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Supporting Information for

Reaction of *N*-Propargylic *β*-enaminones with Acetylene Dicarboxylates: Catalst-Free Synthesis of 3-Azabicyclo[4.1.0]hepta-2,4-dienes†

Kommuru Goutham,[a,b] Vemu Nagaraju,[a] Surisetti Suresh,[c] Pallepogu Raghavaiah,[d] and Galla V. Karunakar,*[a,b]

[a]*Crop protection Chemicals Division,*
CSIR-Indian Institute of Chemical Technology,
Hyderabad, 500007, India.
gallavk@iict.res.in
[b]*Academy of Scientific and Innovative Research,*
New Delhi, 110001, India
[c]*Organic and Biomolecular Chemistry Division,*
CSIR-Indian Institute of Chemical Technology,
Hyderabad, 500007, India.
[d]*School of Chemistry, University of Hyderabad, Hyderabad, 500046, India.*

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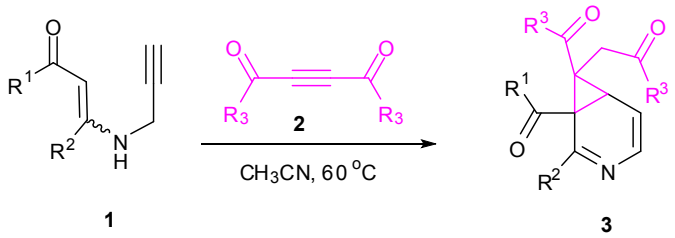
†Electronic Supplementary Material (ESI) for Chemical Communications

1.1 General

Reactions were carried out in oven dried reaction flasks under nitrogen atmosphere and also solvents and reagents were transferred by oven-dried syringes to ambient temperature. TLC was performed on Merck silica gel aluminium sheets using UV as a visualizing agent and a 0.5% aqueous potassium permanganate solution and heat as developing agents. Solvents were removed under reduced pressure. Columns were packed as slurry of silica gel in hexane and ethyl acetate solvent mixture. The elution was assisted by applying pressure with an air pump. ^{13}C NMR spectra were recorded on 75 and 125 MHz spectrometers. ^{1}HNMR spectra were recorded on 300 and 500 MHz spectrometers in appropriate solvents using TMS as internal standard. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, dd = double doublet, t = triplet, m = multiplet. All reactions were performed under nitrogen atmosphere with freshly distilled and dried solvents. All solvents were distilled using standard procedures. Unless otherwise noted, reagents were obtained from Aldrich, Alfa Aesar, and TCI used without further purification. *N*-propargyl β-enaminones (**1a-1h**) were prepared by following the reported procedure.[1]

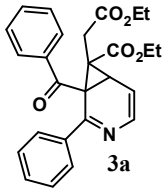
1. A. S. Karpov and T. J. J. Mu1ller, *Org. Lett.,* 2003, **5**, 3451.

1.2 General procedure for synthesis of 3-azabicyclo[4.1.0]hepta-2,4-dienes



In a 25mL round bottomed flask taken *N*-propargyl β-enaminones **1** (0.383 mmol, 1 equiv.), and kept for high vacuum for 10-15 min and then dissolved in 5mL of dry acetonitrile. Reaction temperature was raised to 50 °C and then added diethyl acetylenedicarboxylate **2** (0.383 mmol, 1 equiv), drop wise under nitrogen atmosphere and reaction mixture was stirred for 5 h. After completion of reaction (monitered by TLC) it was cooled to room temperature. The crude reaction mixture was concentrated under reduced pressure. The desired 3-azabicyclo[4.1.0]hepta-2,4-diene **3** compound was obtained after purification by column chromatography on silica gel with hexane/ethyl acetate (10/1) as the eluent.

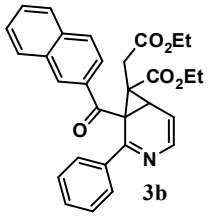
1.3 Spectroscopic data for 3-azabicyclo[4.1.0]hepta-2,4-dienes (3a-l)



3a

ethyl 1-benzoyl-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

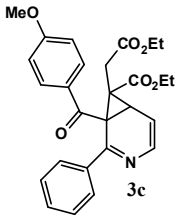
R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 91%; white solid; Melting Point: 121-123 ^oC; ^1H NMR (300 MHz, $CDCl_3$): δ 7.65-7.60 (m, 2H), 7.47-7.35 (m, 4H), 7.33-7.21 (m, 5H), 6.10 (t, *J* = 12.842 Hz, 1H), 4.42-4.31 (m, 2H), 4.12 (q, *J* = 6.043, Hz, 2H), 3.60 (d, *J* = 6.043 Hz, 1H), 3.17 (d, *J* = 18.129 Hz,1H), 1.72 (d, *J* = 18.129 Hz, 1H), 1.33 (t, *J* = 6.043, Hz, 3H), 1.22 (t, *J* = 6.043 Hz, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.8, 171.4, 170.5, 161.6, 138.1, 136.0, 133.3, 130.9, 128.46, 128.43, 128.3, 128.2, 113.1, 62.1, 60.6, 46.1, 34.7, 30.6, 19.6, 14.0, 13.9; HRMS (ESI): calcd for $C_{26}H_{26}$ NO_5 [M+H]+ 432.1805; found 432.1791.



3b

ethyl 1-(2-naphthoyl)-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 97%; white solid; Melting Point: 103-105 ^oC; ^1H NMR (300 MHz, $CDCl_3$): δ 7.95 (s, 1H), 7.83-7.73 (m, 2H), 7.71-7.62 (m, 3H), 7.58-7.44 (m, 4H), 7.25-7.17 (m, 3H), 6.16 (t, *J* = 12.841Hz, 1H), 4.44-4.33 (m, 2H), 4.13 (q, *J* = 7.176 Hz, 2H), 3.67 (d, *J* = 6.043 Hz, 1H), 3.21 (d, *J* = 17.751 Hz, 1H), 1.76 (d, *J* = 17.751 Hz, 1H), 1.34 (t, *J* = 7.176 Hz, 3H), 1.23 (t, *J* = 7.176 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) : δ 191.7, 171.4, 170.5, 161.7, 138.1, 136.1, 135.5, 133.6, 132.1, 130.9, 130.4, 129.7, 128.7, 128.49, 128.42, 128.2, 127.6, 126.6, 123.8, 113.2, 62.2, 60.6, 46.3, 34.9, 30.7, 19.7, 14.08, 14.01 ; HRMS (ESI): calcd for $C_{30}H_{28}NO_5$ [M+H]+ 482.1962, found 482.1946.

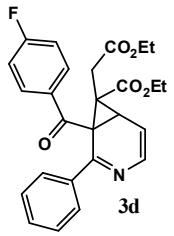


3c

ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-methoxybenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 95%; pale yellow oil; ^1H NMR (300 MHz, $CDCl_3$): δ 7.68-7.63 (m, 2H), 7.47-7.41 (m, 2H), 7.39-7.35 (m, 1H), 7.32-7.22 (m, 3H), 6.77-6.72 (m, 2H), 6.08 (t, *J* =

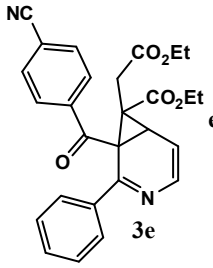
12.842 Hz, 1H), 4.35 (q, J = 6.798 Hz, 2H), 4.11 (q, J = 6.798 Hz, 2H), 3.78 (s, 3H), 3.56 (d, J = 6.043 Hz, 1H), 3.16 (d, J = 18.129 Hz, 1H), 1.70 (d, J = 18.129 Hz, 1H), 1.32 (t, J = 6.798 Hz, 3H), 1.21 (t, J = 6.798 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.0, 171.5, 170.5, 163.5, 161.8, 138.0, 135.8, 130.8, 130.7, 129.2, 128.3, 128.1, 113.6, 113.1, 62.0, 60.5, 55.2, 46.1, 34.6, 30.5, 19.4, 14.0, 13.9; HRMS (ESI): calcd for $C_{27}H_{28}$ N O_6 [M+H]+ 462.1911, found 462.1899.



ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3d

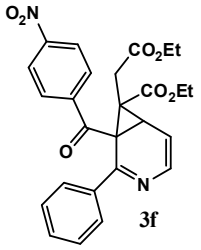
R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 89%; pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.65-7.60 (m, 2H), 7.50-7.42 (m, 2H), 7.41-7.36 (m, 1H), 7.33-7.22 (m, 3H), 6.98-6.89 (m, 2H), 6.09 (t, J = 12.841 Hz, 1H), 4.41-4.32 (m, 2H), 4.12 (q, J = 6.987Hz, 2H), 3.59 (d, J = 6.043 Hz, 1H), 3.16 (d, J = 17.751 Hz, 1H), 1.71 (d, J = 17.751 Hz, 1H), 1.33 (t, J = 6.987 Hz, 3H), 1.22 (t, J = 6.987 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 189.9, 171.0, 170.1, 166.9, 163.6, 161.0, 137.6, 135.7, 132.2, 130.7, 130.6, 128.1, 127.9, 115.5, 115.2, 112.7, 61.8, 60.3, 45.6, 34.4, 30.2, 19.4, 13.7, 13.6; HRMS (ESI): calcd for $C_{26}H_{25}FNO_5$ [M+H]+ 450.1711, found 450.1697.



ethyl 1-(4-cyanobenzoyl)-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3e

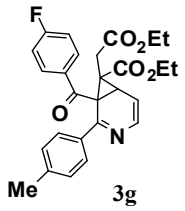
R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 84%; lemon yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60-7.53 (m, 4H), 7.50-7.45 (m, 2H), 7.43-7.39 (m, 1H), 7.35-7.30 (m, 1H), 7.29-7.27 (m, 2H) , 6.12 (t, J = 12.817 Hz, 1H), 4.41-4.34 (m, 2H), 4.12 (q, J = 7.019 Hz, 2H), 3.62 (d, J = 6.409 Hz, 1H), 3.15(d, J = 17.700 Hz, 1H), 1.72 (d, J = 17.700 Hz, 1H), 1.35 (t, J = 7.019 Hz, 3H), 1.22 (t, J = 7.019 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) : δ 190.8, 170.9, 170.1, 160.6, 139.0, 137.7, 136.1, 132.2, 131.2, 128.4, 117.5, 116.3, 112.8, 62.3, 60.7, 45.8, 35.1, 30.5, 20.0, 13.99, 13.91; HRMS (ESI): calcd for $C_{27}H_{25}$ N_2O_5 [M+H]+ 457.1758, found 457.1743.



ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-nitrobenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3f

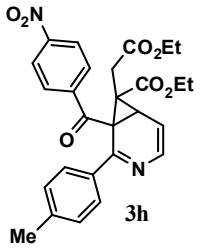
R_f: 0.5; Hexane: Ethyl acetate mixture(10:1); Yield: 65%; pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.12-8.08 (m, 2H), 7.60-7.53 (m, 3H), 7.43-7.41 (m, 1H), 7.36-7.31 (m, 2H), 7.28-7.26 (m, 2H), 6.13 (t, *J* = 12.970 Hz, 1H), 4.43-4.34 (m, 2H), 4.15-4.10 (m, 2H), 3.65 (d, *J* = 5.591 Hz, 1H), 3.15 (d, *J* = 17.700 Hz, 1H), 1.73 (d, *J* = 17.700Hz, 1H), 1.35 (t, *J* = 7.172 Hz, 3H), 1.23 (t, *J* = 7.172 Hz, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.7, 171.0, 170.2, 160.6, 150.1, 140.5, 137.8, 136.3, 131.3, 129.1, 128.56, 128.51, 123.7, 112.9, 62.4, 60.8, 45.9, 35.2, 30.6, 20.2, 14.08, 14.00; HRMS (ESI): calcd for $C_{26}H_{25}N_2O_7$ [M+H]+ 477.1656, found 477.1640.



ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-*p*-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3g

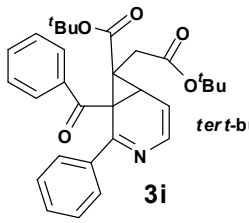
R_f: 0.4; Hexane: Ethyl acetate mixture(10:1); Yield: 93%; colourless oil ; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.554 Hz, 2H), 7.47-7.41 (m, 2H), 7.33 (d, *J* = 7.554 Hz, 1H), 7.02 (d, *J* = 8.309 Hz, 2H), 6.89 (t, *J* = 17.374, 8.309 Hz, 2H), 6.03 (t, *J* = 12.842 Hz, 1H), 4.38-4.28 (m, 2H), 4.07 (q, *J* = 6.798 Hz, 2H), 3.53 (d, *J* = 6.043 Hz, 1H), 3.11 (d, *J* = 17.374 Hz, 1H), 2.23 (s, 3H), 1.67 (d, *J* = 17.374 Hz, 1H), 1.29 (t, *J* = 6.798 Hz, 3H), 1.17(t, *J* = 6.798, Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 190.0, 171.0, 170.1, 167.0, 163.6, 160.9, 141.2, 135.8, 135.1, 132.3, 130.8, 130.7, 128.7, 128.1, 115.4, 115.2, 112.3, 61.8, 60.3, 45.6, 34.4, 30.3, 21.0, 19.6, 13.7, 13.6; HRMS (ESI): calcd for $C_{27}H_{27}FNO_5$ [M+H]+ 464.1867, found 464.1850.



ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-nitrobenzoyl)-2-*p*-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3h

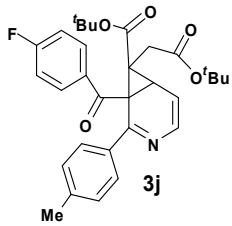
R_f: 0.5; Hexane: Ethyl acetate mixture(10:1); Yield: 57%; yellow oil; ^{1}H NMR (300 MHz, CDCl3): δ 8.14-8.08 (m, 2H), 7.58-7.54 (m, 2H), 7.51-7.46 (m, 2H),7.41-7.38 (m, 1H), 7.10-7.03 (m, 2H), 6.10 (t, *J* = 12.841 Hz 1H), 4.45-4.32 (m, 2H), 4.12 (q, *J* = 6.987 Hz, 2H), 3.61 (d, *J* = 6.043 Hz, 1H), 3.12 (d, *J* = 17.751 Hz, 1H), 2.28 (s, 3H), 1.71 (d, *J* = 17.751 Hz, 1H), 1.35 (t, *J* = 6.987 Hz, 3H), 1.22 (t, *J* = 6.987 Hz, 3H); ^{13}C NMR (125 MHz, CDCl3) : δ 190.7, 171.0, 170.2, 160.4, 150.0, 142.0, 140.4, 136.2, 135.1, 129.2, 129.1, 128.4, 123.6, 112.5, 62.3, 60.7, 45.7, 35.0, 30.6, 21.3, 20.3, 14.0, 13.9 ; HRMS (ESI): calcd for $C_{27}H_{27}N_2O_7$ [M+H]+ 491.1812, found 491.1803.



***tert*-butyl 1-benzoyl-7-(2-*tert*-butoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate**

3i

R_f: 0.6; Hexane: Ethyl acetate mixture(10:1); Yield: 56%; pale yellow solid; Melting Point: 133-135 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.66-7.61 (m, 2H), 7.49-7.44 (m, 2H), 7.43-7.34 (m, 2H), 7.31-7.22 (m, 5H), 6.09 (t, *J* = 12.842 Hz, 1H), 3.50 (d, *J* = 6.043 Hz, 1H), 3.05 (d, *J* = 17.374 Hz, 1H), 1.59 (s, 10H), 1.42 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) :191.6, 170.0, 169.9, 161.7, 138.1, 136.2, 135.6, 133.1, 130.7, 128.4, 128.3, 128.1, 113.6, 82.2, 80.7, 46.0, 34.7, 31.7, 27.9, 27.8, 20.1; HRMS (ESI): calcd for $C_{30}H_{34}NO_5$ [M+H]+ 488.2407, found 488.2410.

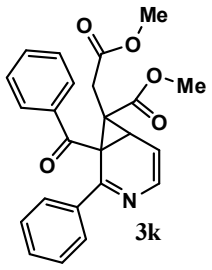


tert-butyl 7-(2-*tert*-butoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-*p*-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3j

R_f: 0.6; Hexane: Ethyl acetate mixture(10:1); Yield: 52%; pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56-7.46 (m, 4H), 7.34 (d, *J* = 6.987 Hz, 1H), 7.07 (d, *J* = 7.931 Hz, 2H), 6.97-6.90 (m, 2H), 6.06 (t, *J* = 12.841 Hz, 1H), 3.45 (d, *J* = 6.043 Hz, 1H), 3.01 (d, *J* = 17.751 Hz, 1H), 2.28 (s, 3H), 1.58 (s, 10H), 1.42

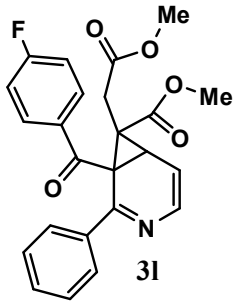
(s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) : 190.1, 170.0, 169.9, 167.2, 163.8, 161.4, 141.4, 135.7, 135.5, 132.8, 131.1, 131.0, 129.0, 128.5, 115.7, 115.4, 113.2, 82.2, 80.7, 45.8, 34.6, 31.8, 28.0, 27.9, 21.4, 20.3; HRMS (ESI): calcd for $C_{31}H_{35}FNO_5$ [M+H]+ 520.2493, found 520.2483.



methyl 1-benzoyl-7-(2-methoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3k

R_f: 0.5; Hexane: Ethyl acetate mixture(10:1); Yield: 49%; pale yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.62-7.59 (m, 2H), 7.43-7.37 (m, 4H), 7.32-7.28 (m, 1H), 7.25-7.22 (m, 4H), 6.09 (t, *J* = 12.970 Hz, 1H), 3.89 (s, 3H), 3.66 (s, 3H), 3.62 (d, *J* = 5.951 Hz, 1H), 3.18 (d, *J* = 17.853 Hz, 1H), 1.74 (d, *J* = 17.853 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) : 192.1, 172.0, 171.0, 161.5, 138.1, 136.1, 133.3, 130.9, 128.49, 128.42, 128.3, 112.9, 53.2, 51.9, 46.1, 34.7, 30.4, 19.7; HRMS (ESI): calcd for $C_{24}H_{22}NO_5$ [M+H]+ 404.1492, found 404.1489.

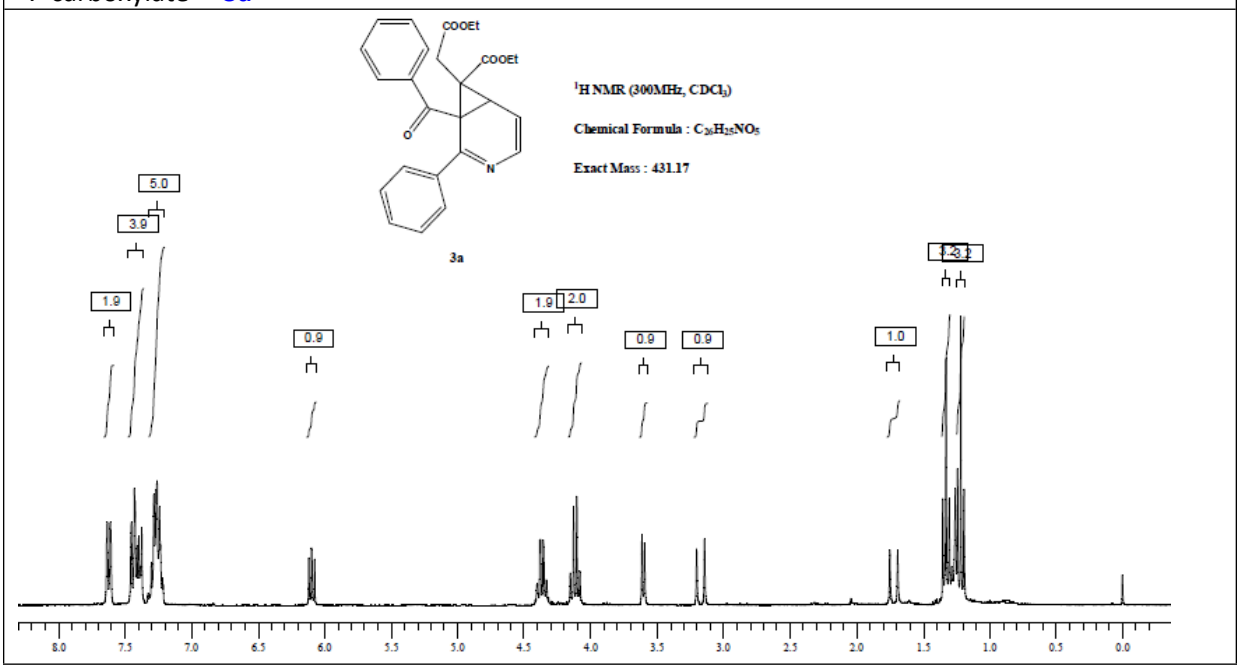


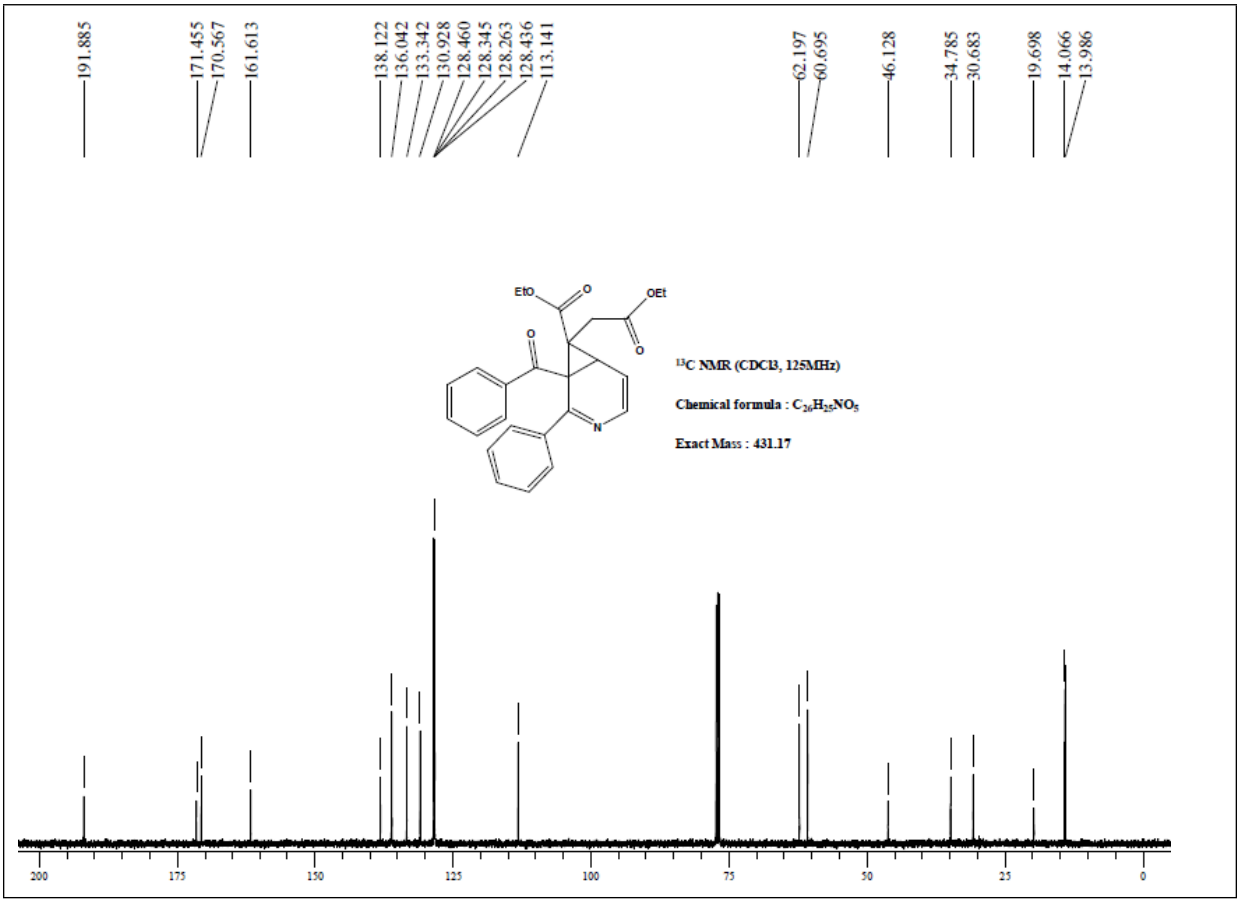
methyl 1-(4-fluorobenzoyl)-7-(2-methoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate

3l

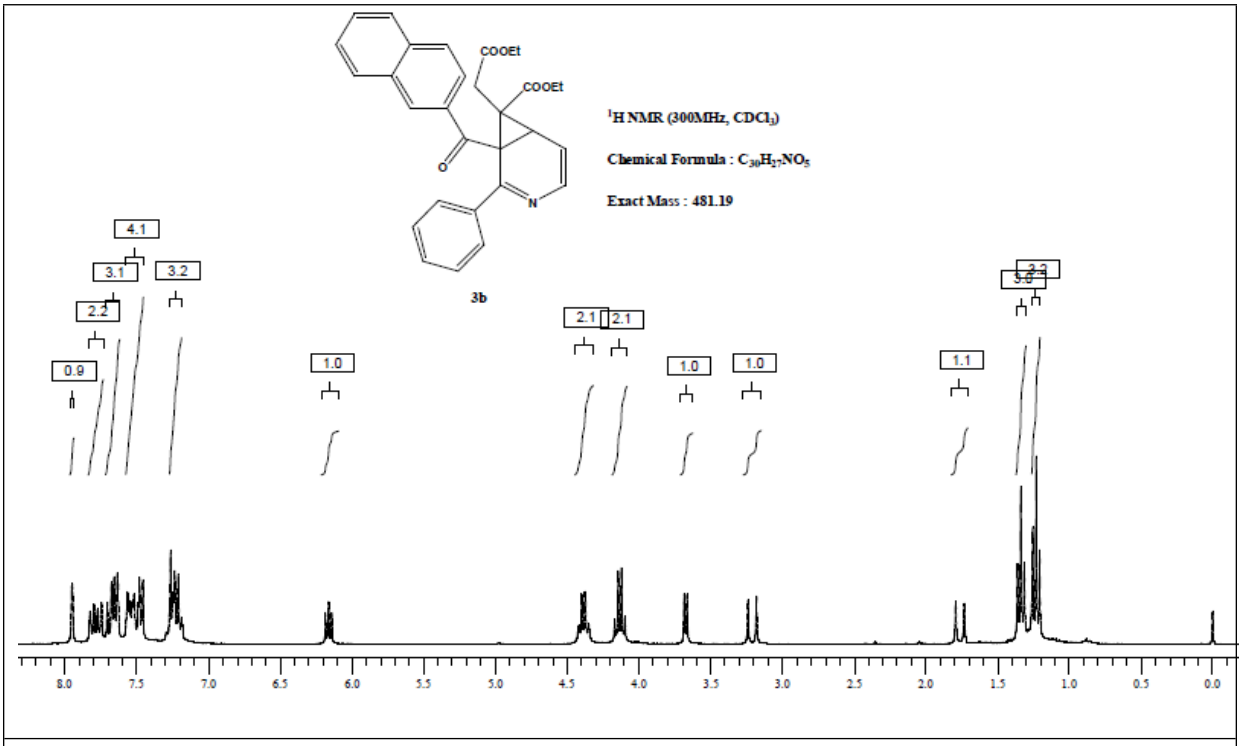
R_f: 0.5; Hexane: Ethyl acetate mixture(10:1); Yield: 55%; pale yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.63-7.59 (m, 2H), 7.47-7.41 (m, 2H), 7.39 (d, *J* = 7.019 Hz, 1H), 7.35-7.30 (m, 1H), 7.28-7.24 (m, 2H), 6.93 (t, *J* = 16.785, 8.392 Hz, 2H), 6.09 (t, *J* = 12.817 Hz, 1H), 3.89 (s, 3H), 3.66 (s, 3H), 3.61 (d, *J* = 5.951 Hz, 1H), 3.17 (d, *J* = 17.853 Hz, 1H), 1.73 (d, *J* = 17.853 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 190.5, 171.8, 170.9, 167.3, 163.9, 161.3, 137.9, 136.1, 131.1, 131.0, 130.9, 128.3, 115.8, 115.6, 112.9, 53.3, 51.9, 45.9, 34.7, 30.3, 19.7; HRMS (ESI): calcd for $C_{24}H_{21}FNO_5$ [M+H]+ 422.1398, found 422.1387.

