Supporting Information

Direct access to isoxazolino and isoxazolo benzazepines from 2-((hydroxyimino)methyl) benzoic acid via a post-Ugi heteroannulation

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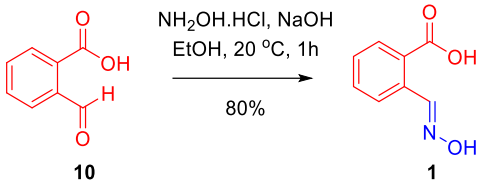
General Methods

Reagents. Unless otherwise stated, reactions were carried out in a small glass balloon at room temperature, no special conditions/atmosphere were needed. Chemicals were purchased from Merck and used without further purification. All solvents were purchased from standard chemical suppliers. Analytical thin layer chromatography (TLC) was performed on silica gel coated plates (Merck 60 F254) with the indicated solvent mixture; visualization was done using ultraviolet (UV) irradiation (λ = 260 nm).

Analytical Methods. All new compounds were characterized by ^{1}H NMR, ^{13}CNMR, IR spectroscopy and HRMS. ^{1}H NMR spectra were recorded on a BRUKER DRX-300 AVANCE (300 MHz), ^{13}C NMR spectra were recorded on a BRUKER DRX-300 (75 MHz) spectrometer. Infrared spectra were recorded on an FTLA 2000 (ABB FT-IR) spectrometer. High-resolution mass spectra were recorded on a Mass-ESI-POS (Apex Qe-FT- ICR instrument) spectrometer. All ^{1}H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for $CHCl_3$ (7.27 ppm). All ^{13}C NMR spectra were reported in ppm relative to residual $CHCl_3$ (77 ppm) and were obtained with ^{1}H decoupling. X-ray analysis was performed on an SIMENS CCD diffractometer and Calculated by SADABS 2006/1 and SHELXTL2001 softwares.

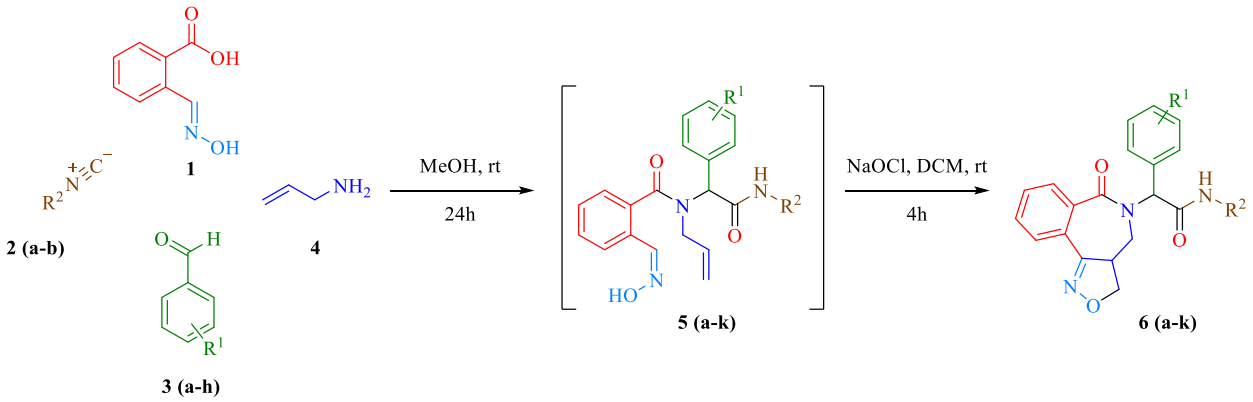
Synthetic procedures

General procedure A: Synthesis of the 2-((hydroxyimino) methyl) benzoic acid



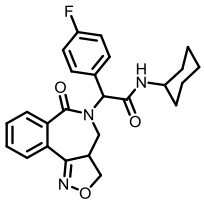
To a solution of hydroxylamine hydrochloride (1.05 equiv) and NaOH (1.05 equiv) in 5 ml EtOH, Phthalaldehyde (1 equiv) was added at room temperature. After 1h stirring, the solution was filtered and the ethanol was evaporated leading to the formation of a Colourless precipitate (80%).

General procedure B: Synthesis of isoxazolino benzazepinones (6a-k) via a post-Ugi heteroannulation involving intramolecular 1,3-dipolar cycloaddition reaction of nitrile oxides with alkenes



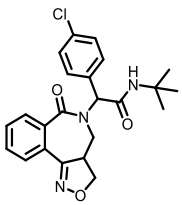
To a solution of aldehyde (1 mmol) in MeOH (5 ml) was added allylamine as a primary amine (1 mmol), and the reaction was stirred at room temperature (25°C) for 30 min. Then 2-((hydroxyimino) methyl) benzoic acid (1 mmol) was added and stirring was continued for 5 min, followed by addition of isocyanide (1 mmol). The mixture was stirred for 24 h. after completion of reaction, the solvent was removed under vacuum. Without any isolation or purification, DCM (5 ml) was added to the residue. Then NaOCl (10%, 1.8 ml) was added to the mixture at 0°C within 15 min. Then, the reaction was stirred at room temperature for 4 h. The progress of the reaction was monitored using TLC (n-hexane/ EtOAc 3:1). Next the reaction was quenched by H_2O (30 ml), the resultant mixture was extracted by CH_2Cl_2 (2 × 30 ml), the combined organic layers were washed with brine (30 ml) and dried over $MgSO_4$. Removal of the solvent followed by recrystallization in MeOH gave the desired product as a solid precipitate.

N-cyclohexyl-2-(4-fluorophenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6a):



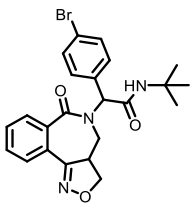
Colourless powder, isolated yield 84%; m.p.: 238-240 ºC; IR (KBr, cm^{-1}): υ = 1643, 2932, 3079, 3256; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.06-1.21 (m, 3H, H-cyclohexyl), 1.25-1.42 (m, 2H, H-cyclohexyl), 1.58-1.71 (m, 3H, H-cyclohexyl), 1.91-1.95 (m, 2H, H-cyclohexyl), 3.26 (*dd*, *J* = 13.7, 8.4Hz, 1H, CH_2O), 3.43 (*dd*, *J* = 15.2, 5.8Hz, 1H, CH_2N), 3.68 (*dd*, J = 15.1, 2.9Hz, 1H, CH_2N), 3.74-3.87 (m, 1H, CH-cyclohexyl), 3.89-4.00 (m, 1H, CHC=N), 4.46 (*dd*, *J* = 10.6, 8.5Hz, 1H, CH_2O), 5.99 (*d*, *J* = 7.0Hz, 1H, NH), 6.32 (s, 1H, CHCON), 7.07-7.13 (m, 2H, H-Ar), 7.36-7.41 (m, 2H, H-Ar), 7.53-7.55 (m, 2H, H-Ar), 7.88-7.91 (m, 1H, H-Ar), 8.02-8.06 (m, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 24.7, 25.4, 32.7, 32.8, 42.7, 48.7, 52.6, 61.1, 71.9, 116.20 (*d*, *J* = 21.5Hz, C-C-F), 126.2, 126.8, 130.4, 131.2, 131.3, 131.6, 132.5, 133.5, 159.0, 162.8 (*d*, *J* = 247.8Hz, C-F), 168.3, 170.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{27}FN_3O_3$ $[M+H]^+$ 436.20318, Found 436.20310. Calc. $C_{25}H_{26}FN_3NaO_3$ $[M+Na]^+$ 458.18509, Found 458.18504.

N-(tert-butyl)-2-(4-chlorophenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6b):



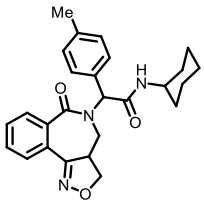
Colourless powder, isolated yield 82%; m.p.: 171-173 ºC; IR (KBr, cm^{-1}): υ = 1647, 2978, 3086, 3312; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.38 (s, 9H, t-Bu), 3.32 (*dd*, *J* = 13.7, 8.4Hz, 1H, CH_2O), 3.41 (*dd*, *J* = 15.1, 6.1Hz, 1H, CH_2N), 3.65 (*dd*, *J* = 15.1, 2.7Hz, 1H, CH_2N), 3.88-3.99 (m, 1H, CHC=N), 4.49 (*dd*, *J* = 10.6, 8.5Hz, 1H, CH_2O), 5.84 (s, 1H, NH), 6.20 (s, 1H, CHCON), 7.33 (*d*, *J* = 8.5Hz, 2H, H-Ar), 7.40 (*d*, *J* = 8.5Hz, 2H, H-Ar), 7.53-7.56 (m, 2H, H-Ar), 7.91-7.94 (m, 1H, H-Ar), 8.04-8.07 (m, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 28.6, 42.8, 52.0, 52.4, 61.6, 72.0, 126.1, 126.8, 129.4, 130.5, 130.7, 131.6, 132.5, 133.5, 133.8, 135.0, 159.1, 168.3, 170.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{23}H_{25}N_3O_3Cl$ $[M+H]^+$ 426.15796, Found 426.15790. Calc. $C_{23}H_{24}N_3NaO_3Cl$ $[M+Na]^+$ 448.13989, Found 448.13984.

2-(4-bromophenyl)-N-(tert-butyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4e] azepin-5(6H)-yl) acetamide (6c):



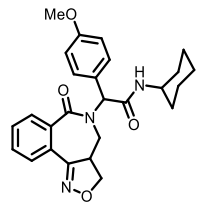
Colourless powder, isolated yield 75%; m.p.: 177-179 ºC; IR (KBr, cm^{-1}): υ = 1643, 2971, 3302; ^{1}H NMR (300 Mz, $CDCl_3$): δ(ppm) = 1.38 (s, 9H, t-Bu), 3.33 (*dd*, *J* = 13.6, 8.3Hz, 1H, CH_2O) 3.40 (*dd*, *J* = 15.1, 6.1Hz, 1H, CH_2N), 3.65 (*dd*, *J* = 15.1, 2.8Hz, 1H, CH_2N), 3.89-3.99 (m, 1H, CHC=N), 4.50 (*dd*, *J* = 10.7, 8.4Hz, 1H, CH_2O), 5.80 (s, 1H, NH), 6.20 (s, 1H, CH), 7.27 (*d*, *J* = 8.2Hz, 2H, H-Ar), 7.51-7.56 (m, 4H, H-Ar), 7.92-7.95 (m, 1H, H-Ar), 8.05-8.08 (m, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 28.6, 42.8, 52.0, 52.4, 61.7, 72.0, 123.1, 126.1, 1268, 130.4, 130.9, 131.6, 132.3, 132.5, 133.4, 134.3, 159.1, 168.3, 170.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{23}H_{24}N_3NaO_3Br$ $[M+Na]^+$ 492.08941, Found 492.08933.

N-cyclohexyl-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e]azepin-5(6H)-yl)-2-(p-tolyl) acetamide (6d):



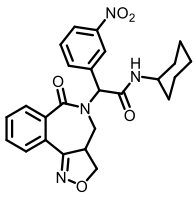
Colourless powder, isolated yield 73%; m.p.: 245-248 ^{o}C; IR (KBr, cm^{-1}): υ = 1640, 2930, 3263; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.02-1.21 (m, 3H, H-cyclohexyl), 1.29-1.41 (m, 2H, H-cyclohexyl), 1.57-1.72 (m, 3H, H-cyclohexyl), 1.91-1.95 (m, 2H, H-cyclohexyl), 2.37 (s, 3H, CH_3), 3.17 (*dd*, *J* = 13.7, 8.4Hz, 1H, CH_2O), 3.44 (*dd*, *J* = 15.3, 5.4, 1H, CH_2N), 3.68 (*dd*, *J* = 15.3, 2.9Hz, 1H, CH_2N), 3.78-3.89 (m, 1H, CH-cyclohexyl), 3.91-4.01 (m, 1H, CHC=N), 4.37 (*dd*, *J* = 10.8, 8.5Hz, 1H, CH_2O), 5.89 (*d*, *J* = 7.86Hz, 1H, NH), 6.31(s, 1H, CHCON), 7.21 (*d*, *J* = 8.1Hz, 2H, H-Ar), 7.26 (*d*, *J* = 8.1Hz, 2H, H-Ar), 7.49-7.54 (m, 2H, H-Ar), 7.83-7.86 (m, 1H, H-Ar), 8.02-8.05 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 21.2, 24.7, 24.8, 25.4, 32.8, 42.5, 48.7, 52.8, 61.9, 71.8, 126.2, 126.7, 129.5, 129.9, 130.3, 131.5, 132.2, 132.4, 133.8, 139.0, 159.2, 168.8, 170.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{26}H_{29}N_3NaO_3$ $[M+Na]^+$ 454.21015, Found 454.21011.

N-cyclohexyl-2-(4-methoxyphenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6e):



Colourless powder, isolated yield 80%; m.p.: 219-221 ^{o}C; IR (KBr, cm^{-1}): υ = 1643, 2933, 3263; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.08-1-25 (m, 3H, H-cyclohexyl), 1.25-1.52 (m, 2H, H-cyclohexyl), 1.55-1.80 (m, 3H, H-cyclohexyl), 1.92 (brs, 2H, H-cyclohexyl), 3.17 (*dd*, *J* = 12.9, 8.3Hz, 1H, CH_2O), 3.43-3.47 (m, 1H, CH_2N), 3.67-3.72 (m, 1H, CH_2N), 3.82 (s, 3H, CH_3), 3.75-3.85 (m, 1H, CH-cyclohexyl), 3.85-3.95 (m, 1H, CHC=N), 4.34-4.40 (m, 1H, CH_2O), 5.83 (*d*, *J* = 6.2Hz, 1H, NH), 6.27 (s, 1H, CHCON), 6.92 (*d*, *J* = 7.5Hz, 2H, H-Ar), 7.31 (*d*, *J* = 7.7Hz, 2H, H-Ar), 7.52 (brs, 2H, H-Ar), 7.81 (brs, 1H, H-Ar), 8.04 (s, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 24.7, 25.4, 32.8, 42.3, 48.7, 52.9, 55.3, 61.6, 71.7, 114.5, 126.3, 126.7, 127.0, 130.3, 131.0, 131.5, 132.4, 133.8, 159.2, 160.0, 168.9, 170.6; Mass: HR-MS (ESI-POS) = Calc. for $C_{26}H_{29}N_3NaO_4$ $[M+Na]^+$ 470.20507, Found 470.20503.

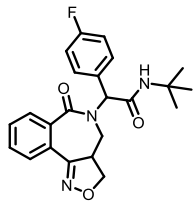
N-cyclohexyl-2-(3-nitrophenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6f):



Yellow powder, isolated yield 66%; m.p.: 180-182 ^{o}C; IR (KBr, cm^{-1}): υ = 1638, 1674, 3266; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 1.11-1.25 (m, 3H, H-cyclohexyl), 1.35-1.41 (m, 2H, H-cyclohexyl), 1.60-1.26 (m, 3H, H-cyclohexyl), 1.91-2.00 (m, 2H, H-cyclohexyl), 3.42 (*dd*, *J* = 14.8, 7.5Hz, 1H, CH_2O), 3.53 (*dd*, *J* = 13.5, 8.5Hz, 1H, CH_2N), 3.65-3.75 (m, 1H, CH_2N), 3.81-4.01 (m, 2H, CH-cyclohexyl, CHC=N), 4.55-4.69 (m, 1H, CH_2O), 6.30 (*d*, *J* = 7.5Hz, 1H, NH), 6.49 (s, 1H, CHCON), 7.50-

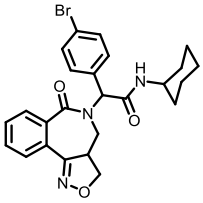
7.74 (m, 4H, H-Ar), 8.04-8.09 (m, 2H, H-Ar), 8.24 (*d*, *J* = 9.2Hz, 2H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 24.7, 25.3, 32.7, 32.8, 43.6, 48.9, 51.7, 60.1, 72.5, 123.6, 125.8, 126.9, 130.1, 130.6, 131.8, 132.8, 132.9, 134.9, 137.4, 148.6, 158.9, 167.1, 170.4; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{27}N_4O_5$ $[M+1]^+$ 463.1994, Found 463.1976. Calc. $C_{25}H_{26}N_4NaO_5$ $[M+Na]^+$ 485.1810, Found 485.1795.

