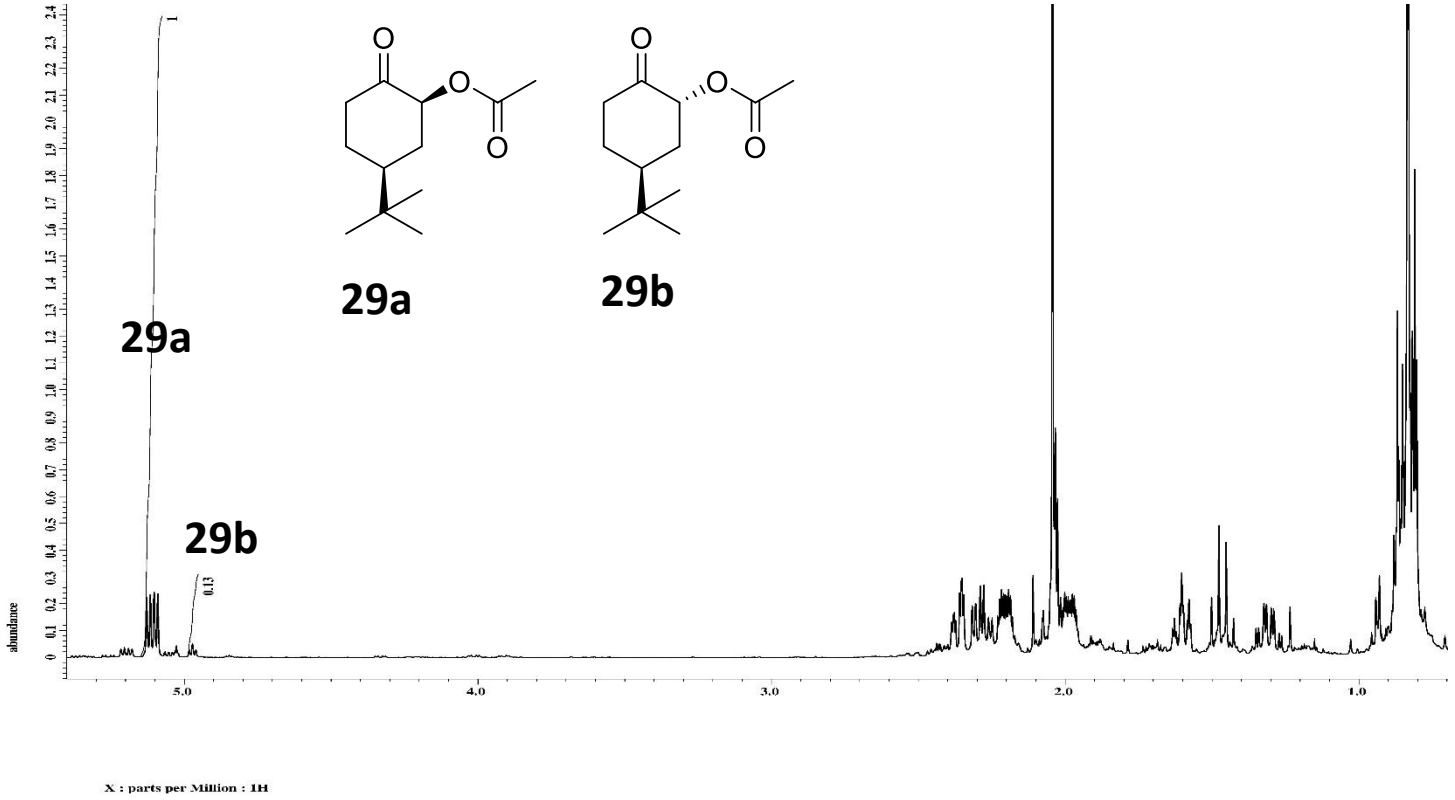


X : parts per Million : 1H





X : parts per Million : 1H


29a
29b
29a
29b
abundance
0.13
5.0
4.0
3.0
2.0
1.0
X : parts per Million : 1H