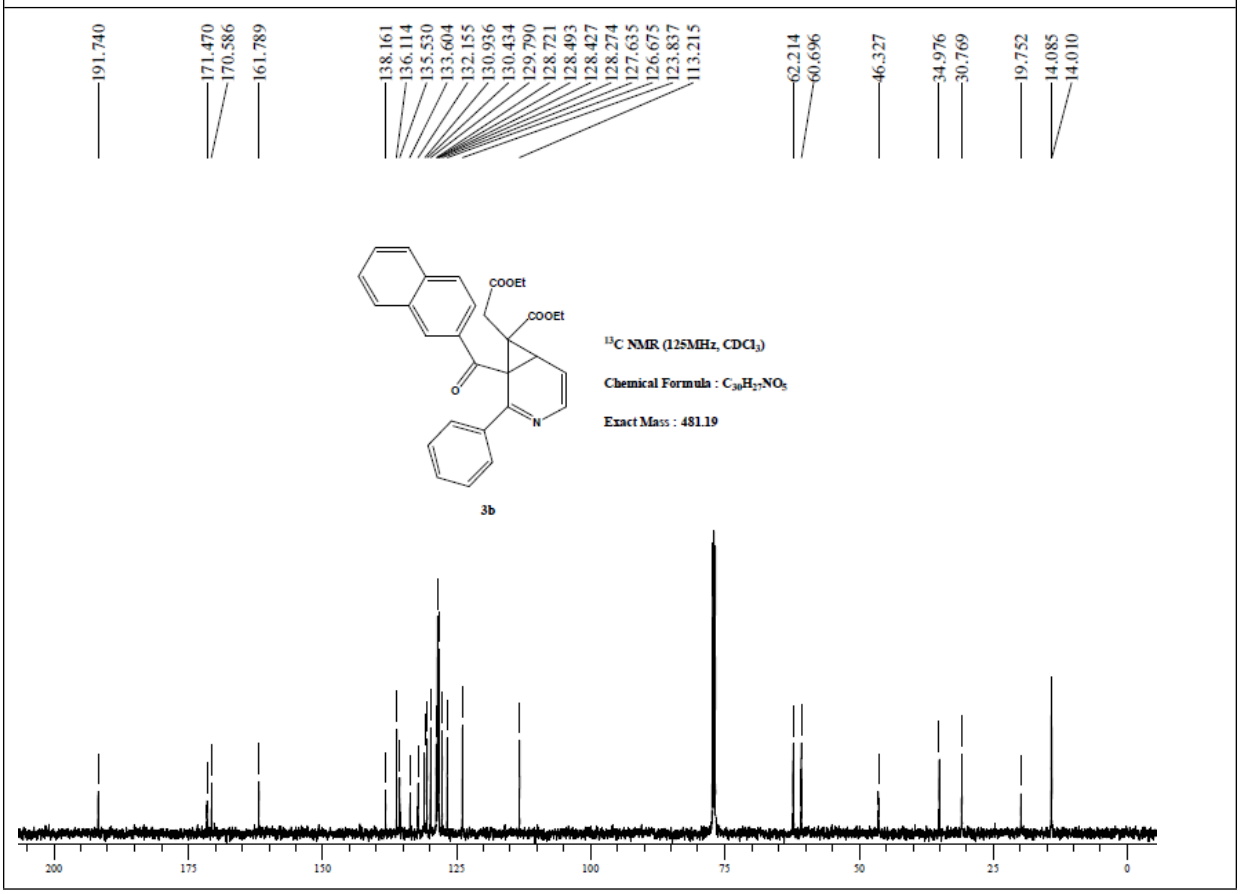
Ethyl 1-benzoyl-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene -7-carboxylate **3a**





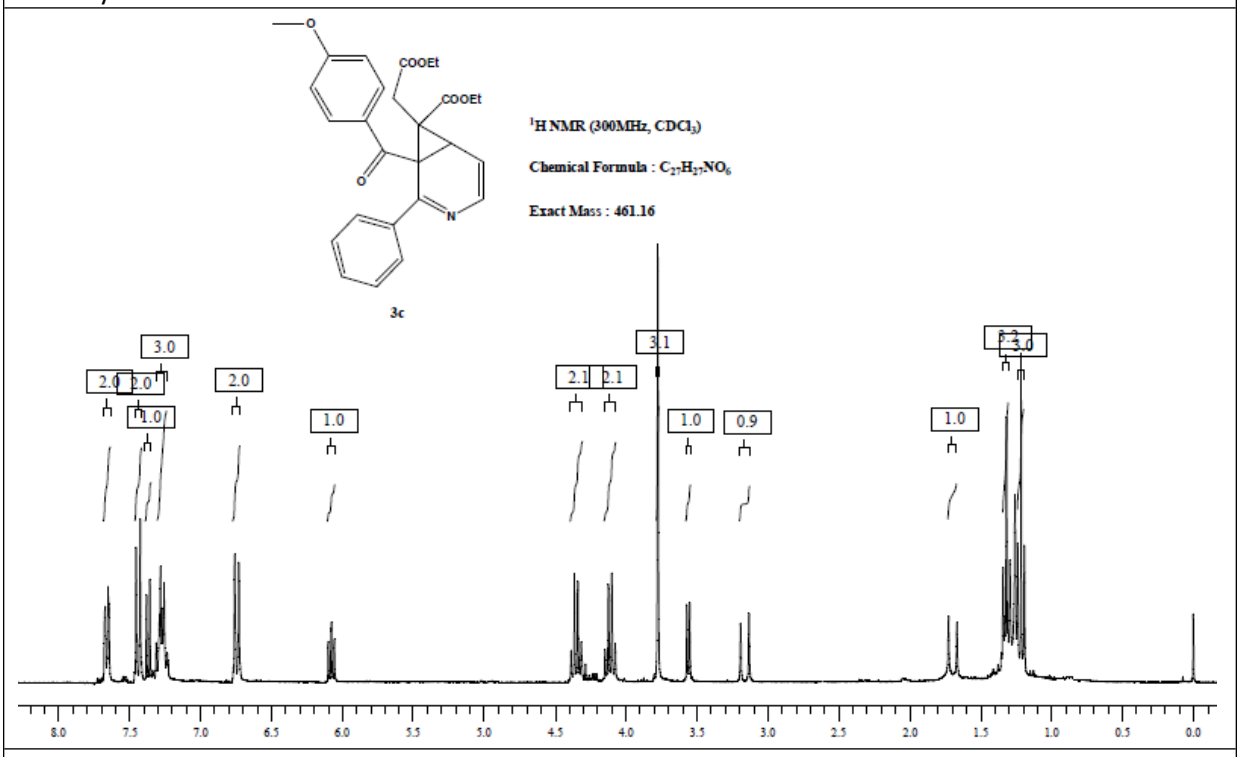
Ethyl 1-(2-naphthoyl)-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3b**

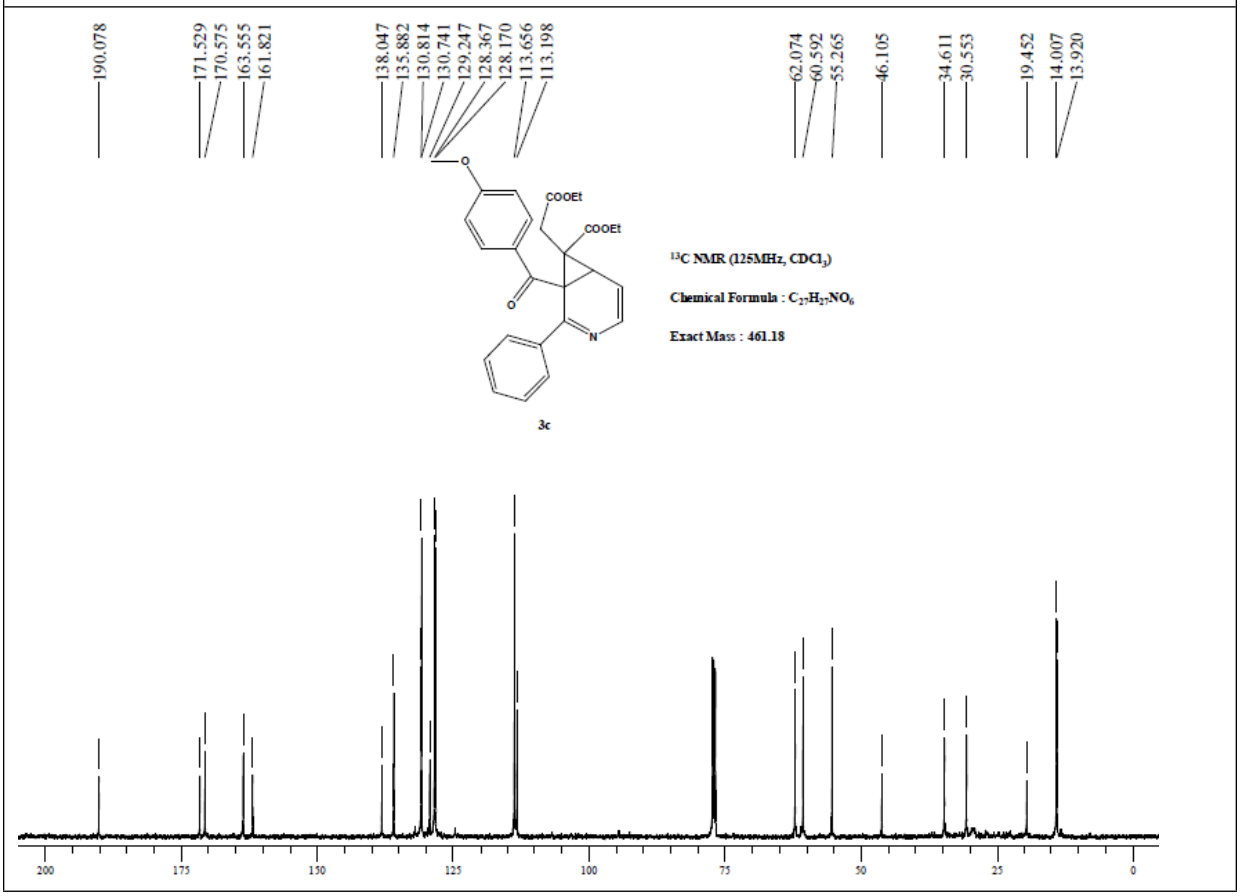




Ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-methoxybenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-

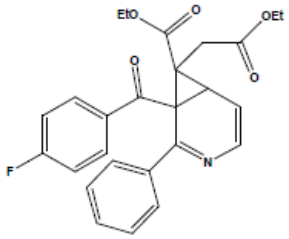
carboxylate **3c**





Ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-

carboxylate **3d**

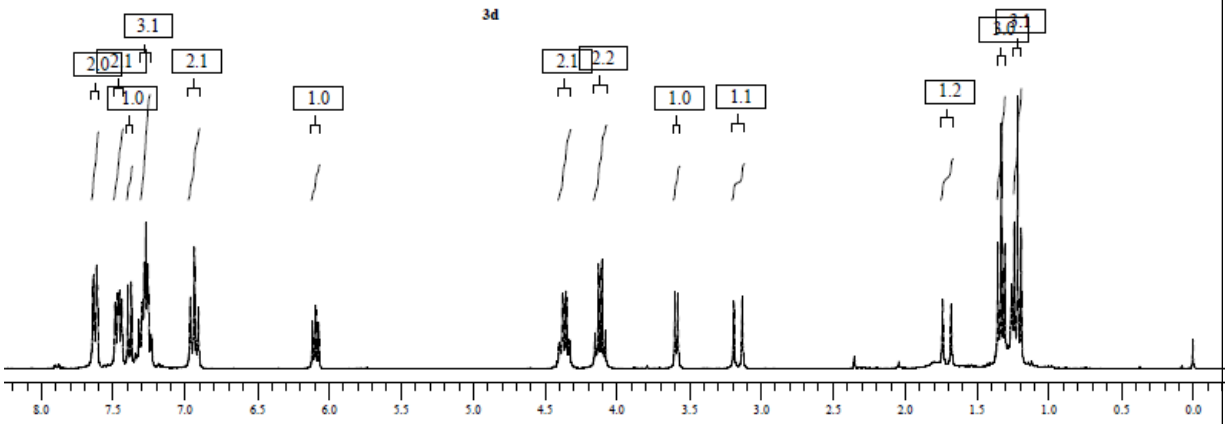


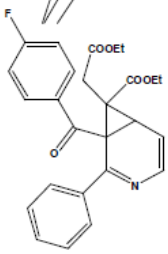
^{1}H NMR (CDCl3, 300MHz)