N-(tert-butyl)-2-(4-fluorophenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6g):



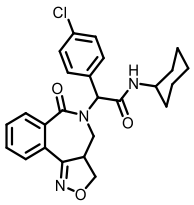
Colourless powder, isolated yield 79%; m.p.: 168-171 ºC; IR (KBr, cm^{-1}): υ = 1643, 2974, 3306; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.39 (s, 9H, *t*-Bu), 3.26 (*dd*, *J* = 13.7, 8.4Hz, 1H, CH_2O), 3.43 (*dd*, *J* = 15.2, 5.8Hz, 1H, CH_2N), 3.67 (*dd*, *J* =15.2, 2.9Hz, 1H, CH_2N), 3.87-3.98 (m, 1H, CHC=N), 4.45 (*dd*, *J* = 10.7, 8.4Hz, 1H, CH_2O), 5.71 (s, 1H, NH), 6.20 (s, 1H, CHCON), 7.09-7.14 (m, 2H, H-Ar), 7.39 (*dd*, *J* = 8.5, 5.3Hz, 2H, H-Ar), 7.52-7.59 (m, 2H, H-Ar), 7.88-7.92 (m, 1H, H-Ar), 8.01-8.10 (m, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 28.6, 51.9, 52.6, 61.5, 116.1, 116.3, 126.2, 126.8, 130.4, 131.1, 131.2, 131.3, 131.6, 132.5, 133.6, 159.1, 162.5 (d, J=225, C-F), 168.6, 170.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{23}H_{25}FN_3O_3$ $[M+H]^+$ 410.18751, Found 410.18745. Calc. $C_{23}H_{24}FN_3NaO_3$ $[M+Na]^+$ 432.16944, Found 432.16939.

2-(4-bromophenyl)-N-cyclohexyl-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6h):



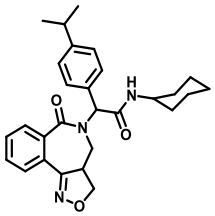
Colourless powder, isolated yield 83%; m.p.: 261-264 ºC; IR (KBr, cm^{-1}): υ = 1646, 2926, 3261; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.08-1.15 (m, 3H, H-cyclohexyl), 1.29-1.36 (m, 2H, H-cyclohexyl), 1.57-1.65 (m, 3H, H-cyclohexyl), 1.89-1.93 (m, 2H, H-cyclohexyl), 3.33 (*dd*, *J* = 13.5, 8.4Hz, 1H, CH_2O), 3.41 (*dd*, *J* = 15.2, 6.0Hz, 1H, CH_2N), 3.66 (*dd*, *J* = 15.2, 2.3Hz, 1H, CH_2N), 3.77-3.82 (m, 1H, CH-cyclohexyl), 3.88-3.97 (m, 1H, CHC=N), 4.50 (*dd*, *J* = 8.4, 10.5, 1H, CH_2O), 6.12 (*d*, *J* = 7.5Hz, 1H, NH), 6.34 (s, 1H, CHCON), 7.30-7.40 (m, 4H, H-Ar), 7.51-7.54 (m, 2H, H-Ar), 7.91-7.95 (m, 1H, H-Ar), 7.99-8.02 (m, 1H, H-Ar) ; ^{13}C NMR (75 MHz, DMSO-d_6): δ(ppm) = 24.4, 24.5, 25.1, 32.1, 42.2, 47.9, 52.6, 60.8, 70.9, 121.7, 126.0, 126.3, 130.3, 131.6, 131.8, 132.0, 133.7, 135.8, 159.0, 167.9, 169.5; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{27}N_3O_3Br$ $[M+H]^+$ 498.12104, Found 498.12098. Calc. $C_{25}H_{26}N_3NaO_3Br$ $[M+Na]^+$ 518.10504, Found 518.10498.

2-(4-chlorophenyl)-N-cyclohexyl-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6i):



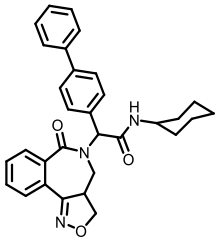
Colourless powder, isolated yield 87%; m.p.: 256-258 ºC; IR (KBr, cm^{-1}): υ = 1638, 2931, 3260; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.08-1.15 (m, 3H, H-cyclohexyl), 1.29-1.36 (m, 2H, H-cyclohexyl), 1.57-1.75 (m, 3H, H-cyclohexyl), 1.89-1.93 (m, 2H, H-cyclohexyl), 3.33 (*dd*, *J* = 13.8, 8.5Hz, 1H, CH_2O), 3.41 (*dd*, *J* = 15.0, 6.1Hz, 1H, CH_2N), 3.66 (*dd*, *J* = 15.2, 2.3Hz, 1H, CH_2N), 3.77-3.82 (m, 1H, CH-cyclohexyl), 3.88-3.97 (m, 1H, CHC=N), 4.50 (*d*, *J* = 8.5, 10.5Hz, 1H, CH_2O), 6.12 (*d*, *J* = 7.7Hz, 1H, NH), 6.34 (s, 1H, CH), 7.22-7.40 (m, 4H, H-Ar), 7.51-7.54 (m, 2H, H-Ar), 7.91-7.95 (m, 1H, H-Ar), 7.99-8.02 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.7, 25.3, 32.8, 42.9, 48.7, 52.4, 61.1, 72.1, 126.1, 126.8, 129.4, 130.4, 130.6, 131.7, 132.5, 133.4, 133.7, 135.0; Mass: HR-MS (ESI-POS) = Cal. for $C_{25}H_{27}N_3O_3Cl$ $[M+H]^+$ 452.17363, Found 452.17355. Calc. $C_{25}H_{26}N_3NaO_3Cl$ $[M+Na]^+$ 474.15556, Found 474.15549.

N-cyclohexyl-2-(4-isopropylphenyl)-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (6j):



Colourless powder, isolated yield 84%; m.p.: 183-186 ºC; IR (KBr, cm^{-1}): υ = 1633, 2926, 3304; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 1.10-1.15 (m, 3H, H-cyclohexyl), 1.25 (*d*, *J* = 6.7Hz, 6H, CH_3-iso), 1.32-1.35 (m, 2H, H-cyclohexyl), 1.56-1.76 (m, 3H, H-cyclohexyl), 1.91-1.94 (m, 2HJ, H-cyclohexyl), 2.87-2.94 (m, 1H, CH-iso), 3.12 (*dd*, *J* = 13.5, 8.4Hz, 1H, CH_2O), 3.44 (*dd*, *J* = 15.2, 4.8Hz, 1H, CH_2N), 3.69 (*d*, *J* = 13.0Hz, 1H, CH_2N), 3.80-3.97 (m, 2H, CHCN, CH-cyclohexyl), 4.32 (*t*, *J* = 9.5Hz, 1H, CH_2N), 5.89 (*d*, *J* = 7.2Hz, 1H, NH), 6.31 (s, 1H, CHCO), 7.25-7.27 (m, 4H, H-Ar), 7.48-7.51 (m, 2H, H-Ar), 7.81-7.83 (m, 1H, H-Ar), 8.01-8.03 (m, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 23.8, 23.9, 24.7, 24.8, 25.4, 32.8, 32.9, 33.8, 42.5, 48.8, 52.8, 61.9, 71.7, 126.2, 126.7, 127.2, 129.6, 130.3, 131.5, 132.4, 132.5, 133.8, 150.0, 159.2, 168.8, 170.6. Mass: HR-MS (ESI-POS) = Calc. for $C_{28}H_{33}N_3NaO_3$ $[M+Na]^+$ 482.2410, Found 482.2414. Calc. for $C_{28}H_{33}KN_3O_3$ $[M+K]^+$ 498.2153, Found 498.2154.

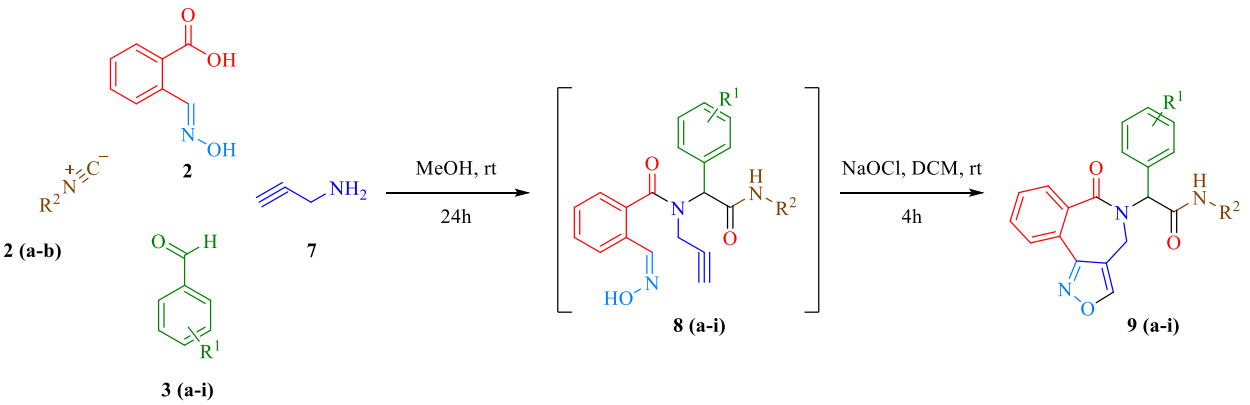
2-([1,1'-biphenyl]-4-yl)-N-cyclohexyl-2-(6-oxo-3a,4-dihydro-3H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl)acetamide (6k):



Colourless powder, isolated yield 86%; m.p.: 187-190 ºC; IR (KBr, cm^{-1}): υ =1649,2926, 3288; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) = 1.11-1.14 (m, 3H, H-cyclohexyl), 1.34-1.38 (m, 2H, H-cyclohexyl), 1.58-1.68 (m, 3H, H-cyclohexyl), 1.94-1.96 (m, 2H, H-cyclohexyl), 3.30 (*dd*, *J* = 12.6, 9.0Hz, 1H, CH_2O), 3.48 (*dd*, *J* = 14.8, 4.6Hz, 1H, CH_2N), 3.73 (*d*, *J* = 14.8Hz, 1H, CH_2N), 3.86 (brs, 1H, CH-cyclohexyl), 4.00 (brs, 1H, CHC=N), 4.44 (*t*, *J* = 9.0Hz, 1H, CH_2O), 6.03 (*d*, *J* = 6.5Hz, 1H, NH), 6.40 (s, 1H, CHCON), 7.38-7.46 (m, 5H, H-Ar), 7.53 (brs,

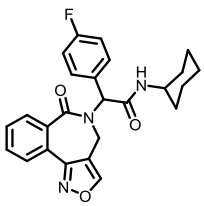
2H, Ar), 7.58-7.65 (m, 4H, H-Ar), 7.89 (brs, 1H, H-Ar), 8.05 (brs, 1H, H-Ar); ^{13}C NMR (75 MHz, $CDCl_3$): δ(ppm) = 24.8, 25.4, 32.8, 32.9, 42.9, 48.8, 52.6, 61.8, 72.0,126.2, 126.7, 127.0, 127.8, 128.9, 129.9, 130.4, 131.6, 132.5, 133.6, 134.1, 139.9, 141.8, 159.2, 168.5, 170.5. Mass: HR-MS (ESI-POS) = Calc. for $C_{31}H_{31}N_3NaO_3$ $[M+Na]^+$ 516.2266, Found 516.2258. Calc. for $C_{31}H_{31}KN_3O_3$ $[M+K]^+$ 532.1997, Found 532.1997.

General procedure B: Synthesis of isoxazolo benzazepinones (9a-i) via a post-Ugi heteroannulation involving intramolecular 1,3-dipolar cycloaddition reaction of nitrile oxides with alkynes



To a solution of aldehyde (1 mmol) in MeOH (5 ml) was added propargylamine as a primary amine (1 mmol), and the reaction was stirred at room temperature (25°C) for 30 min. Then 2-((hydroxyimino) methyl) benzoic acid (1 mmol) was added and stirring was continued for 5 min, followed by addition of isocyanide (1 mmol). The mixture was stirred for 24 h. after completion of reaction, the solvent was removed under vacuum. Without any isolation or purification, DCM (5 ml) was added to the residue. Then NaOCl (10%, 1.8 ml) was added to the mixture at 0°C within 15 min. Then, the reaction was stirred at room temperature for 4 h. The progress of the reaction was monitored using TLC (n-hexane/ EtOAc 3:1). Next the reaction was quenched by H_2O (30 ml), the resultant mixture was extracted by CH_2Cl_2 (2 × 30 ml), the combined organic layers were washed with brine (30 ml) and dried over $MgSO_4$. Removal of the solvent followed by recrystallization in MeOH gave the desired product as a solid precipitate.

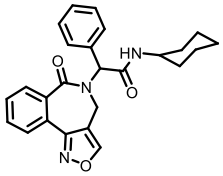
N-cyclohexyl-2-(4-fluorophenyl)-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (9a):



Colourless powder, isolated yield 83%; m.p.: 216-218 °C; IR (KBr, cm^{-1}): υ = 1641, 2934, 3283; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.11-1.21 (m, 3H, H-cyclohexyl), 1.30-1.38 (m, 2H, H-cyclohexyl), 1.59-1.73 (m, 3H, H-cyclohexyl), 1.94-2.04 (m, 2H, H-cyclohexyl), 3.80-3.91 (m, 1H, CH-cyclohexyl), 3.79-3.94 (m, 2H, CH-cyclohexyl, CH_2N), 4.05-4.12 (m, 1H, CH_2N), 6.07-16 (m, 2H, NH, CHCON), 7.09 (*t*, *J* = 8.6Hz, 2H, H-Ar),7.51-7.60 (m, 5H, H-Ar,=CH), 7.64-7.70 (m, 1H, H-Ar), ^{13}C NMR (75 MHz, DMSO-d_6): δ(ppm) = 24.2, 24.9, 31.9,47,8, 114.8, 115.1, 116.2, 126.5, 129.4, 130.9, 132.3,

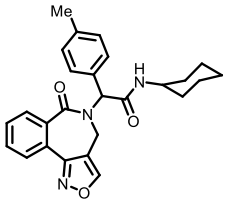
132.7, 139.0, 160.3, 163.6, 167.1; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{24}FN_3NaO_3$ $[M+Na]^+$ 456.14689, Found 456.14915.