Chemical formula : $C_{26}H_{24}FNO_5$

Exact Mass : 449.16

3d



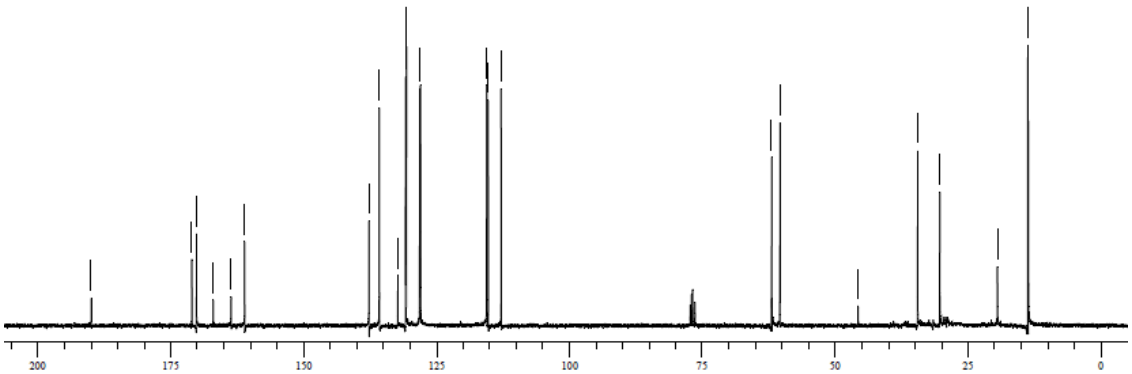


^{13}C NMR (75MHz, $CDCl_3$)

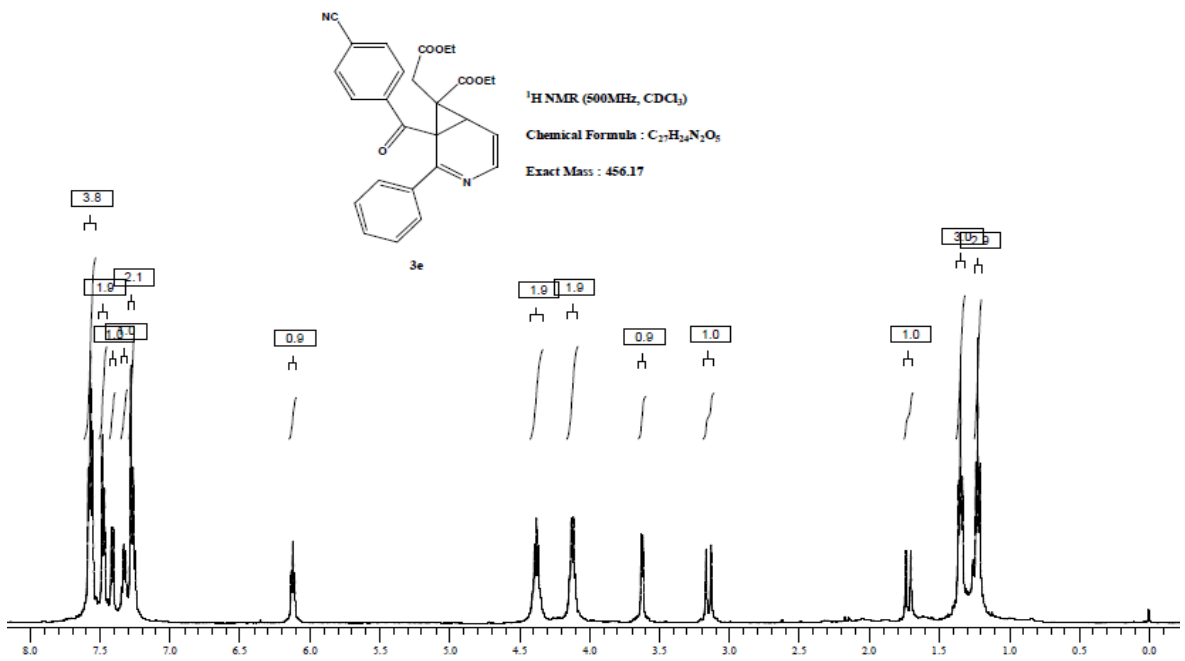
Chemical Formula : $C_{26}H_{24}FNO_5$

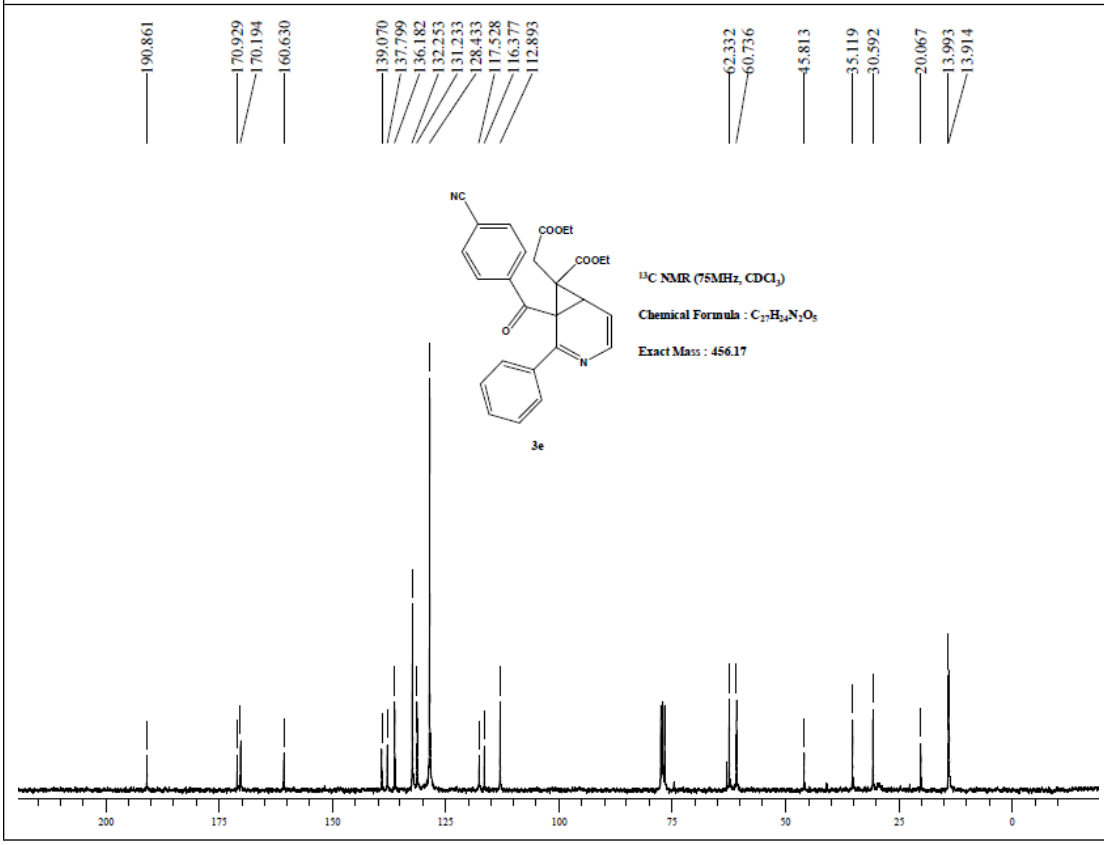
Exact Mass : 449.16

3d

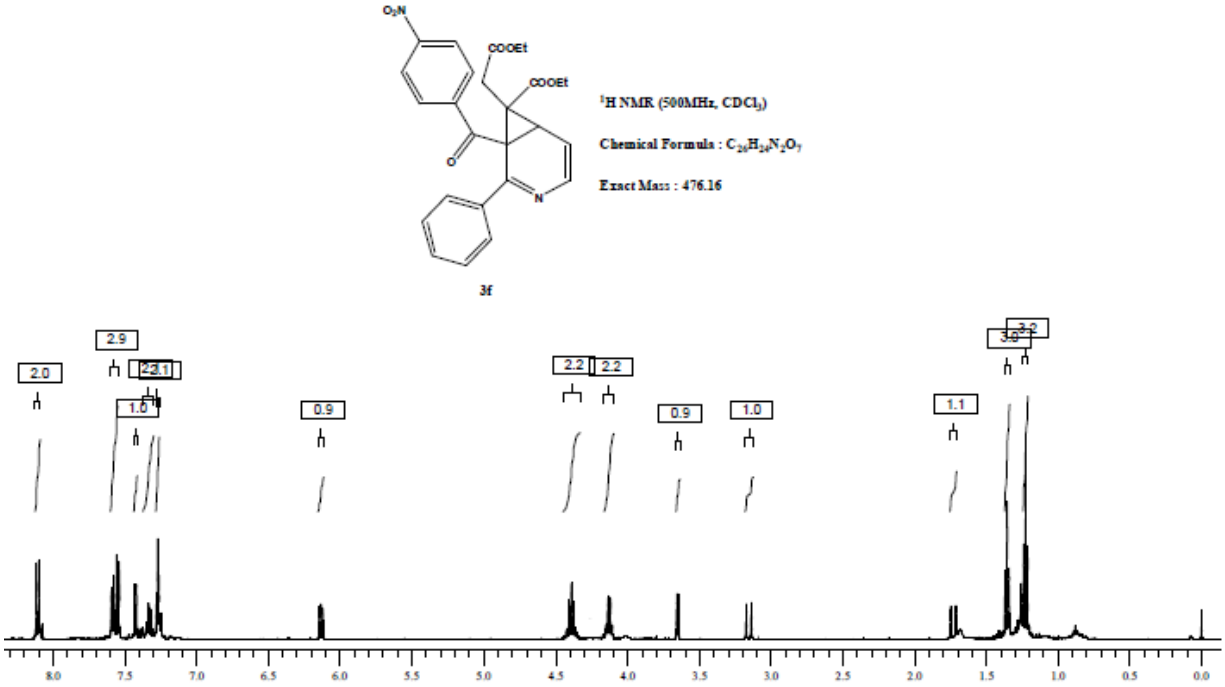


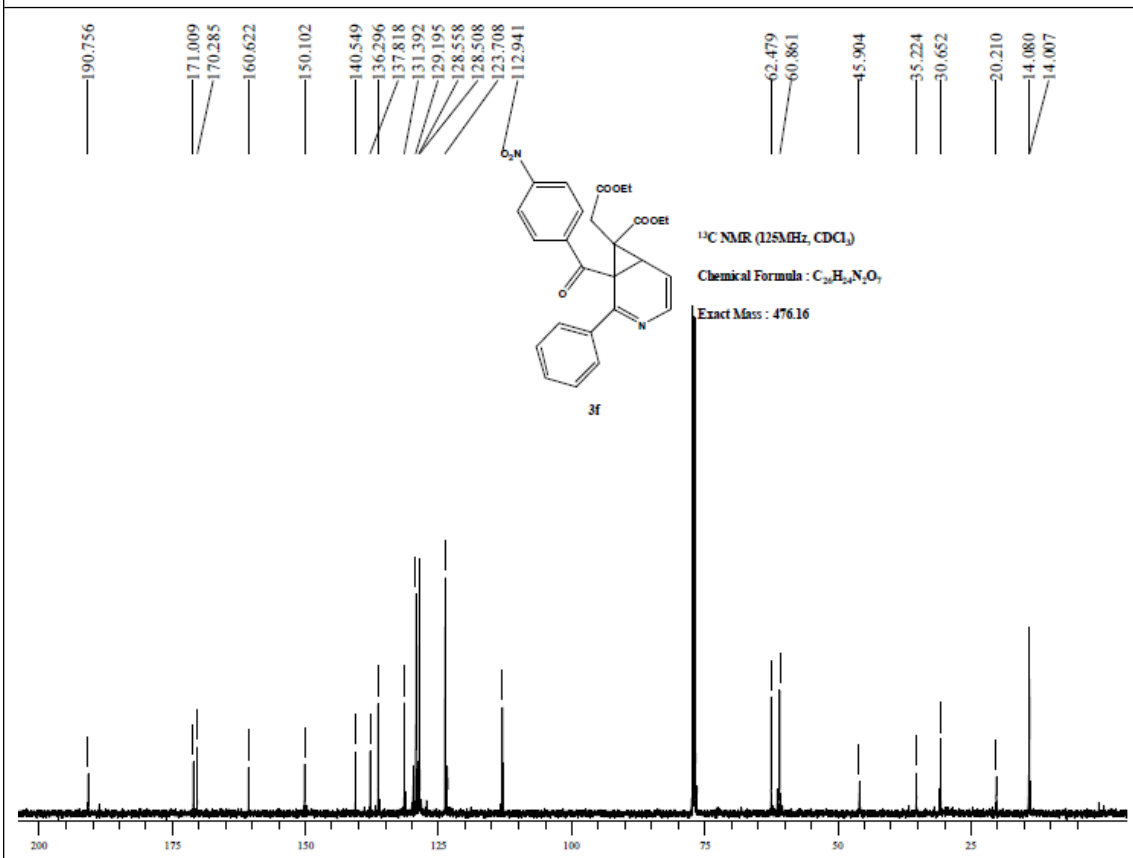
Ethyl 1-(4-cyanobenzoyl)-7-(2-ethoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3e**



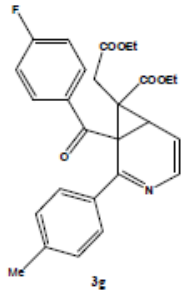


Ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-nitrobenzoyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3f**





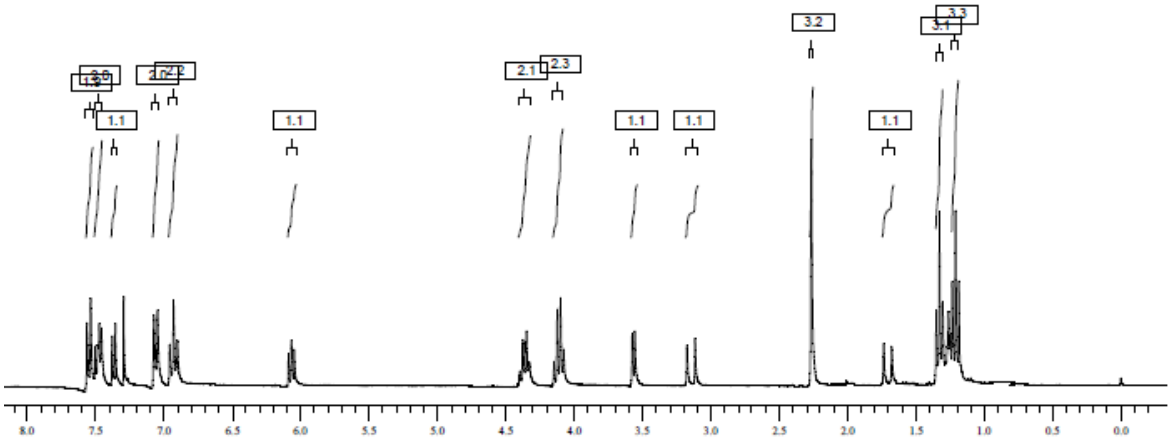
Ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-p-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3g**

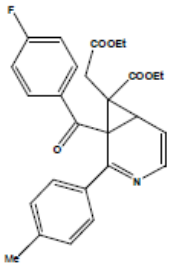


^{1}H NMR (300MHz, $CDCl_3$)

Chemical Formula : $C_{27}H_{20}FNO_5$

Exact Mass : 463.18

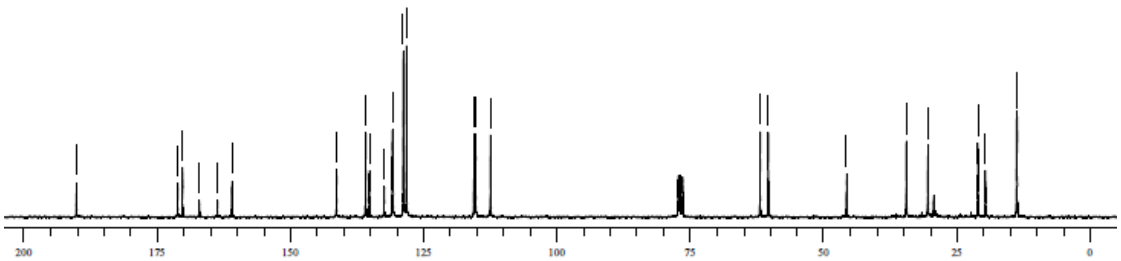




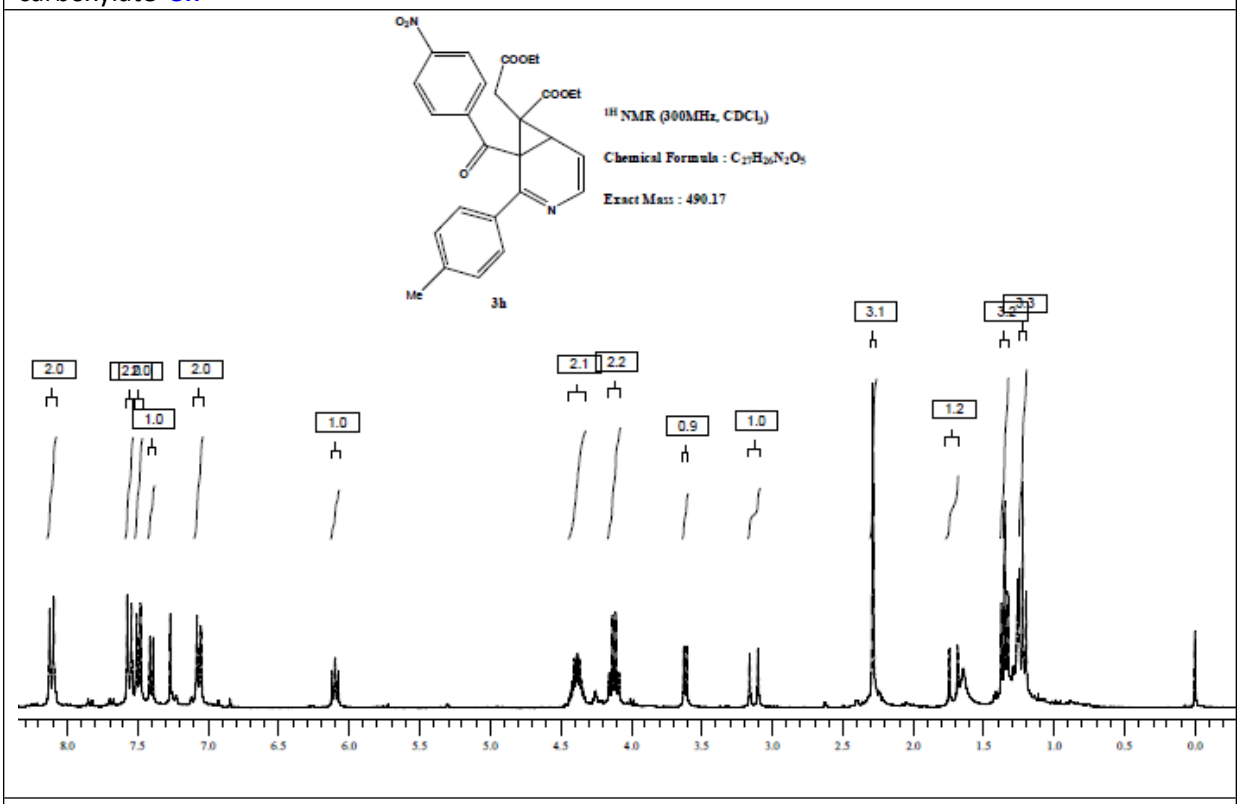
^{13}C NMR (75MHz, $CDCl_3$)