N-cyclohexyl-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl)-2-phenylacetamide (9b):



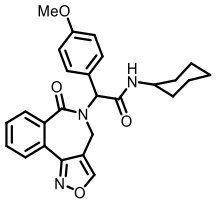
Colourless powder, isolated yield 68%; m.p.: 190-192 ºC; IR (KBr, cm^{-1}): υ = 1641, 2931, 3272; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.14-1.21(m, 3H, H-cyclohexyl), 1.30-1.42 (m, 2H, H-cyclohexyl), 1.60-1.73 (m, 3H, H-cyclohexyl), 1.94-1.98 (m, 2H, H-cyclohexyl), 3.81-3.93 (m, 2H, CH-cyclohexyl, CH_2N), 4.02-4.1 (m, 1H, CH_2), 6.04-6.16 (m, 2H, NH, CHCON), 7.37-7.43 (m, 4H, =CH, H-Ar), 7.51-7.56 (m, 3H, H-Ar), 7.61-7.68 (m, 2H, H-Ar), 7.74 (*d*, *J* = 7.6Hz, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.8, 24.9, 5.4, 32.7, 48.9, 62.0, 72.7, 107.5, 110.5, 116.8, 127.3, 129.0, 129.7, 130.0, 132.8, 133.2, 133.9, 139.3, 167.5, 168.8; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{25}N_3NaO_3$ $[M+Na]^+$ 438.15849, Found 438.15841.

N-cyclohexyl-2-(6-oxo-4H benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl)-2-(p-tolyl)acetamide (9c):



Colourless powder isolated yield 76%; m.p.: 205-207 ºC; IR (KBr, cm^{-1}): υ = 1640, 2933, 3308; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 1.06-1.11 (m, 3H, H-cyclohexyl), 1.34-1.42 (m, 2H, H-cyclohexyl), 1.57-1.69 (m, 3H, H-cyclohexyl), 1.81-1.95 (m, 2H, H-cyclohexyl), 2.38 (s, 3H, CH_3), 3.80-3.83 (m, 1H, CH-cyclohexyl), 4.31 (brs, 2H, CH_2N), 6.02 (*d*, *J* = 7.4Hz, 1H, NH), 6.44 (s, 1H, CHCON), 7.16 (*d*, *J* = 7.7Hz, 2H, H-Ar), 7.22 (*d*, *J* = 7.8Hz, 2H, H-Ar), 7.57-7.60 (m, 3H, =CH, H-Ar), 7.85-7.88 (m, 1H, H-Ar), 8.05-8.08 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 21.1, 24.7, 25.4, 32.6, 32.6, 32.8, 36.7, 48.6, 60.4, 117.7, 125.3, 127.3, 129.0, 129.4, 130.3, 131.5, 132.2, 132.6, 134.6, 138.6, 153.0, 161.1, 168.2, 169.0; Mass: HR-MS (ESI-POS) = Calc. for $C_{26}H_{28}N_3O_3$ $[M+H]^+$ 430.21260, Found 430.21252. Calc. $C_{26}H_{27}N_3NaO_3$ $[M+Na]^+$ 452.19453, Found 452.19446.

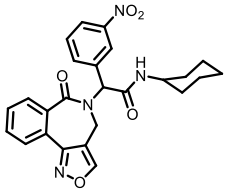
N-cyclohexyl-2-(4-methoxyphenyl)-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl)acetamide (9d):



Colourless powder, isolated yield 70% m.p.: 196-199 ºC; IR (KBr, cm^{-1}): υ = 1636, 2931, 3274 ^{1}H NMR (300MHz, $CDCl_3$): δ(ppm) = 1.05-1.12 (m, 3H, H-cyclohexyl), 1.30-1.36 (m, 2H, H-cyclohexyl), 1.56-1.68 (m, 3H, H-cyclohexyl), 1.83-1.93 (m, 2H, H-cyclohexyl), 3.79-3.85 (m, 1H, CH-cyclohexyl), 3.83 (s, 3H, OCH_3), 4.25-4.35 (m, 2H, CH_2N), 6.08 (*d*, *J* = 7.9Hz, 1H, NH), 6.42 (s, 1H, CHCON), 6.87 (*d*, *J* = 8.7Hz, 2H, H-Ar), 7.25 (*d*, *J* = 5.0Hz, 2H, H-Ar), 7.55-7.65 (m, 3H, =CH, H-Ar), 7.83-7.87 (m, 1H, H-Ar), 8.03-8.07 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.7, 25.4, 32.6, 32.8, 36.5, 48.5, 55.4, 60.1, 114.0, 117.8, 125.3, 127.1, 127.3, 130.3,

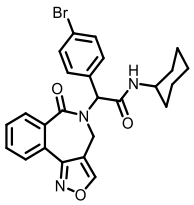
130.4, 131.6, 132.6, 134.6, 152.9, 159.7, 161.1, 168.3, 168.9; Mass: HR-MS (ESI-POS) = Calc. for $C_{26}H_{28}N_3O_4$ $[M+H]^+$ 446.20751, Found 446.20743. Calc. $C_{26}H_{27}N_3NaO_4$ 468.18944, Found 468.18938.

N-cyclohexyl-2-(3-nitrophenyl)-2-(6-oxo-4Hbenzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl)acetamide (9e):



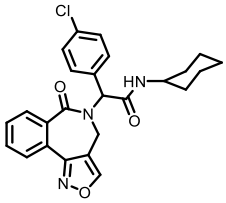
Colourless powder, Isolated yield 67%; m.p.: 194-196 ºC; IR (KBr, cm^{-1}): υ = 1638, 2933, 3264; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 0.93-1.13 (m, 3H, H-cyclohexyl), 1.09-1.40 (m, 2H, H-cyclohexyl), 1.55-1.67 (m, 4H, H-cyclohexyl), 1.9-1.93 (m, 1H, H-cyclohexyl), 3.72-3.78 (m, 1H, CH-cyclohexyl), 4.28 (*d*, *J* = 15.1Hz, 1H, CH_2N), 4.48 (*d*, *J* = 15.4Hz, 1H, CH_2N), 6.51 (*d*, *J* = 7.7Hz, 1H, NH), 6.60 (s, 1H, CHCON), 7.49-7.58 (m, 1H, H-Ar), 7.60-7.63 (m, 3H, =CH, H-Ar), 7.88-7.91 (m, 1H, H-Ar), 8.03-8.06 (m, 2H, H-Ar), 8.21 (*d*, *J* = 8.1Hz, 1H, H-Ar), 8.26 (s, 1H, H-Ar); ^{13}C NMR (75 Mz, $CDCl_3$): δ(ppm) = 24.6, 25.3, 32.3, 32.7, 36.8, 48.7, 59.2, 117.1, 123.2, 123.4, 125.3, 127.6, 129.8, 130.5, 132.1, 132.5, 133.9, 134.6, 137.5, 148.4, 153.8, 160.9, 166.6, 169.6; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{25}N_4O_5$ $[M+1]^+$ 461.1833, Found 461.1819. Calc. $C_{25}H_{24}N_4NaO_5$ $[M+Na]^+$ 483.1651, Found 483.1639.

2-(4-bromophenyl)-N-cyclohexyl-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (9f):



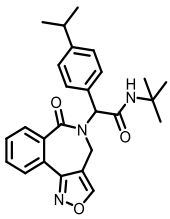
Colourless powder, isolated yield 90%; m.p.: 213-216 ºC; IR (KBr, cm^{-1}): υ = 1648, 2931, 3270; ^{1}H NMR (300 MHz, $CDCl_3$): δ(ppm) = 0.98-1.11 (m, 3H, H-cyclohexyl), 1.28-1.1.36 (m, 2H, H-cyclohexyl), 1.55-1.67 (m, 3H, H-cyclohexyl), 1.73-1.78 (m, 1H, H-cyclohexyl), 1.87-1.90 (m, 1H, H-cyclohexyl), 3.72-3.82 (m, 1H, CH-cyclohexyl), 4.33 (brs, 2H, CH_2N), 6.28 (*d*, *J* = 7.9Hz, 1H, NH), 6.46 (s, 1H, CHCON), 7.21 (*d*, *J* = 8.4, 2H, H-Ar), 7.48 (*d*, *J* = 8.4, 2H, H-Ar), 7.54-7.63 (m, 2H, H-Ar), 7.8 (brs, 1H, =CH), 7.86-7.89 (m, 1H, H-Ar), 8.01-8.04 (m, 1H, H-Ar); ^{13}C NMR (75Mz, $CDCl_3$): δ(ppm) = 24.7, 25.3, 32.5, 32.8, 36.7, 48.6, 59.7, 117.5, 122.7, 125.3, 127.4, 130.3, 130.5, 131.8, 131.9, 132.5, 134.3, 153.2, 161.0, 167.5, 169.2; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{25}N_3O_3Br$ $[M+H]^+$ 494.10749, Found 494.10738. Cal. $C_{25}H_{24}N_3NaO_3Br$ $[M+Na]^+$ 516.08940, Found 516.08933.

2-(4-chlorophenyl)-N-cyclohexyl-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (9g):



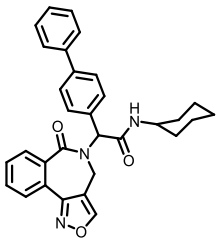
Colourless powder, isolated yield 92%; m.p.: 227-230 ^{o}C; IR (KBr, cm^{-1}): υ = ^{1}H NMR (300MHz, $CDCl_3$): δ(ppm) = 0.98-1.1 (m, 3H, H-cyclohexyl), 1.27-1.35 (m, 2H, H-cyclohexyl), 1.54-1.66 (m, 3H, H-cyclohexyl), 1.73-1.80 (m, 1H, H-cyclohexyl), 1.86-1.90 (m, 1H, H-cyclohexyl), 3.72-3.82 (m, 1H, CH-cyclohexyl), 4.33 (brs, 2H, CH_2N), 6.30-6.33 (*d*, *J* = 7.9Hz, 1H, NH), 6.49 (s, 1H, CHCON), 7.26 (*d*, *J* = 8.4Hz, 2H, H-Ar), 7.32 (*d*, *J* = 8.5Hz, 2H, H-Ar), 7.56-7.63 (m, 2H, H-Ar), 7.75-7.89 (m, 2H, =CH, H-Ar), 7.99-8.03 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.7, 25.3, 32.5, 32.8, 36.7, 48.6, 59.6, 117.5, 125.3, 127.4, 128.9, 130.3, 131.8, 132.5, 133.8, 134.3, 134.6, 153.2, 161.0, 167.6, 169.2; Mass: HR-MS (ESI-POS) = Calc. for $C_{25}H_{25}N_3O_3Cl$ $[M+H]^+$ 450.15799, Found 450.15790. Calc. $C_{25}H_{24}N_3NaO_3Cl$ $[M+Na]^+$ 472.13991, Found 472.13984.

N-(tert-butyl)-2-(4-isopropylphenyl)-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (9h):



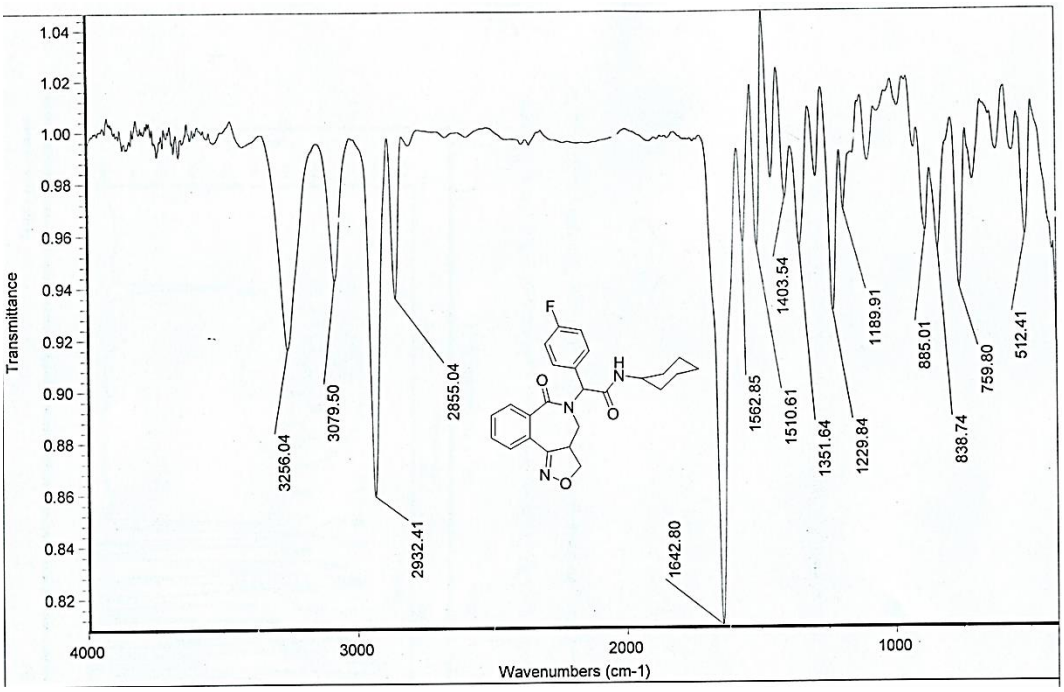
Colourless powder, isolated yield 64%; m.p.: 227-230 ^{o}C; IR (KBr, cm^{-1}): υ = 1611, 1688, 2959, 3298; ^{1}H NMR (300MHz, $CDCl_3$): δ(ppm) = 1.14 (*d*, *J* = 6.7Hz, 1H, CH_3-iso), 1.27 (*d*, *J* = 6.7Hz, 5H, CH_3-iso), 1.35 (s, 9H, t-Bu), 2.93 (sep, *J* = 6.7Hz, 1H, CH-iso), 4.12-4.52 (m, 2H, CH_2N), 5.77 (s, 1H, NH), 6.37 (s , 1H, CHCO), 7.13-7.32 (m, 4H, H-Ar), 7.39-7.45 (m, 1H, H-Ar), 7.52-7.59 (m, 2H, H-Ar), 7.81-7.86 (m, 1H, H-Ar), 8.06-8.10 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.0, 28.6, 33.9, 36.7, 51.9, 60.6, 117.8, 125.2, 126.8, 127.3, 129.1, 130.3, 131.5, 132.6, 132.8, 134.7, 149.7, 161.0, 168.2, 168.6, 168.9; Mass: HR-MS (ESI-POS) = Calc. for $C_{26}H_{29}N_3NaO_3$ $[M+Na]^+$ 454.2101, Found 454.2101. Calc. for $C_{26}H_{29}KN_3O_3$ $[M+K]^+$ 470.1844, Found 4701840.

2-([1,1'-biphenyl]-4-yl)-N-cyclohexyl-2-(6-oxo-4H-benzo[c]isoxazolo[3,4-e] azepin-5(6H)-yl) acetamide (9i):

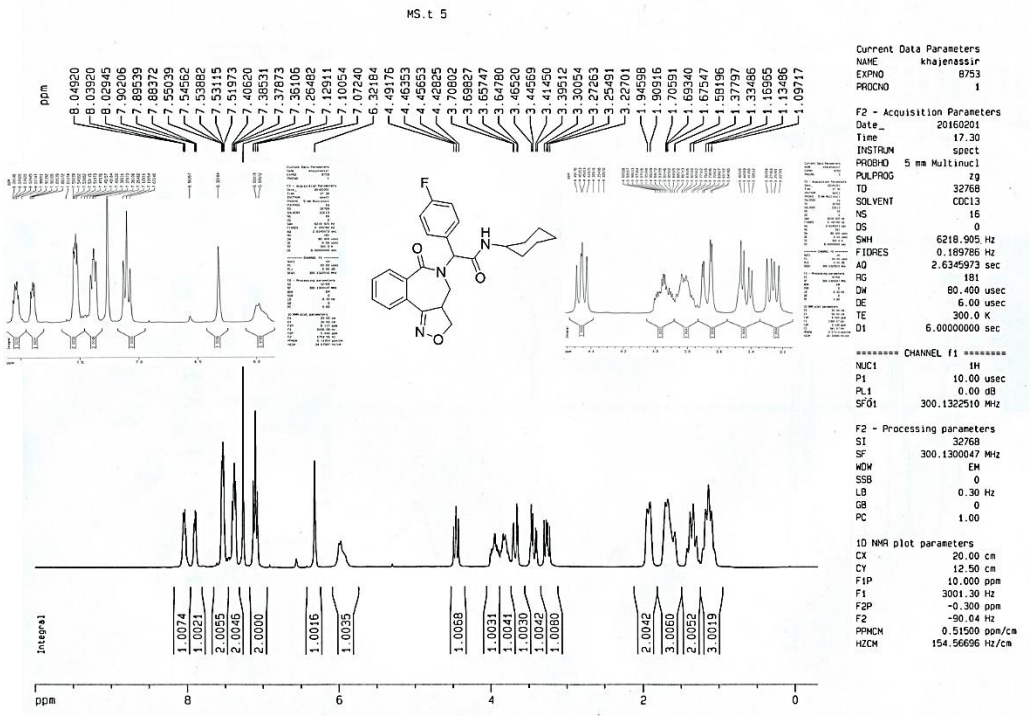


Colourless powder, isolated yield 80%; m.p.: 229-233 ^{o}C; IR (KBr, cm^{-1}): υ = 1638, 2926, 3282; ^{1}H NMR (300MHz, $CDCl_3$): δ(ppm) = 1.08-1.15 (m, 3H, H-cyclohexyl), 1.26-1.38 (m, 2H, H-cyclohexyl), 1.58-1.69 (m, 3H, H-cyclohexyl), 1.82-1.99 (m, 2H, H-cyclohexyl), 3.82-3.85 (m, 1H, CH-cyclohexyl), 4.37 (brs, 2H, CH_2N), 6.17 (*d*, *J* = 7.3Hz, 1H, NH), 6.55 (s, 1H, CHCON), 7.38-7.41 (m, 3H, H-Ar), 7.44-7.49 (m, 2H, H-Ar), (brs, 7H, H-Ar), 7.86-7.88 (m, 1H, H-Ar), 8.06-8.08 (m, 1H, H-Ar); ^{13}C NMR (75MHz, $CDCl_3$): δ(ppm) = 24.7, 25.4, 32.6, 32.8, 32.8, 48.6, 60.3, 117.7, 125.3, 127.0, 127.3, 127.8, 128.9, 129.4, 130.3, 131.6, 132.6, 134.2, 134.5, 140.0, 141.5, 161.0, 168.0, 169.1; Mass: HR-MS (ESI-POS) = Calc. for $C_{31}H_{29}N_3NaO_3$ $[M+Na]^+$ 514.2123, Found 514.2101. Calc. for $C_{31}H_{29}KN_3O_3$ $[M+K]^+$ 530.1868, Found 530.1840.

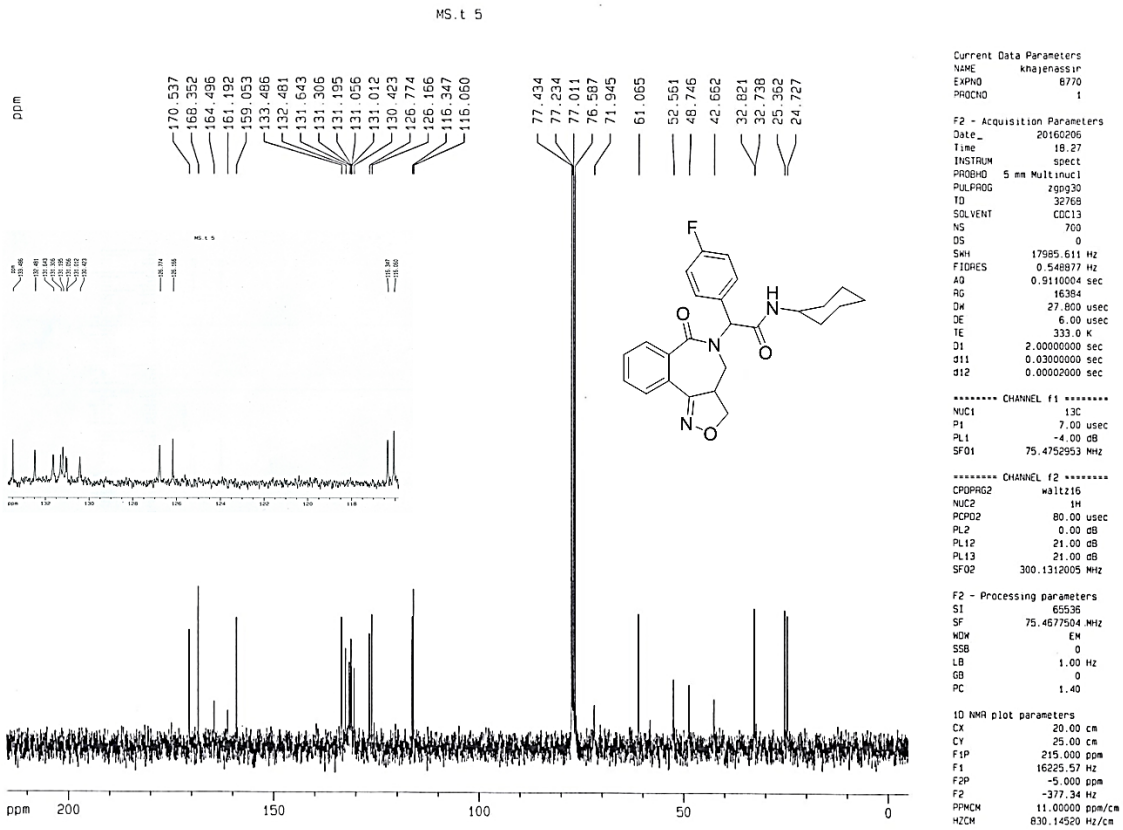
Copies of Spectra



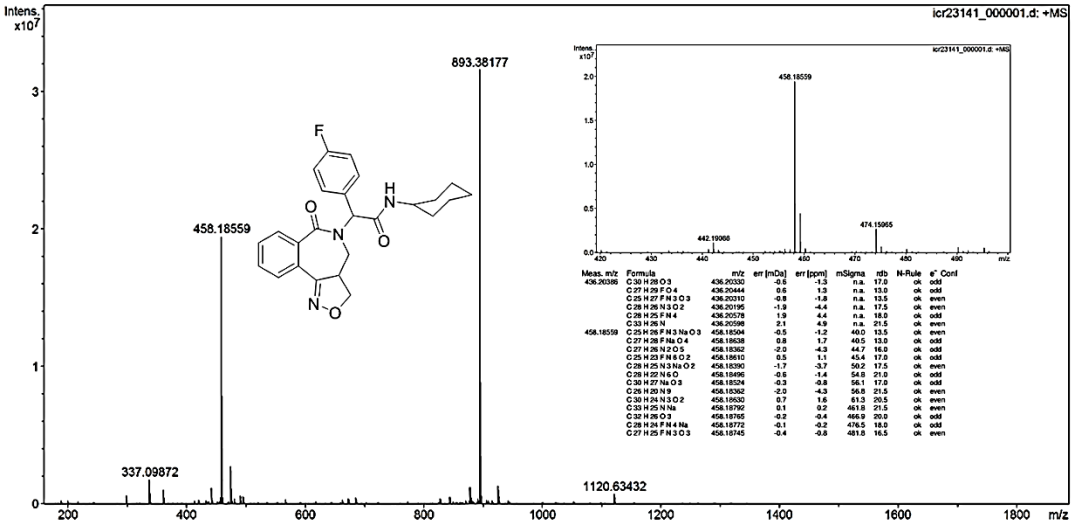
IR (KBr) (6a)



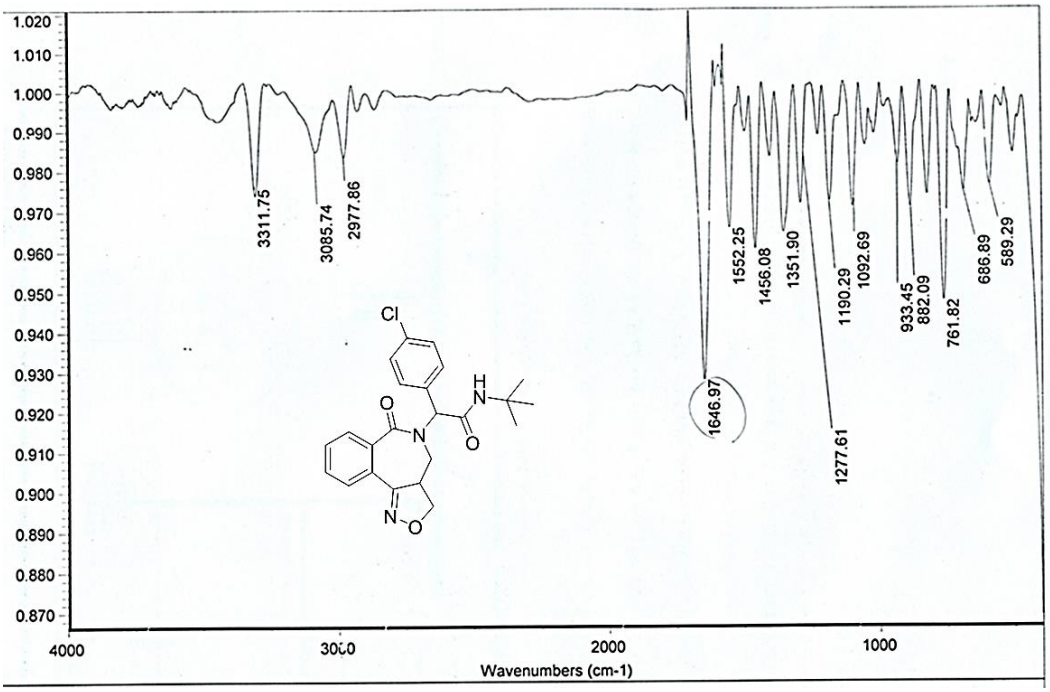
^{1}HNMR (300MHz, $CDCl_3$) (6a)



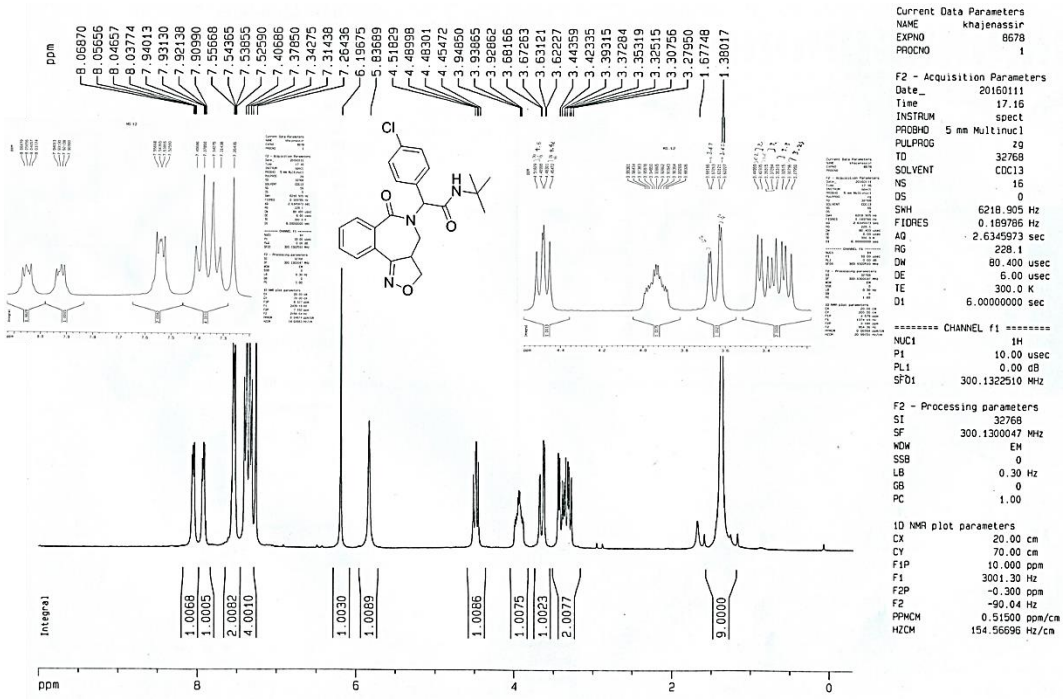
^{13}CNMR (75MHz, $CDCl_3$) (6a)



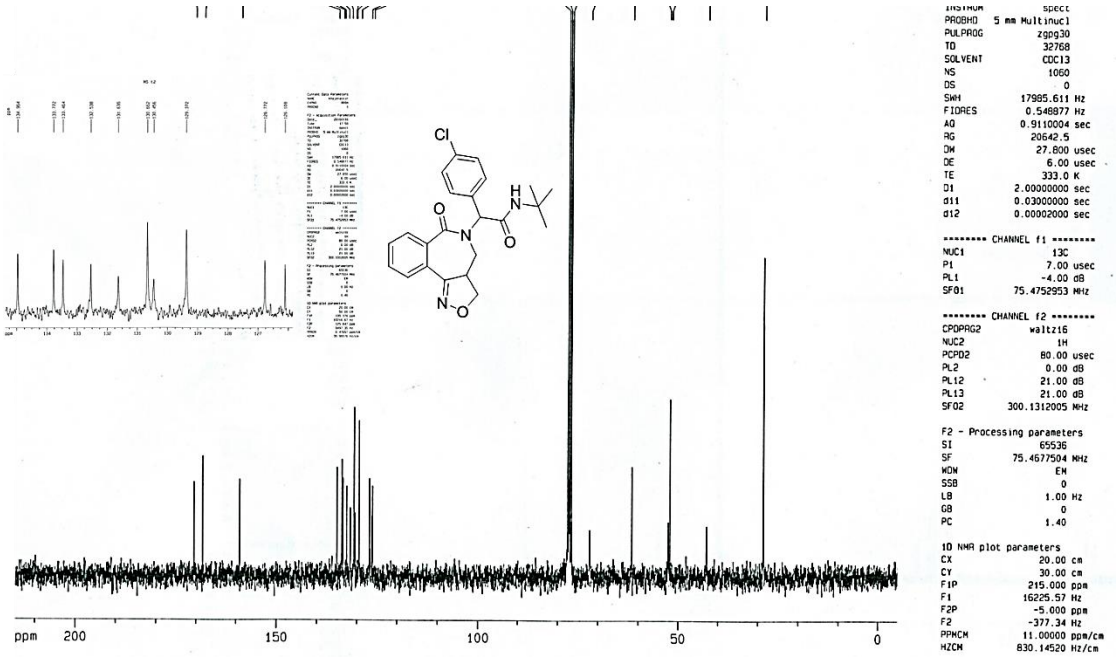
ESI-HRMS (6a)



IR (KBr) (6b)

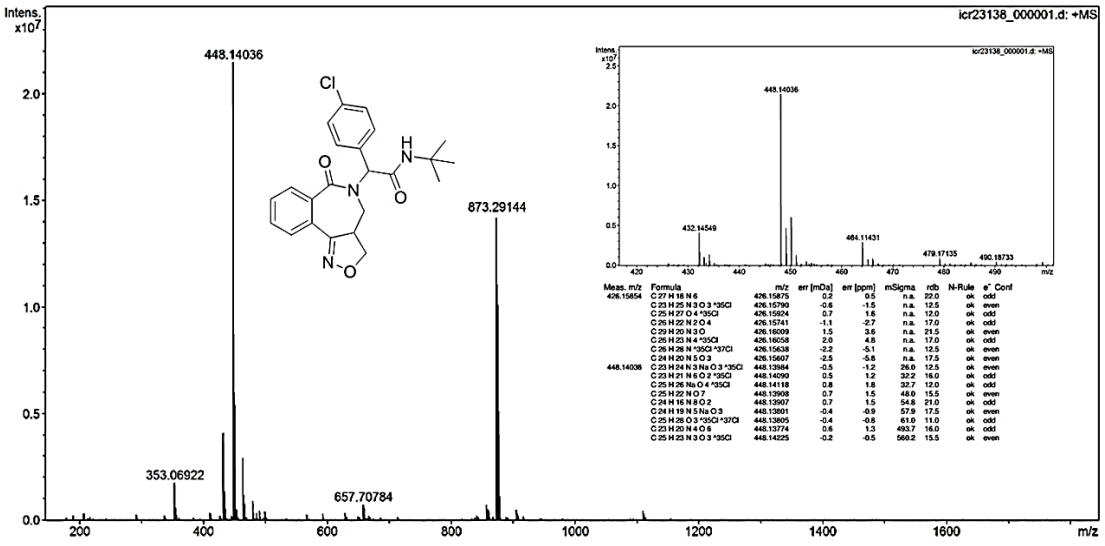


^{1}HNMR (300MHz, $CDCl_3$) (6b)