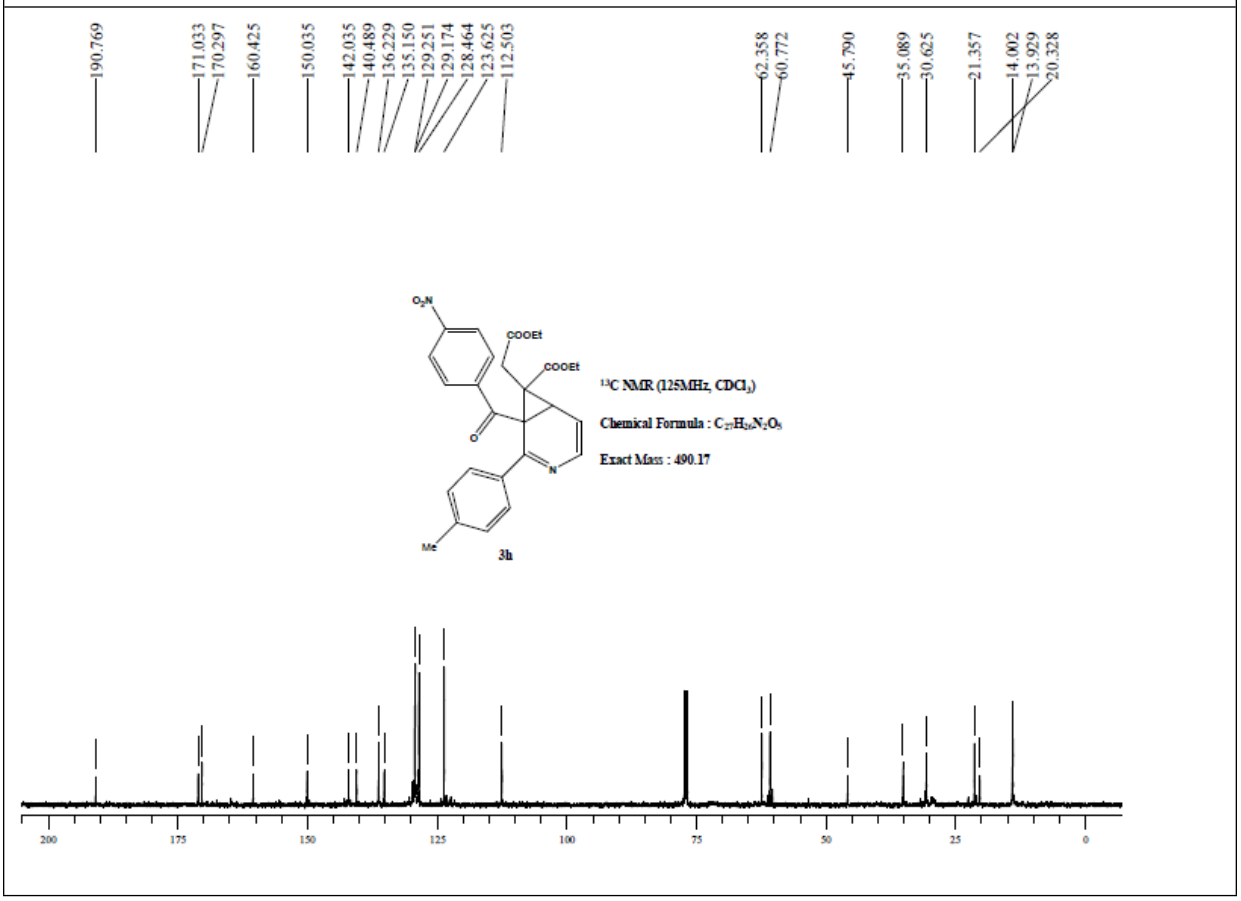
Chemical Formula : $C_{27}H_{20}FNO_5$

Exact Mass : 463.18

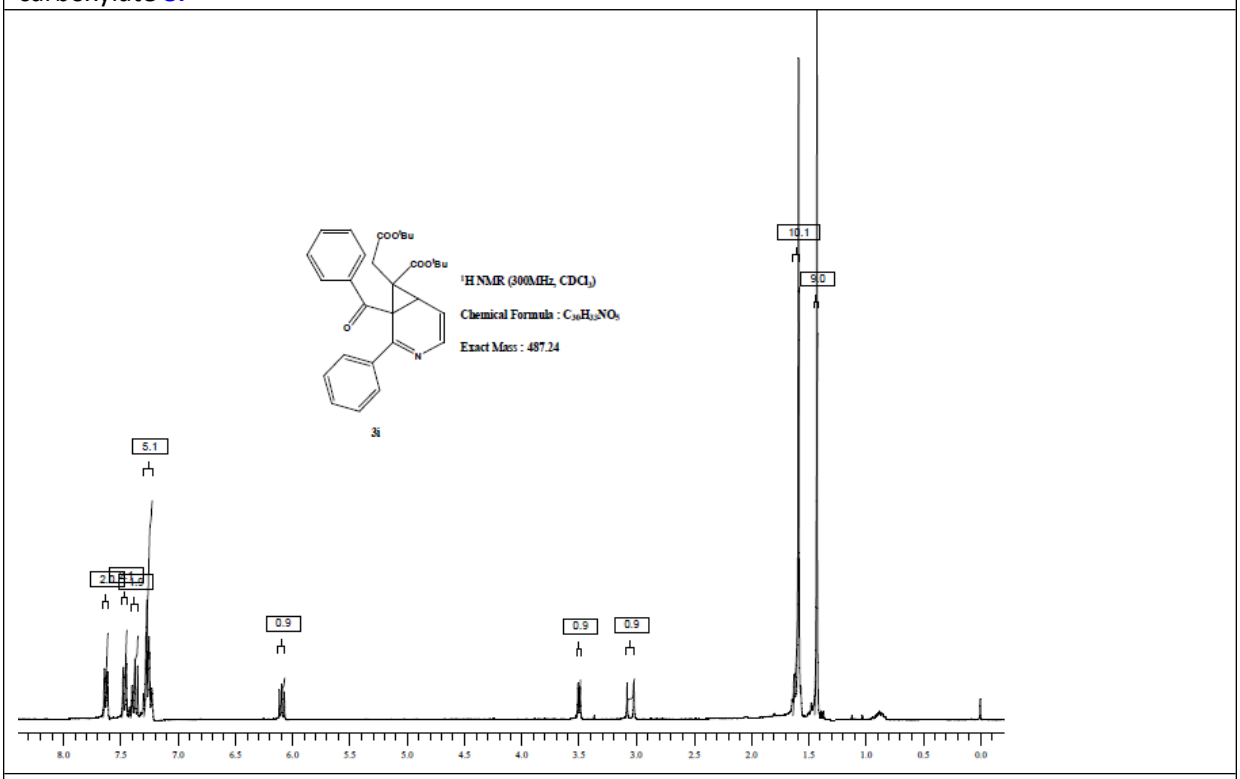


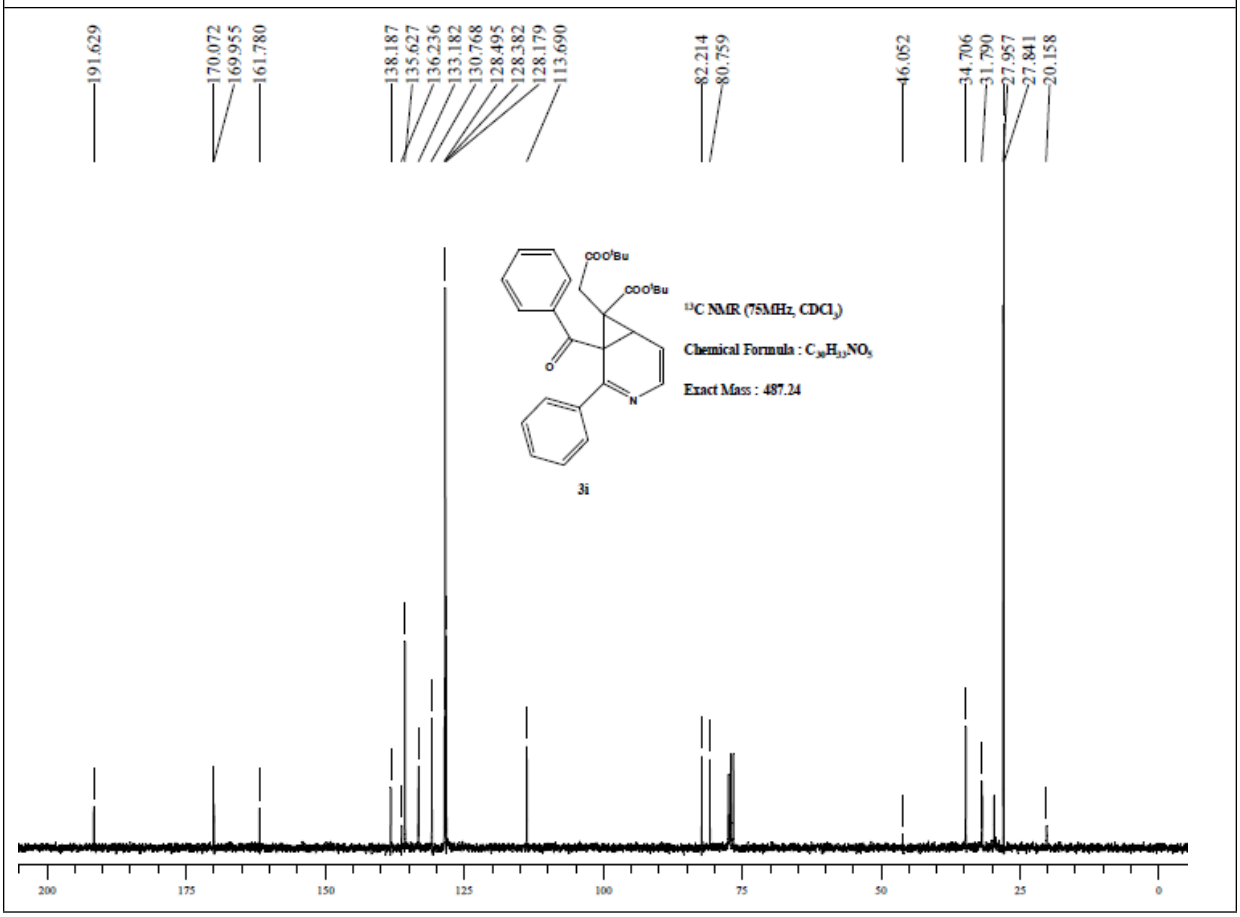
Ethyl 7-(2-ethoxy-2-oxoethyl)-1-(4-nitrobenzoyl)-2-p-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3h**



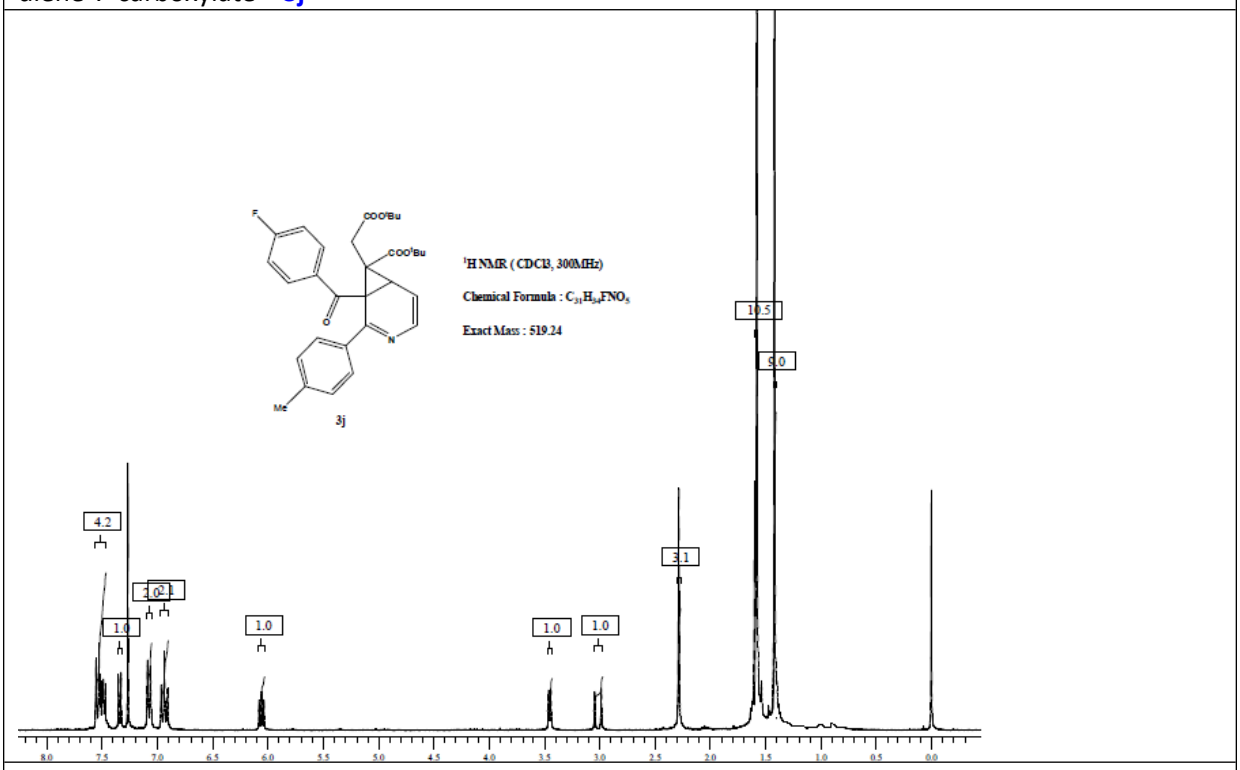


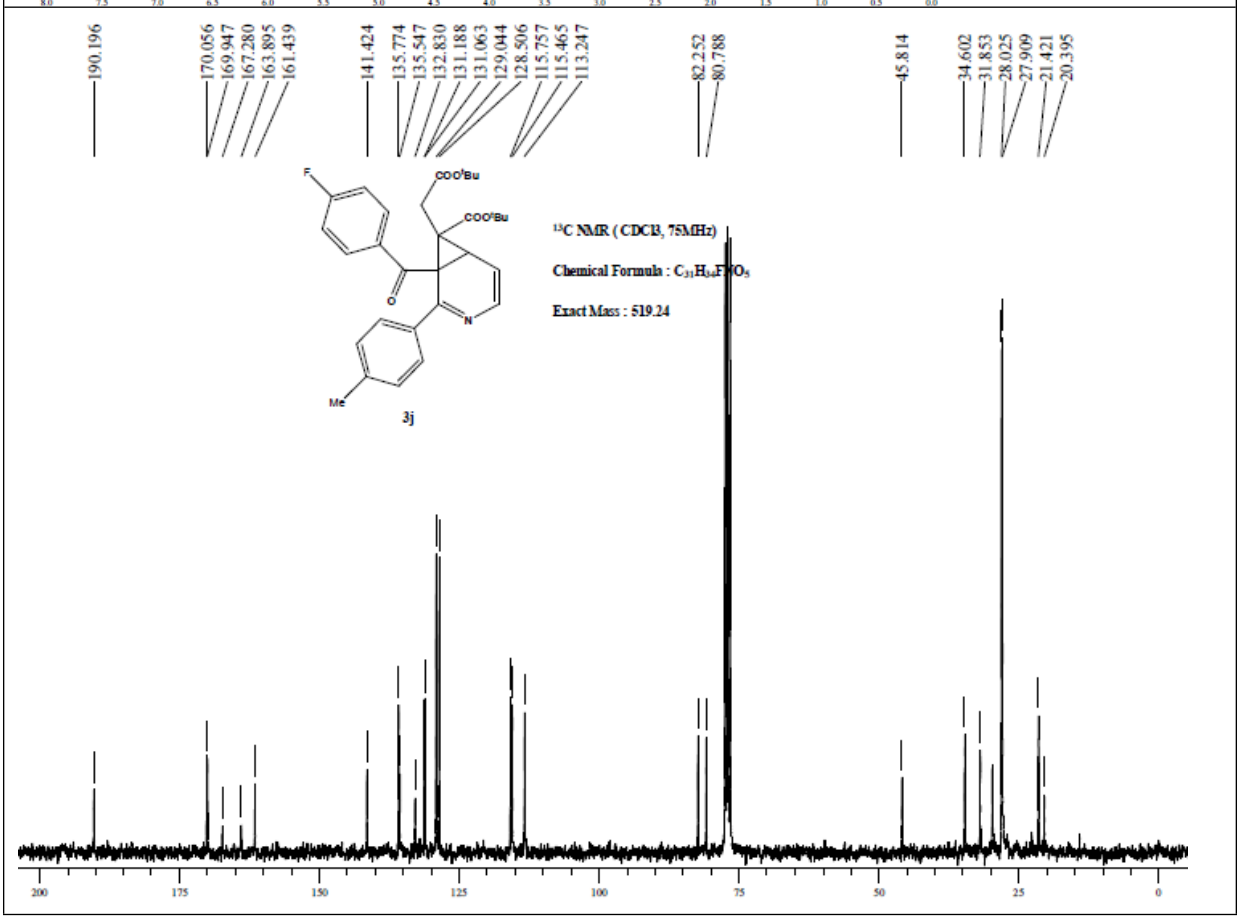
Tert-butyl 1-benzoyl-7-(2-tert-butoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3i**