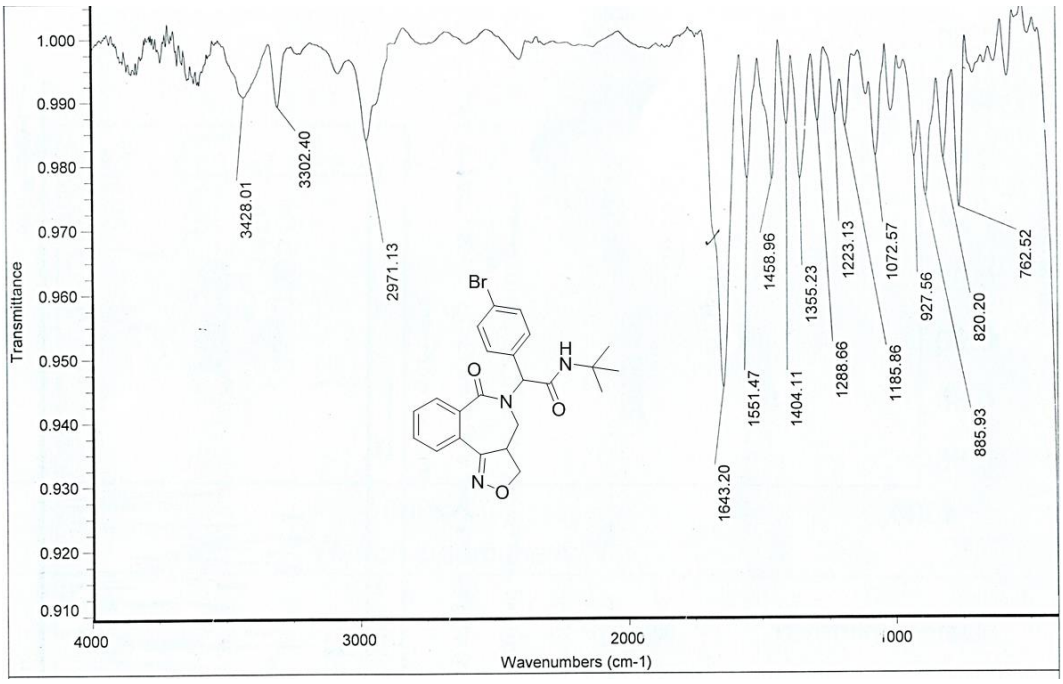
INSTHUM	spect
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1060
DS	0
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110004 sec
RG	20642.5
DW	27.800 usec
DE	6.00 usec
TE	333.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	7.00 usec
PL1	-4.00 dB
SF01	75.4752953 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	21.00 dB
PL13	21.00 dB
SF02	300.1312005 MHz
F2 - Processing parameters	
SI	65536
SF	75.4677504 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
1D NMR plot parameters	
CX	20.00 cm
CY	30.00 cm
F1P	215.000 ppm
F1	16225.57 Hz
F2P	-5.000 ppm
F2	-377.34 Hz
PPMCM	11.00000 ppm/cm
HZCM	830.14520 Hz/cm

^{13}CNMR (75MHz, $CDCl_3$) (6b)

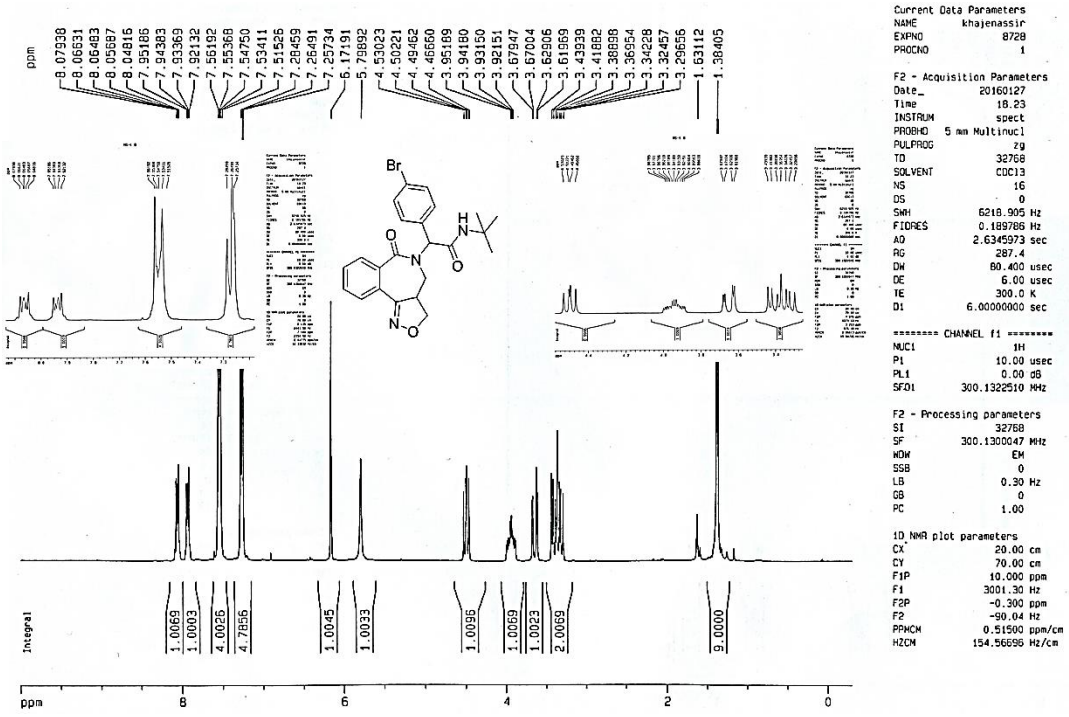


Meas. m/z	Formula	m/z	err [mDa]	err [ppm]	mSigma	rdb	N-Rule	e⁻ Conf
426.15854	C 27 H 18 N 6	426.15875	0.2	0.5	n.a.	22.0	ok	odd
	C 23 H 25 N 3 O 3 *35Cl	426.15790	-0.6	-1.5	n.a.	12.5	ok	even
	C 25 H 27 O 4 *35Cl	426.15924	0.7	1.6	n.a.	12.0	ok	odd
	C 26 H 22 N 2 O 4	426.15741	-1.1	-2.7	n.a.	17.0	ok	odd
	C 29 H 20 N 3 O	426.16009	1.5	3.6	n.a.	21.5	ok	even
	C 26 H 23 N 4 *35Cl	426.16058	2.0	4.8	n.a.	17.0	ok	odd
	C 25 H 26 N *35Cl *37Cl	426.15638	-2.2	-5.1	n.a.	12.5	ok	even
	C 24 H 20 N 5 O 3	426.15607	-2.5	-5.8	n.a.	17.5	ok	even
448.14036	C 23 H 24 N 3 Na O 3 *35Cl	448.13984	-0.5	-1.2	26.0	12.5	ok	even
	C 23 H 21 N 6 O 2 *35Cl	448.14090	0.5	1.2	32.2	16.0	ok	odd
	C 25 H 26 Na O 4 *35Cl	448.14118	0.8	1.8	32.7	12.0	ok	odd
	C 25 H 22 N O 7	448.13908	0.7	1.5	48.6	15.5	ok	even
	C 24 H 16 N 8 O 2	448.13907	0.7	1.5	54.8	21.0	ok	odd
	C 24 H 19 N 5 Na O 3	448.13801	-0.4	-0.9	57.9	17.5	ok	even
	C 25 H 26 O 3 *35Cl *37Cl	448.13805	-0.4	-0.8	61.0	11.0	ok	odd
	C 23 H 20 N 4 O 6	448.13774	0.6	1.3	493.7	16.0	ok	odd
	C 25 H 23 N 3 O 3 *35Cl	448.14225	-0.2	-0.5	560.2	15.5	ok	even

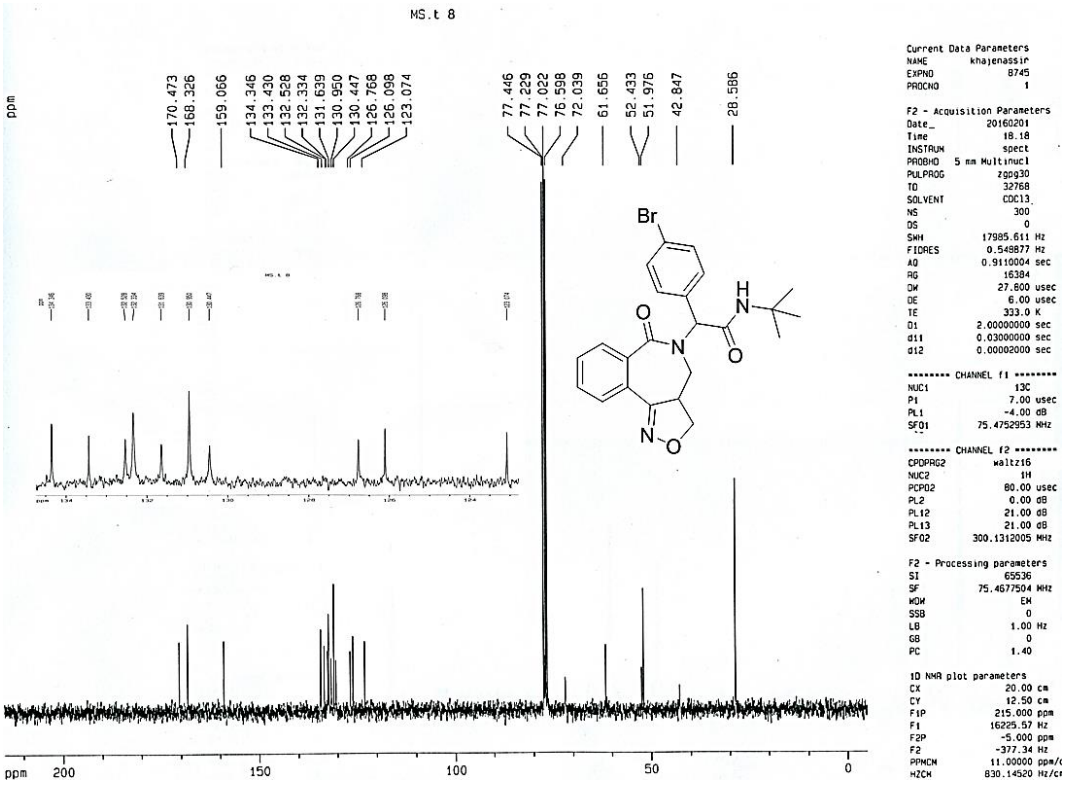
ESI-HRMS (6b)



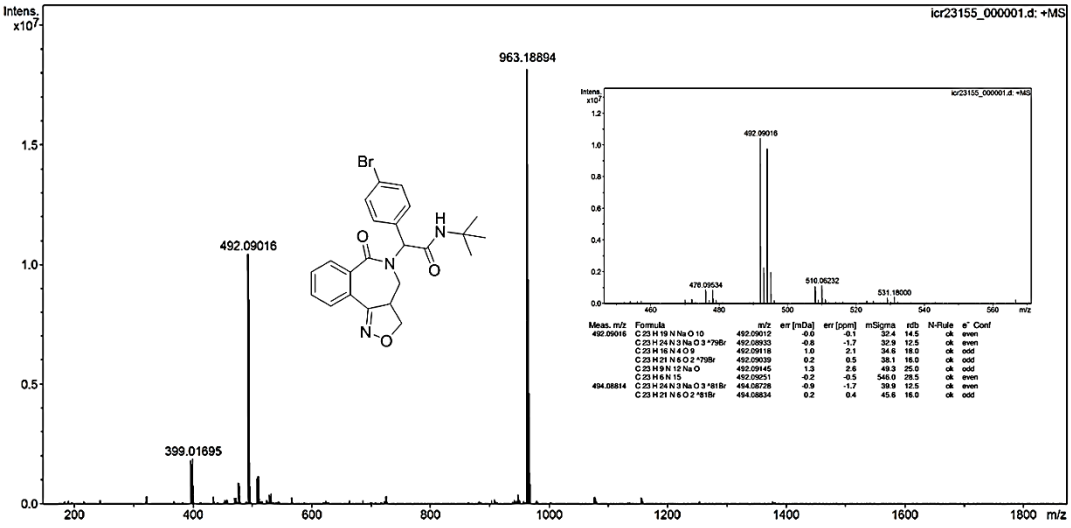
IR (KBr) (6c)



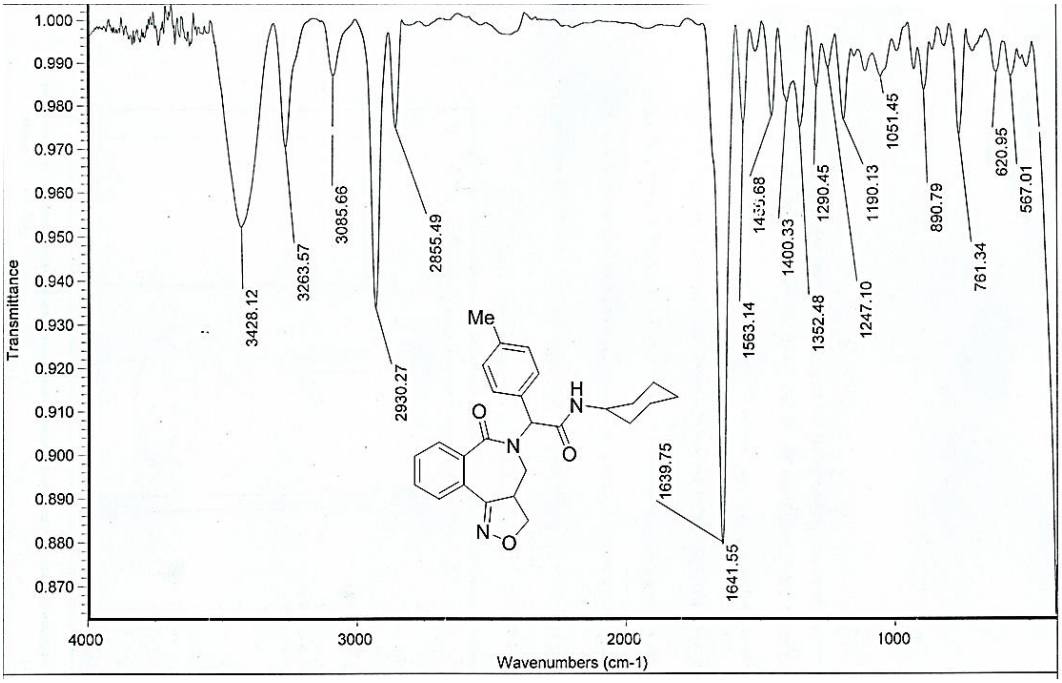
^{1}HNMR (300MHz, $CDCl_3$) (6c)