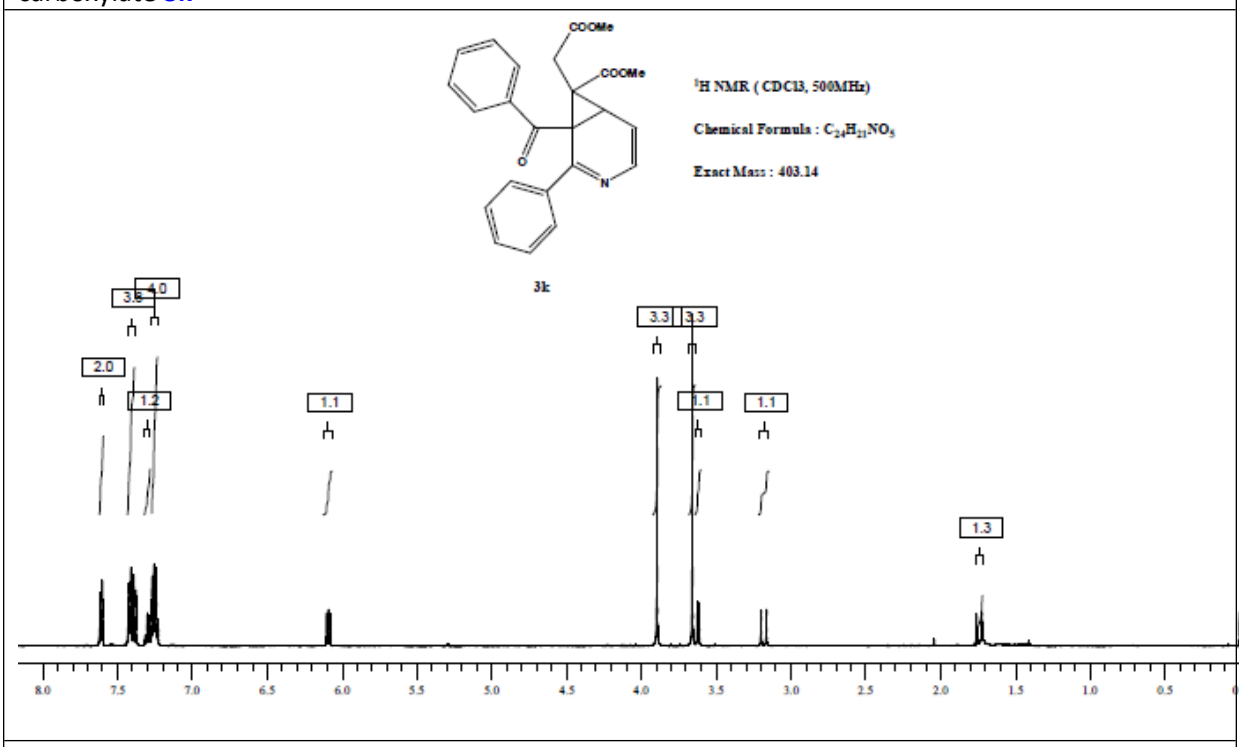


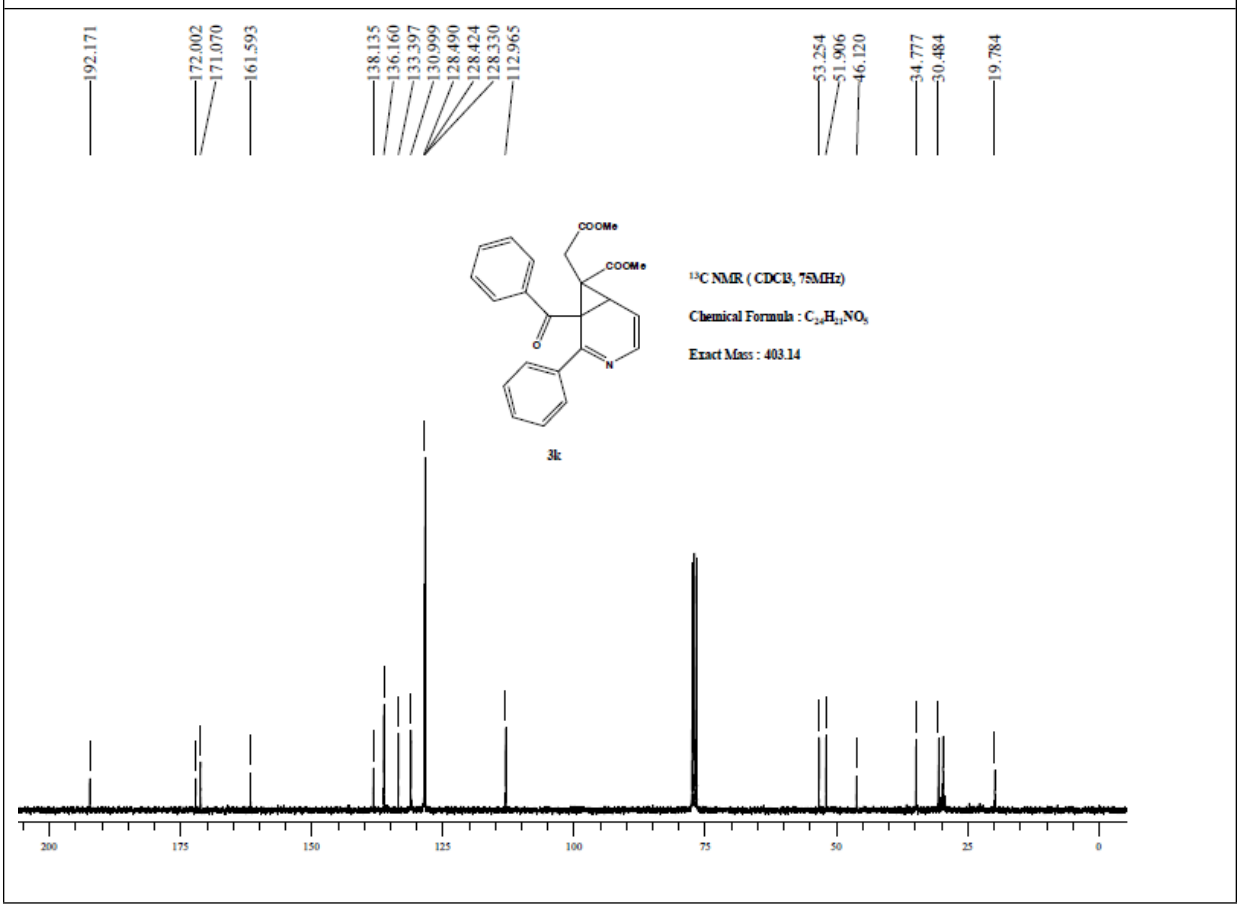
Tert-butyl 7-(2-tert-butoxy-2-oxoethyl)-1-(4-fluorobenzoyl)-2-p-tolyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3j**



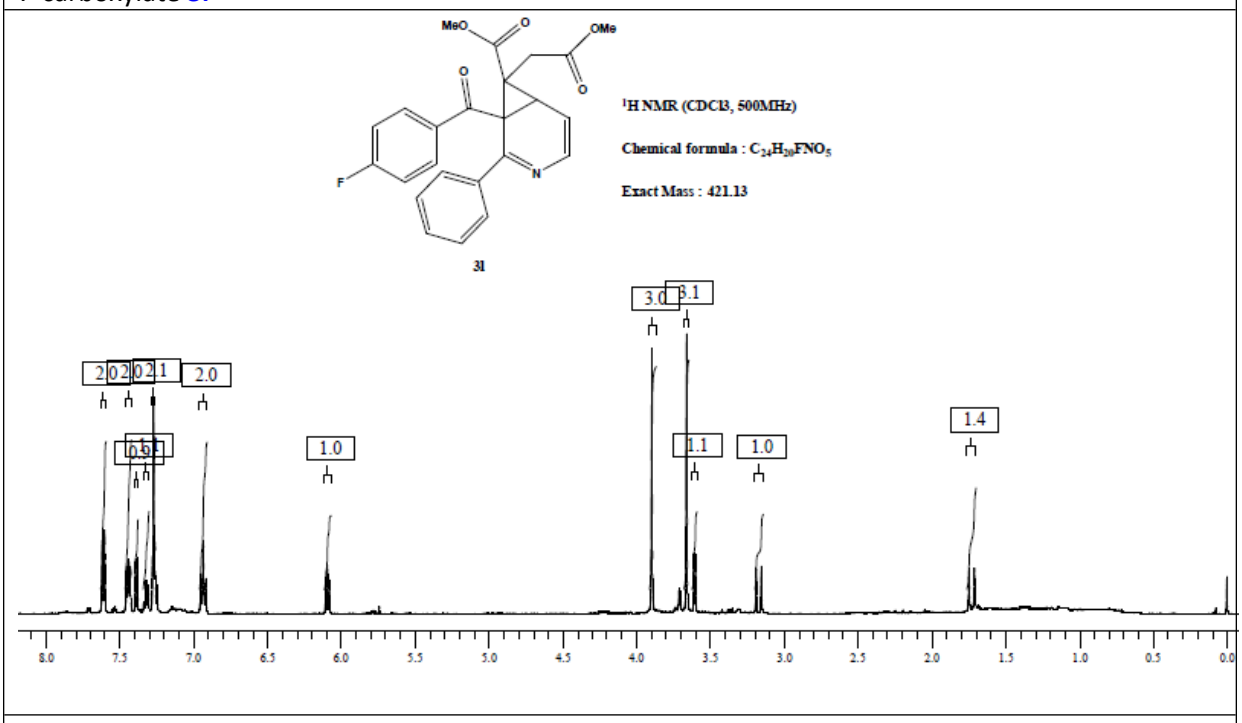


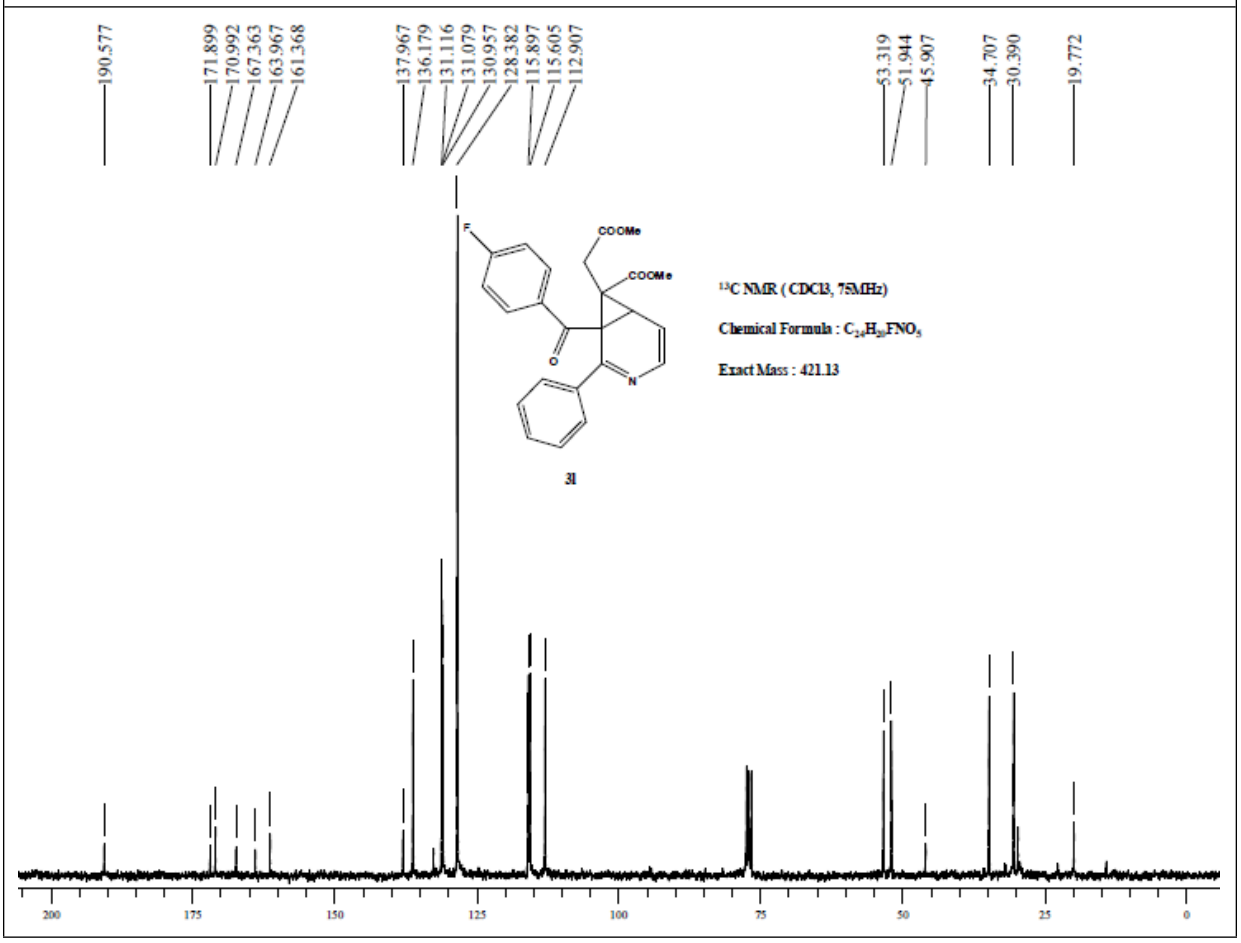
Methyl 1-benzoyl-7-(2-methoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3k**





Methyl 1-(4-fluorobenzoyl)-7-(2-methoxy-2-oxoethyl)-2-phenyl-3-azabicyclo[4.1.0]hepta-2,4-diene-7-carboxylate **3l**





1.4 X-ray crystallography data

Cambridge Crystallographic Data Centre (CCDC)

This CIF contains data from an original supplementary publication deposited with the CCDC, and may include chemical, crystal, experimental, refinement, atomic coordinates, anisotropic displacement parameters and molecular geometry data, as required by the journal to which it was submitted. This CIF is provided on the understanding that it is used for bona fide research purposes only. It may contain copyright material of the CCDC or of third parties, and may not be copied or further disseminated in any form, whether machine-readable or not, except for the purpose of generating routine backup copies on your local computer system. For further information on the CCDC, data deposition and data retrieval see: www.ccdc.cam.ac.uk

Crystal structure data for compound 3a: Crystal structure deposition no: CCDC 932647.

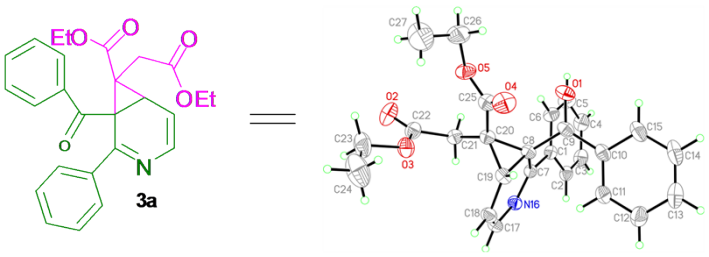


Figure 2. ORTEP diagram of the 3-azabicyclo[4.1.0]hepta-2,4-diene **3a**

Table 1. Crystal data and structure refinement for **3a**.

Identification code	eiict277	
Empirical formula	$C_{26}H_{25}NO_5$	
Formula weight	431.47	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 16.104(6) Å	α= 90°.
	b = 6.924(3) Å	β= 117.522(19)°.
	c = 22.877(7) Å	γ = 90°.
Volume	2262.2(15) $Å^3$	
Z	4	
Density (calculated)	1.267 Mg/m^3	
Absorption coefficient	0.088 mm^{-1}	
F(000)	912	
Crystal size	0.38 x 0.32 x 0.29 mm^3	
Theta range for data collection	1.43 to 25.00°.	
Index ranges	-19<=h<=17, -8<=k<=8, -27<=l<=27	
Reflections collected	15474	
Independent reflections	3984 [R(int) = 0.0298]	
Completeness to theta = 25.00°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9750 and 0.9674	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3984 / 0 / 291	
Goodness-of-fit on F^2	1.068	
Final R indices [I>2sigma(I)]	R1 = 0.0552, wR2 = 0.1616	
R indices (all data)	R1 = 0.0668, wR2 = 0.1718	
Largest diff. peak and hole	0.787 and -0.373 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for eiict277. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3819(1)	5868(3)	6284(1)	46(1)
C(2)	4425(2)	6489(3)	6040(1)	55(1)
C(3)	4734(2)	5221(4)	5724(1)	68(1)
C(4)	4454(2)	3325(4)	5639(1)	73(1)
C(5)	3869(2)	2679(4)	5881(1)	72(1)
C(6)	3544(2)	3948(3)	6201(1)	58(1)
C(7)	3558(1)	7236(3)	6669(1)	44(1)
C(8)	2620(1)	7031(3)	6658(1)	45(1)
C(9)	1805(1)	6409(3)	6007(1)	53(1)
C(10)	1436(1)	7814(3)	5456(1)	55(1)
C(11)	1782(2)	9671(4)	5518(1)	65(1)
C(12)	1384(2)	10970(4)	5001(1)	80(1)
C(13)	617(2)	10413(6)	4421(1)	89(1)
C(14)	273(2)	8575(6)	4355(1)	94(1)
C(15)	681(2)	7264(5)	4865(1)	77(1)
C(17)	3964(2)	9938(3)	7349(1)	56(1)
C(18)	3175(2)	9995(3)	7399(1)	56(1)
C(19)	2454(1)	8524(3)	7087(1)	51(1)
C(20)	2610(1)	6426(3)	7296(1)	49(1)
C(21)	3512(1)	5732(3)	7871(1)	53(1)
C(22)	3493(2)	5601(4)	8520(1)	62(1)
C(23)	2947(3)	7060(6)	9212(2)	102(1)
C(24)	2421(3)	8779(8)	9211(2)	148(2)
C(25)	1716(2)	5488(4)	7212(1)	59(1)
C(26)	1102(2)	2795(5)	7534(2)	103(1)
C(27)	1017(4)	3269(10)	8084(2)	184(3)
N(16)	4169(1)	8539(3)	6998(1)	51(1)
O(1)	1473(1)	4801(3)	5953(1)	76(1)
O(2)	3913(2)	4433(3)	8938(1)	97(1)
O(3)	2975(1)	6993(3)	8586(1)	73(1)
O(4)	954(1)	6205(3)	6929(1)	85(1)
O(5)	1887(1)	3790(3)	7515(1)	72(1)

Table 3. Bond lengths [Å] and angles [°] for eiict277.

C(1)-C(6)	1.386(3)
C(1)-C(2)	1.398(3)
C(1)-C(7)	1.479(3)
C(2)-C(3)	1.369(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.373(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.370(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.391(3)
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(7)-N(16)	1.290(3)
C(7)-C(8)	1.505(3)
C(8)-C(9)	1.523(3)
C(8)-C(20)	1.527(3)
C(8)-C(19)	1.533(3)
C(9)-O(1)	1.217(3)
C(9)-C(10)	1.482(3)
C(10)-C(11)	1.382(3)
C(10)-C(15)	1.391(3)
C(11)-C(12)	1.385(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.385(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.368(5)
C(13)-H(13)	0.9300
C(14)-C(15)	1.381(4)
C(14)-H(14)	0.9300
C(15)-H(15)	0.9300
C(17)-C(18)	1.328(3)
C(17)-N(16)	1.391(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.460(3)
C(18)-H(18)	0.9300
C(19)-C(20)	1.514(3)

C(19)-H(19)	0.9800
C(20)-C(25)	1.509(3)
C(20)-C(21)	1.518(3)
C(21)-C(22)	1.501(3)
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(22)-O(2)	1.195(3)
C(22)-O(3)	1.328(3)
C(23)-O(3)	1.455(3)
C(23)-C(24)	1.460(6)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-H(24A)	0.9600
C(24)-H(24B)	0.9600
C(24)-H(24C)	0.9600
C(25)-O(4)	1.199(3)
C(25)-O(5)	1.328(3)
C(26)-C(27)	1.368(5)
C(26)-O(5)	1.458(3)
C(26)-H(26A)	0.9700
C(26)-H(26B)	0.9700
C(27)-H(27A)	0.9600
C(27)-H(27B)	0.9600
C(27)-H(27C)	0.9600
C(6)-C(1)-C(2)	118.55(19)
C(6)-C(1)-C(7)	122.32(18)
C(2)-C(1)-C(7)	118.93(19)
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(2)-C(3)-C(4)	120.7(2)
C(2)-C(3)-H(3)	119.6
C(4)-C(3)-H(3)	119.6
C(5)-C(4)-C(3)	119.9(2)
C(5)-C(4)-H(4)	120.1
C(3)-C(4)-H(4)	120.1
C(4)-C(5)-C(6)	120.2(2)
C(4)-C(5)-H(5)	119.9

C(6)-C(5)-H(5)	119.9
C(1)-C(6)-C(5)	120.2(2)
C(1)-C(6)-H(6)	119.9
C(5)-C(6)-H(6)	119.9
N(16)-C(7)-C(1)	116.49(17)
N(16)-C(7)-C(8)	124.30(18)
C(1)-C(7)-C(8)	119.21(16)
C(7)-C(8)-C(9)	116.57(16)
C(7)-C(8)-C(20)	117.66(16)
C(9)-C(8)-C(20)	118.20(17)
C(7)-C(8)-C(19)	113.40(16)
C(9)-C(8)-C(19)	119.36(17)
C(20)-C(8)-C(19)	59.31(13)
O(1)-C(9)-C(10)	121.98(19)
O(1)-C(9)-C(8)	119.9(2)
C(10)-C(9)-C(8)	118.15(18)
C(11)-C(10)-C(15)	119.0(2)
C(11)-C(10)-C(9)	122.63(19)
C(15)-C(10)-C(9)	118.3(2)
C(10)-C(11)-C(12)	120.7(2)
C(10)-C(11)-H(11)	119.6
C(12)-C(11)-H(11)	119.6
C(11)-C(12)-C(13)	119.5(3)
C(11)-C(12)-H(12)	120.2
C(13)-C(12)-H(12)	120.2
C(14)-C(13)-C(12)	120.1(3)
C(14)-C(13)-H(13)	120.0
C(12)-C(13)-H(13)	120.0
C(13)-C(14)-C(15)	120.5(3)
C(13)-C(14)-H(14)	119.8
C(15)-C(14)-H(14)	119.8
C(14)-C(15)-C(10)	120.1(3)
C(14)-C(15)-H(15)	119.9
C(10)-C(15)-H(15)	119.9
C(18)-C(17)-N(16)	124.19(19)
C(18)-C(17)-H(17)	117.9
N(16)-C(17)-H(17)	117.9
C(17)-C(18)-C(19)	120.9(2)

C(17)-C(18)-H(18)	119.5
C(19)-C(18)-H(18)	119.5
C(18)-C(19)-C(20)	122.41(18)
C(18)-C(19)-C(8)	116.65(18)
C(20)-C(19)-C(8)	60.14(13)
C(18)-C(19)-H(19)	115.4
C(20)-C(19)-H(19)	115.4
C(8)-C(19)-H(19)	115.4
C(25)-C(20)-C(19)	111.28(18)
C(25)-C(20)-C(21)	116.67(19)
C(19)-C(20)-C(21)	122.06(18)
C(25)-C(20)-C(8)	115.28(17)
C(19)-C(20)-C(8)	60.55(13)
C(21)-C(20)-C(8)	119.14(16)
C(22)-C(21)-C(20)	115.28(18)
C(22)-C(21)-H(21A)	108.5
C(20)-C(21)-H(21A)	108.5
C(22)-C(21)-H(21B)	108.5
C(20)-C(21)-H(21B)	108.5
H(21A)-C(21)-H(21B)	107.5
O(2)-C(22)-O(3)	123.6(2)
O(2)-C(22)-C(21)	124.7(2)
O(3)-C(22)-C(21)	111.7(2)
O(3)-C(23)-C(24)	107.8(3)
O(3)-C(23)-H(23A)	110.1
C(24)-C(23)-H(23A)	110.1
O(3)-C(23)-H(23B)	110.1
C(24)-C(23)-H(23B)	110.1
H(23A)-C(23)-H(23B)	108.5
C(23)-C(24)-H(24A)	109.5
C(23)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(23)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
O(4)-C(25)-O(5)	124.2(2)
O(4)-C(25)-C(20)	124.7(2)
O(5)-C(25)-C(20)	111.02(19)

C(27)-C(26)-O(5)	111.8(3)
C(27)-C(26)-H(26A)	109.3
O(5)-C(26)-H(26A)	109.3
C(27)-C(26)-H(26B)	109.3
O(5)-C(26)-H(26B)	109.3
H(26A)-C(26)-H(26B)	107.9
C(26)-C(27)-H(27A)	109.5
C(26)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(26)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(7)-N(16)-C(17)	120.29(17)
C(22)-O(3)-C(23)	116.2(2)
C(25)-O(5)-C(26)	117.4(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for eiict277. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	38(1)	54(1)	45(1)	3(1)	19(1)	2(1)
C(2)	51(1)	69(1)	49(1)	4(1)	27(1)	-3(1)
C(3)	63(1)	92(2)	63(1)	7(1)	42(1)	5(1)
C(4)	79(2)	81(2)	74(2)	2(1)	48(1)	19(1)
C(5)	85(2)	56(1)	91(2)	-2(1)	55(2)	7(1)
C(6)	60(1)	55(1)	70(1)	3(1)	41(1)	2(1)
C(7)	38(1)	50(1)	42(1)	3(1)	18(1)	-2(1)
C(8)	38(1)	48(1)	51(1)	-6(1)	21(1)	-2(1)
C(9)	40(1)	61(1)	61(1)	-16(1)	25(1)	-9(1)
C(10)	38(1)	74(2)	52(1)	-13(1)	20(1)	-5(1)
C(11)	55(1)	74(2)	54(1)	-4(1)	15(1)	0(1)
C(12)	81(2)	84(2)	68(2)	9(1)	28(1)	13(2)
C(13)	72(2)	133(3)	56(2)	16(2)	25(1)	23(2)
C(14)	64(2)	154(3)	49(1)	-11(2)	14(1)	-8(2)
C(15)	58(1)	111(2)	56(1)	-19(1)	21(1)	-21(1)
C(17)	54(1)	53(1)	53(1)	-8(1)	17(1)	-13(1)
C(18)	61(1)	49(1)	56(1)	-7(1)	24(1)	0(1)
C(19)	42(1)	55(1)	55(1)	-7(1)	21(1)	4(1)
C(20)	44(1)	51(1)	57(1)	-6(1)	29(1)	-3(1)
C(21)	49(1)	56(1)	60(1)	1(1)	31(1)	2(1)
C(22)	59(1)	69(2)	64(1)	4(1)	34(1)	0(1)
C(23)	107(2)	147(3)	73(2)	-22(2)	59(2)	-9(2)
C(24)	134(3)	214(5)	126(3)	-54(3)	86(3)	14(3)
C(25)	51(1)	69(2)	70(1)	-9(1)	38(1)	-6(1)
C(26)	95(2)	119(3)	117(2)	-12(2)	69(2)	-45(2)
C(27)	155(4)	296(7)	147(4)	-59(4)	108(4)	-116(5)
N(16)	41(1)	58(1)	49(1)	-2(1)	18(1)	-9(1)
O(1)	65(1)	69(1)	83(1)	-17(1)	26(1)	-26(1)
O(2)	105(2)	113(2)	85(1)	39(1)	55(1)	30(1)
O(3)	81(1)	85(1)	64(1)	-6(1)	44(1)	5(1)
O(4)	46(1)	102(2)	111(1)	4(1)	42(1)	0(1)
O(5)	68(1)	71(1)	95(1)	-1(1)	53(1)	-14(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for eiict277.

	x	y	z	U(eq)
H(2)	4619	7770	6092	65
H(3)	5138	5650	5564	81
H(4)	4662	2480	5419	87
H(5)	3688	1389	5832	86
H(6)	3140	3507	6360	69
H(11)	2288	10052	5911	78
H(12)	1630	12208	5043	96
H(13)	336	11290	4077	106
H(14)	-238	8206	3963	113
H(15)	449	6011	4812	92
H(17)	4408	10894	7562	67
H(18)	3081	10986	7637	68
H(19)	1807	8982	6915	61
H(21A)	3661	4466	7764	64
H(21B)	4013	6601	7922	64
H(23A)	3578	7120	9574	123
H(23B)	2645	5910	9266	123
H(24A)	2738	9910	9173	222
H(24B)	2375	8835	9614	222
H(24C)	1805	8722	8844	222
H(26A)	529	3131	7144	123
H(26B)	1192	1411	7527	123
H(27A)	1574	2891	8471	276
H(27B)	487	2610	8075	276
H(27C)	930	4638	8093	276