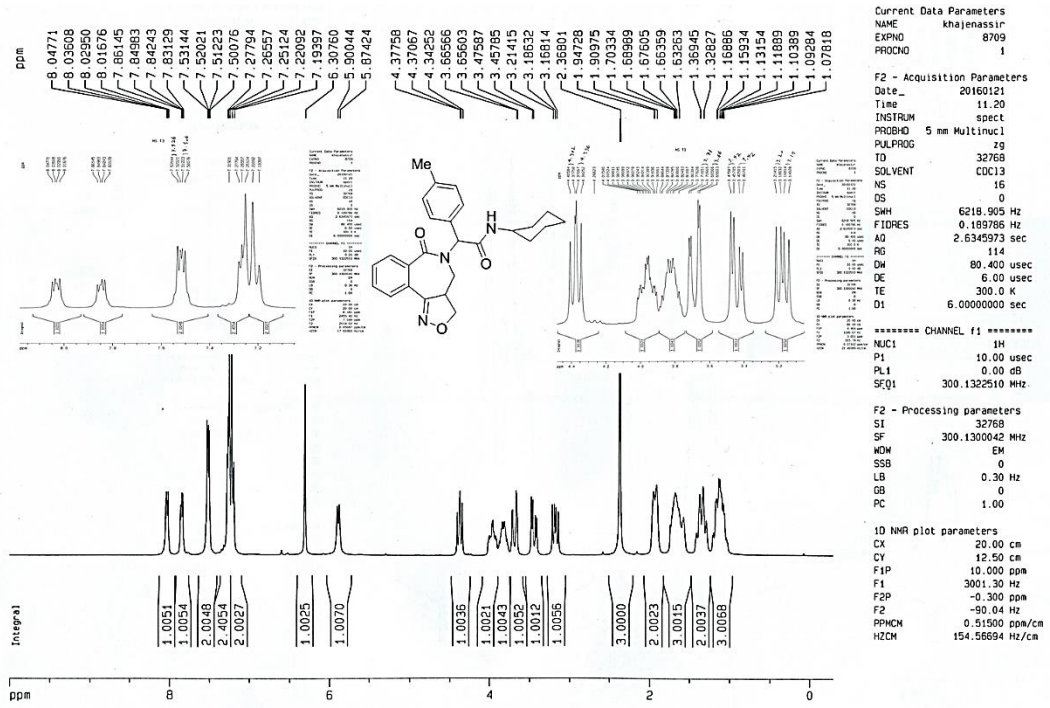
^{13}CNMR (75MHz, $CDCl_3$) (6c)



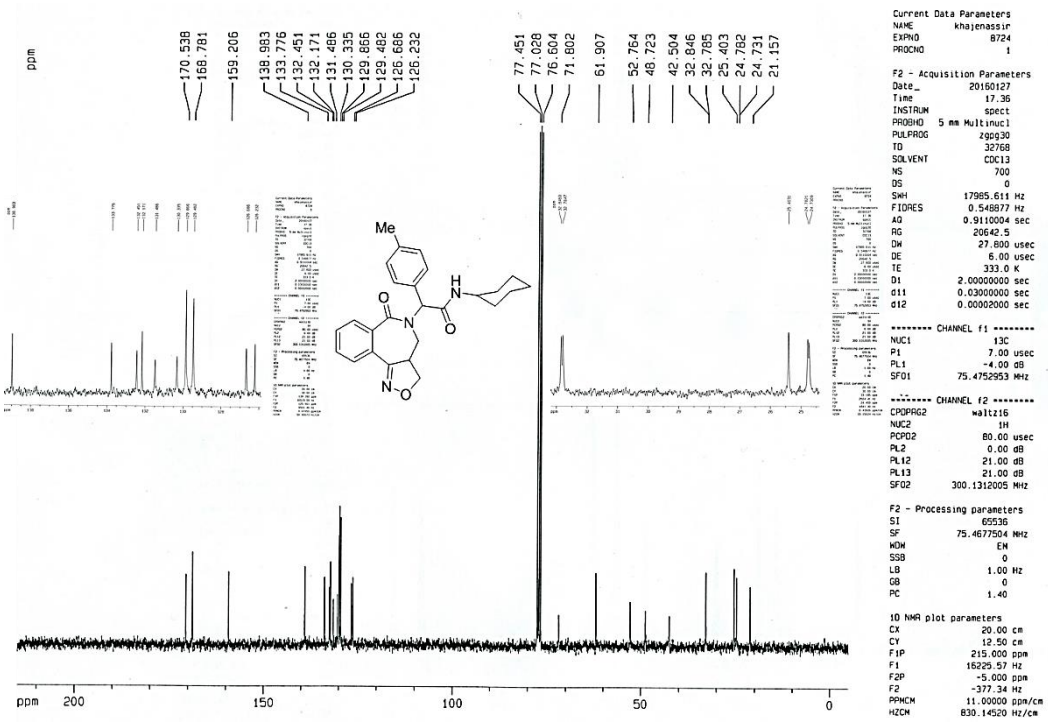
ESI-HRMS (6c)



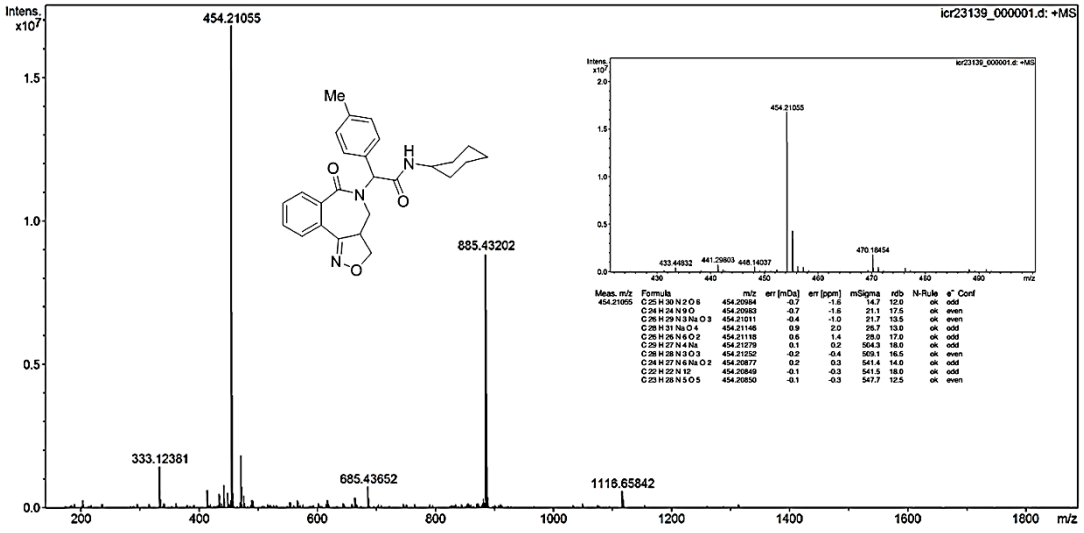
IR (KBr) (6d)



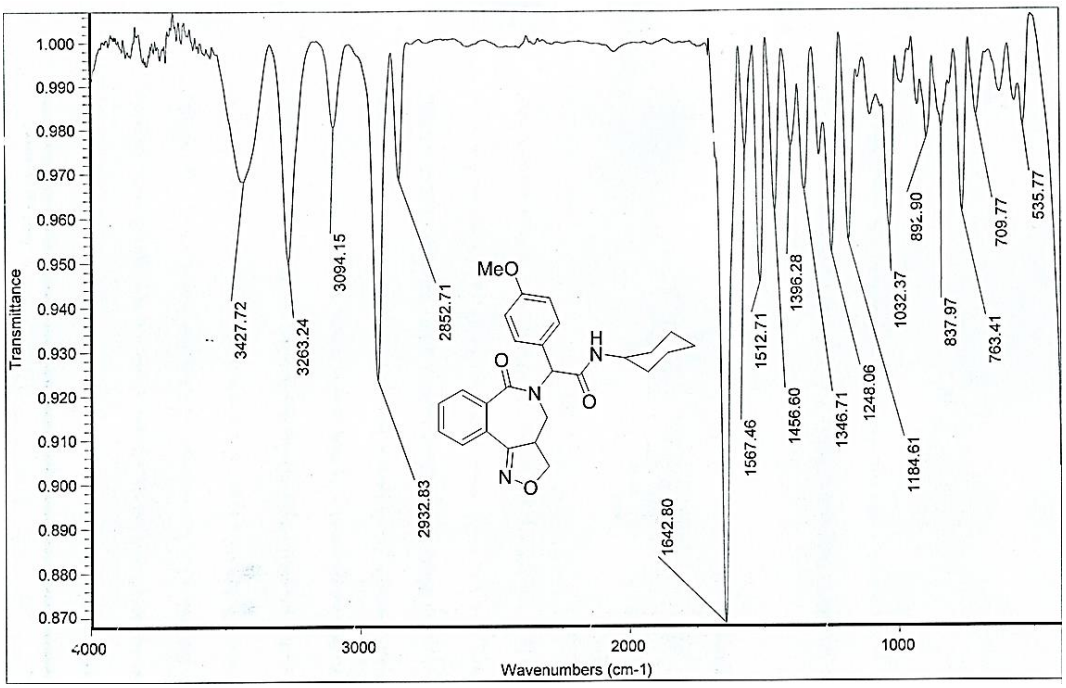
^{1}HNMR (300MHz, $CDCl_3$) (6d)



^{13}CNMR (75MHz, $CDCl_3$) (6d)



ESI-HRMS (6d)



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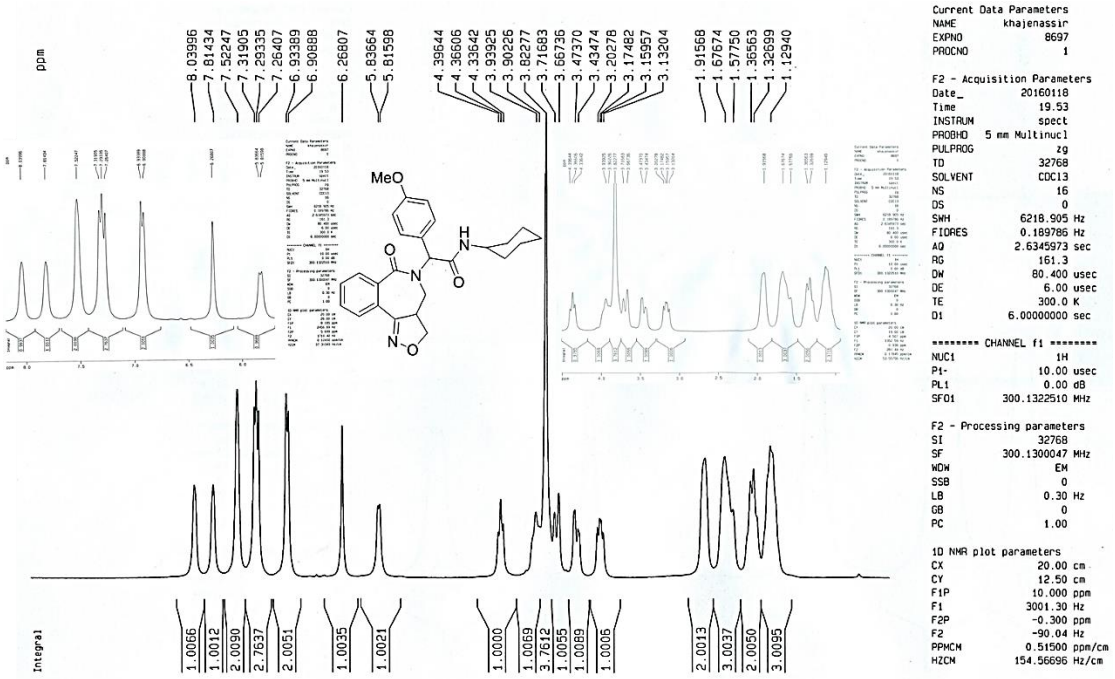
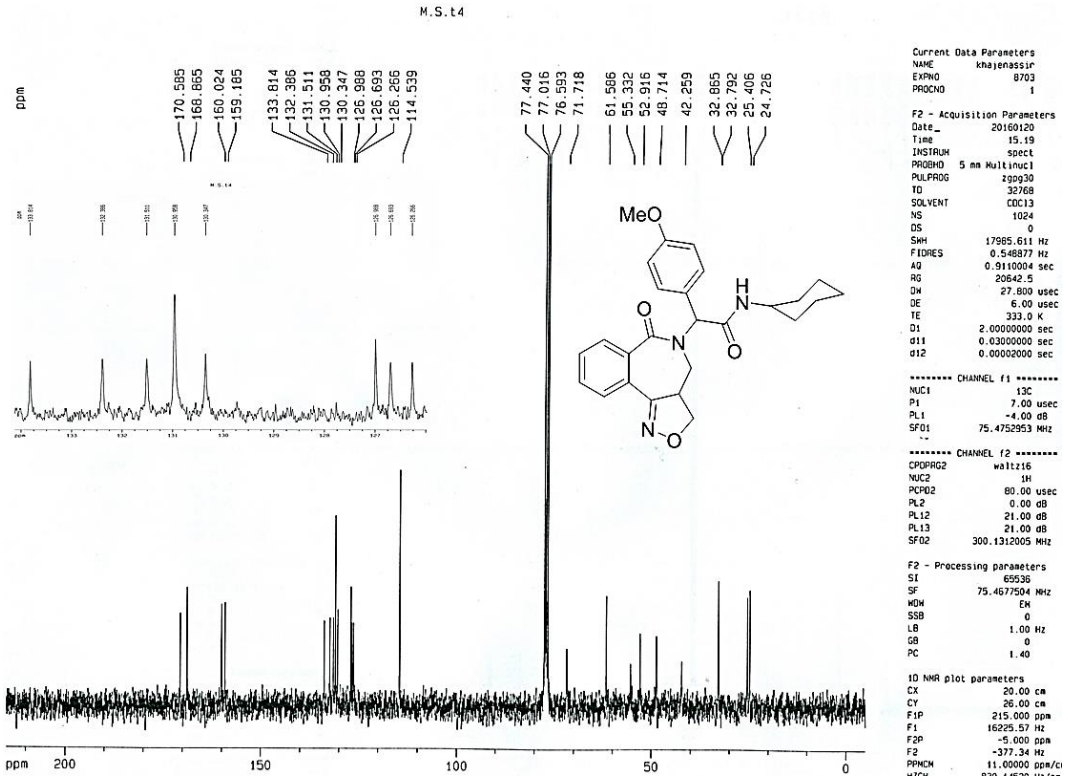
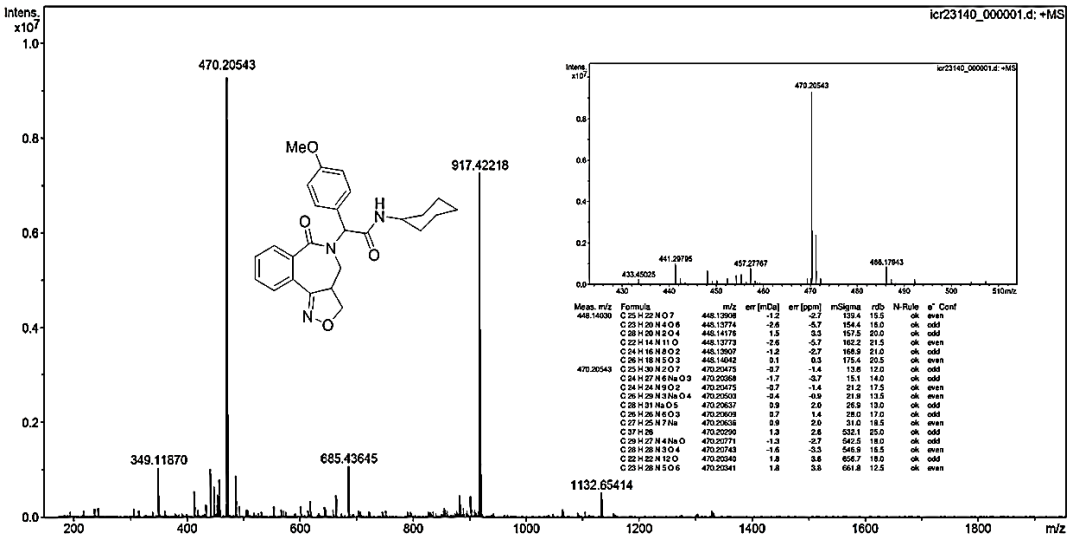


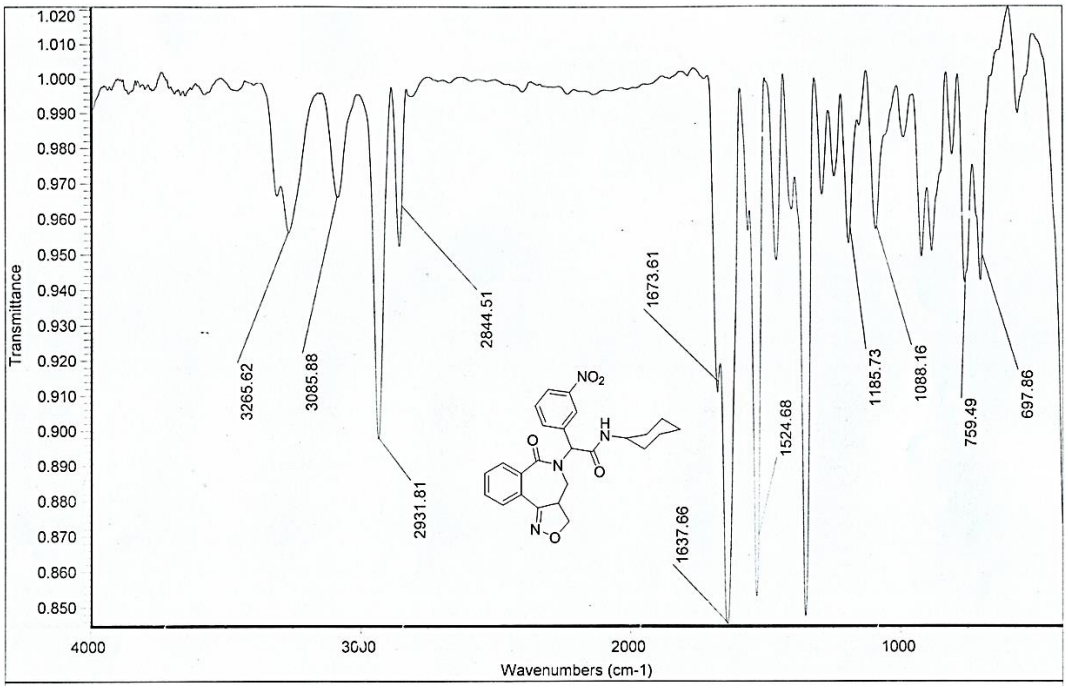
^{1}HNMR (300MHz, $CDCl_3$) (6e)



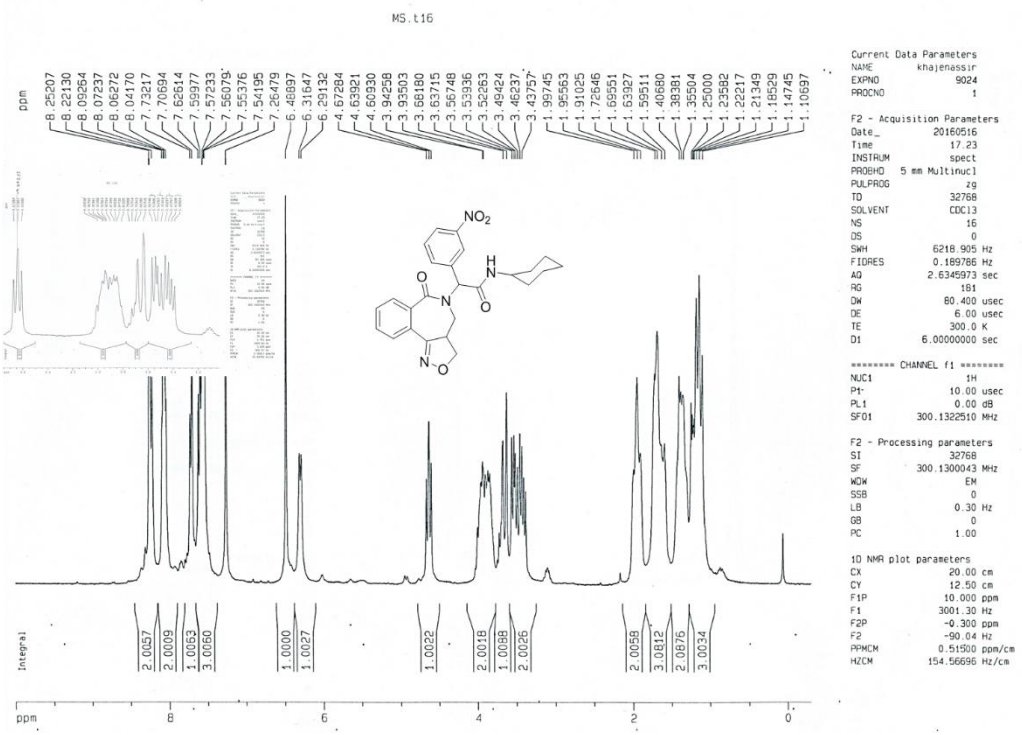
^{13}CNMR (75MHz, $CDCl_3$) (6e)



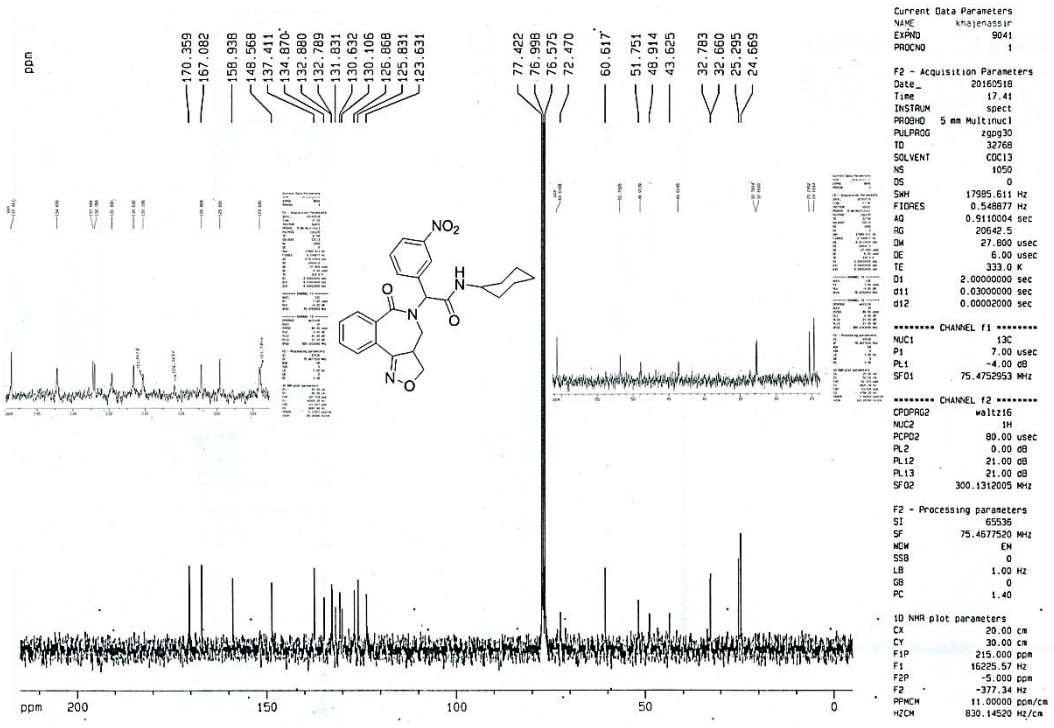
ESI-HRMS (6e)



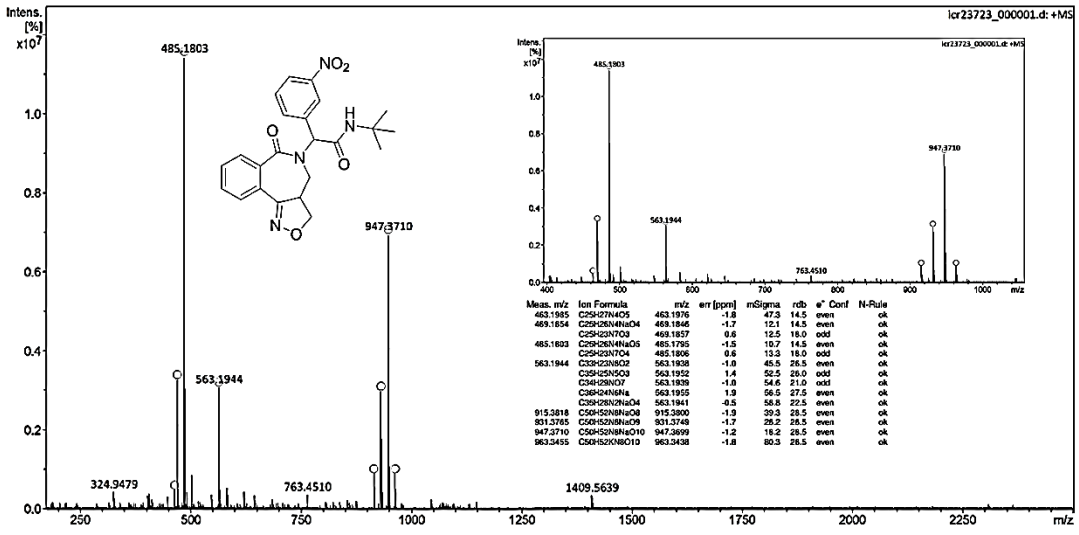
IR (KBr) (6f)



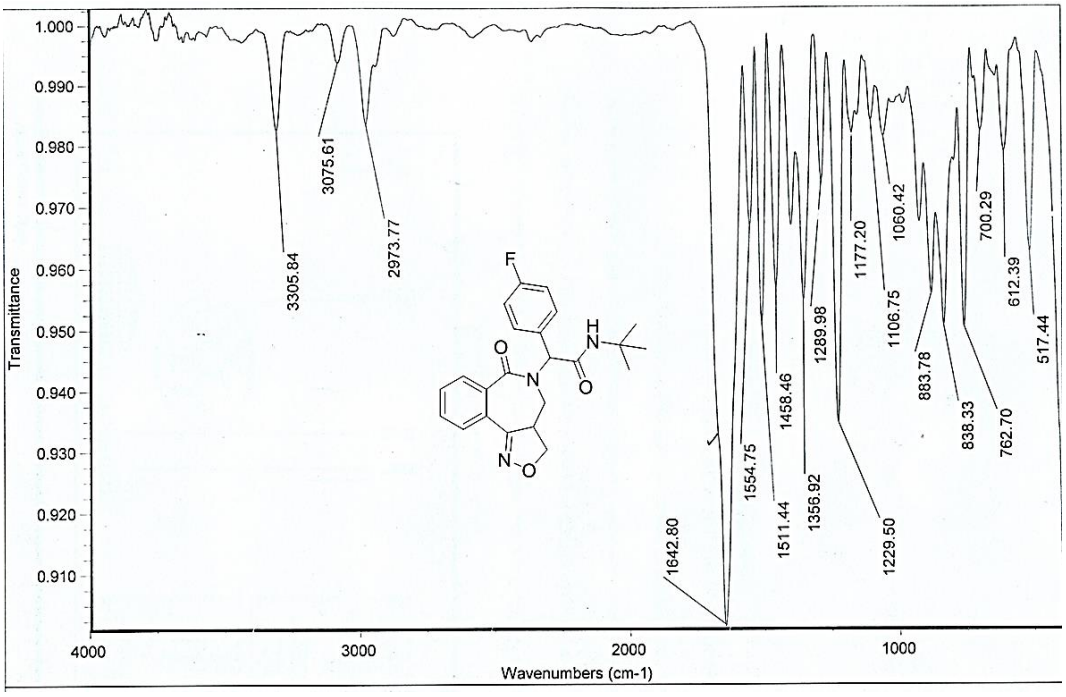
^{1}HNMR (300MHz, $CDCl_3$) (6f)



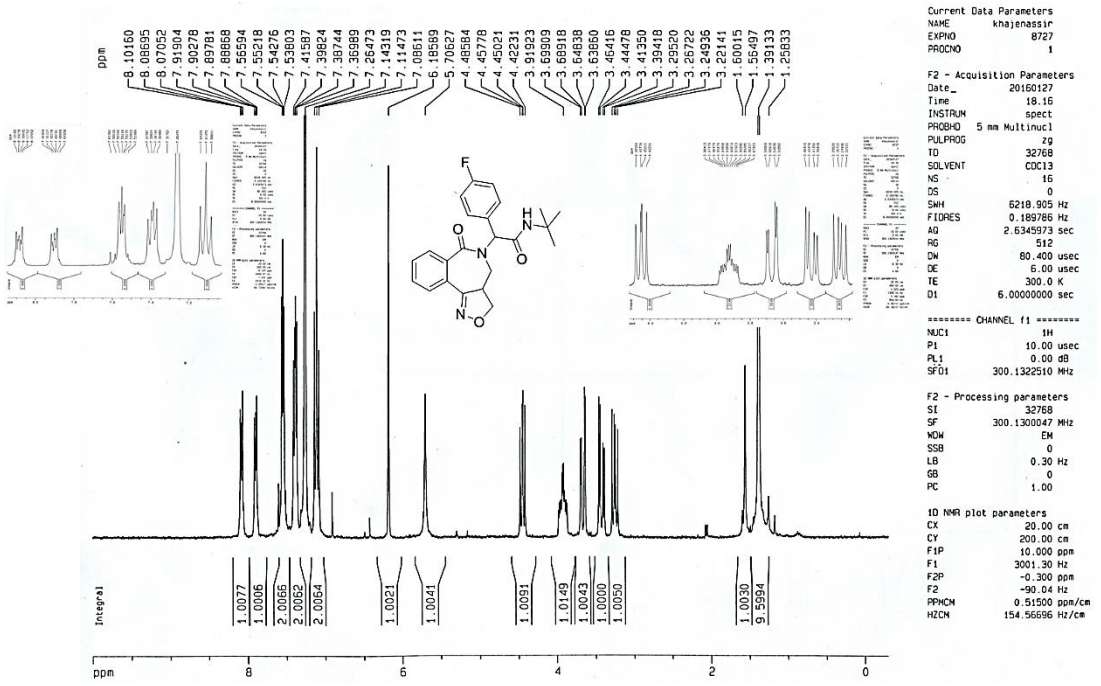
^{13}CNMR (75MHz, $CDCl_3$) (6f)



ESI-HRMS (6f)



IR (KBr) (6g)



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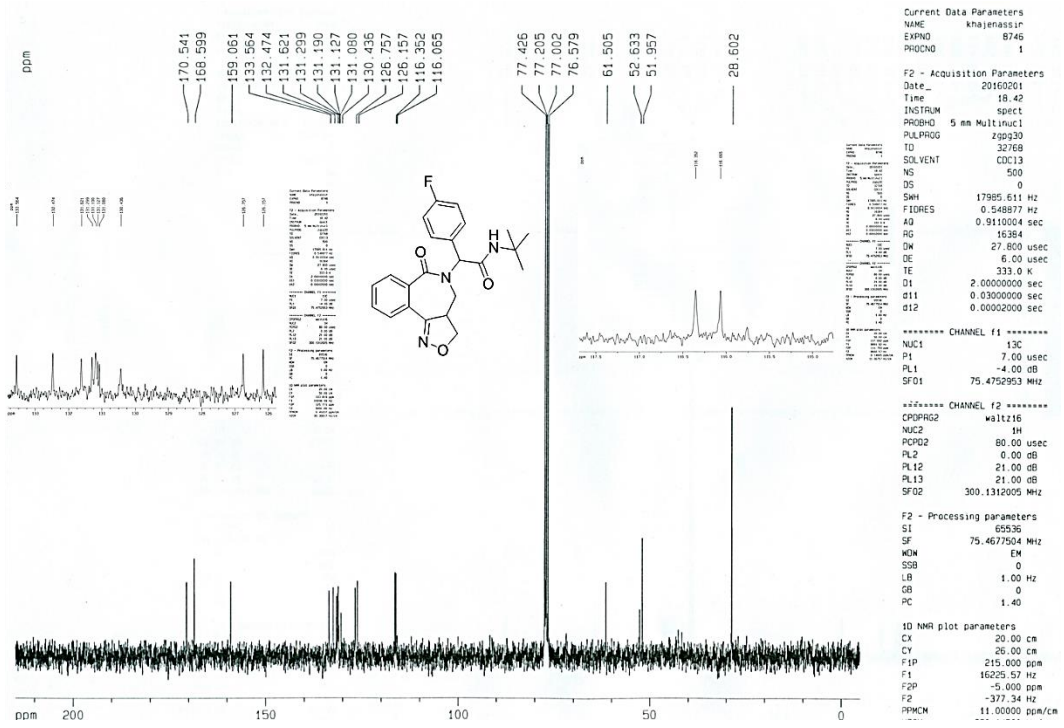
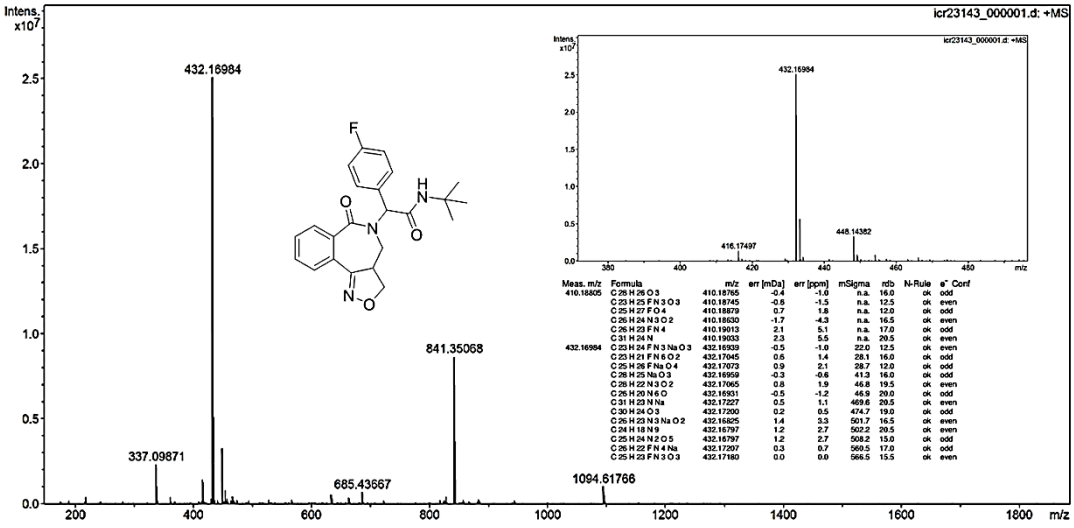
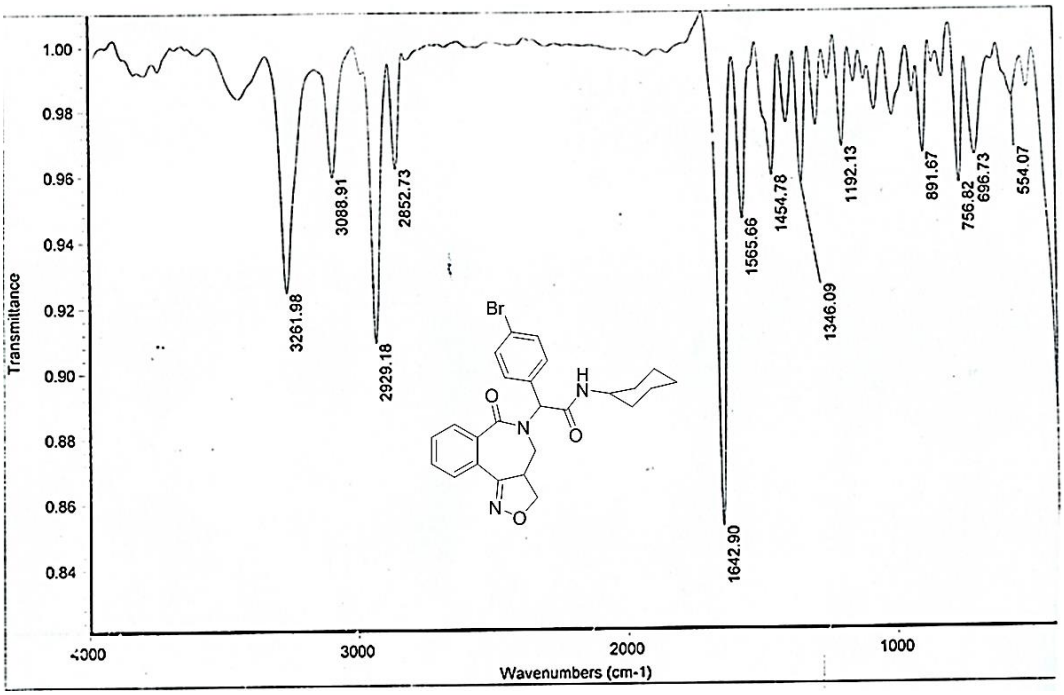


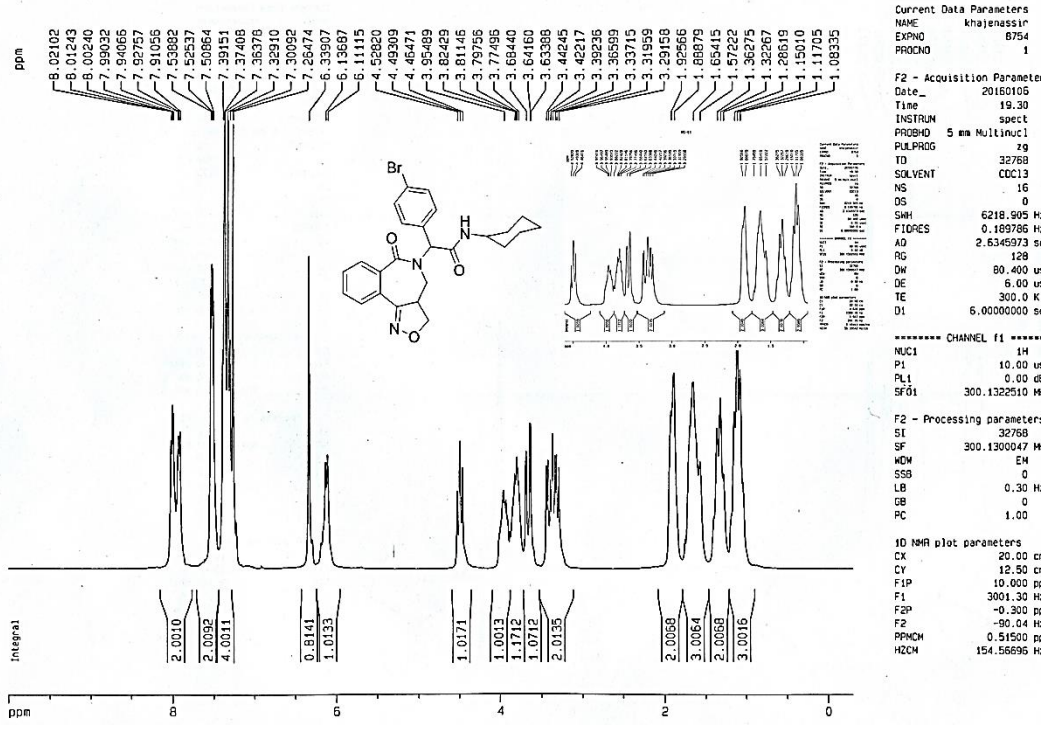
^{13}CNMR (75MHz, $CDCl_3$) (6g)



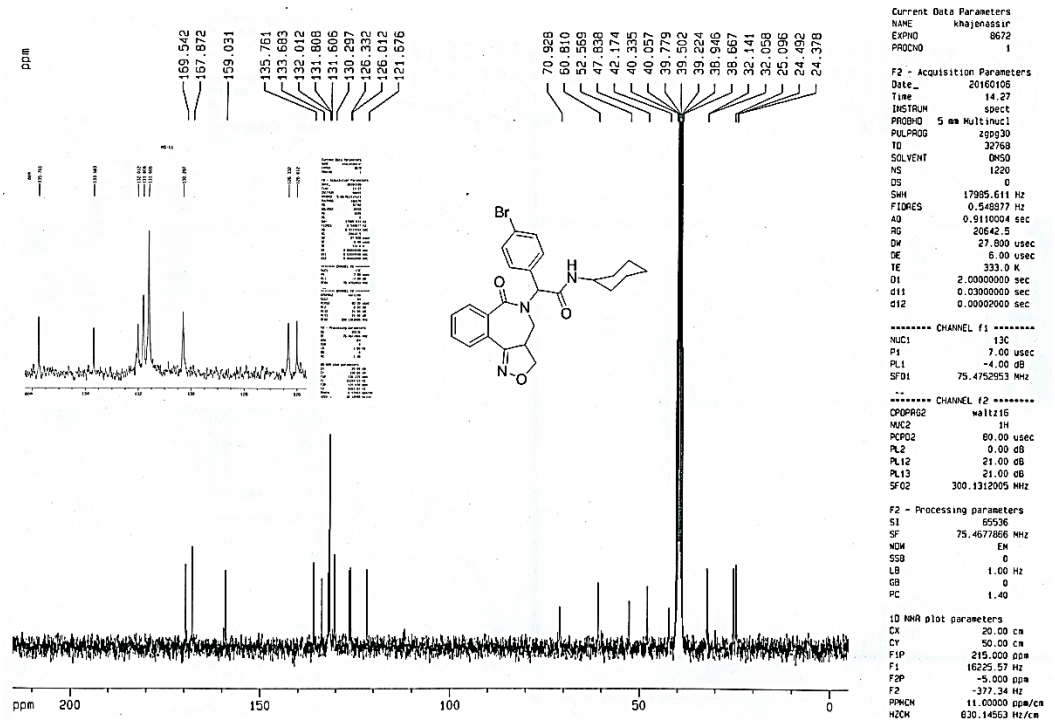
ESI-HRMS (6g)



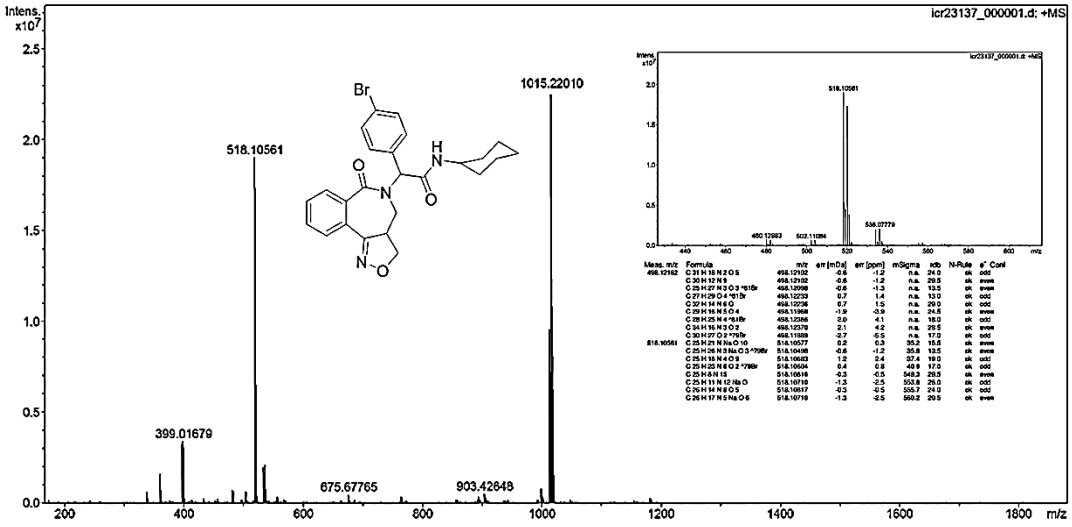
IR (KBr) (6h)



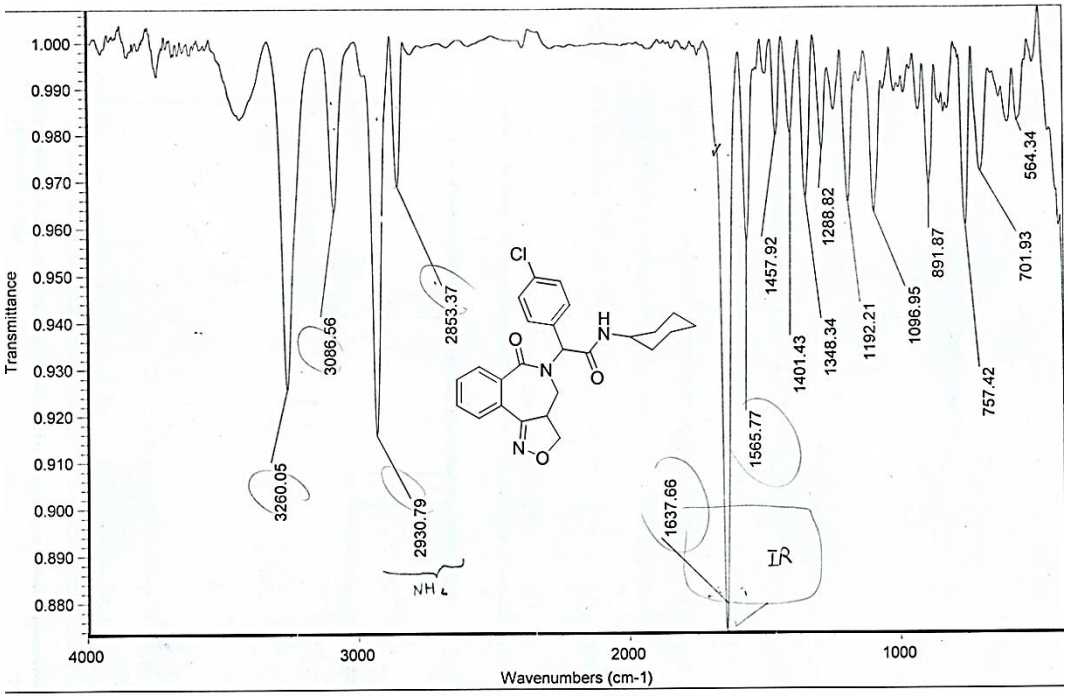
^{1}HNMR (300MHz, $CDCl_3$) (6h)



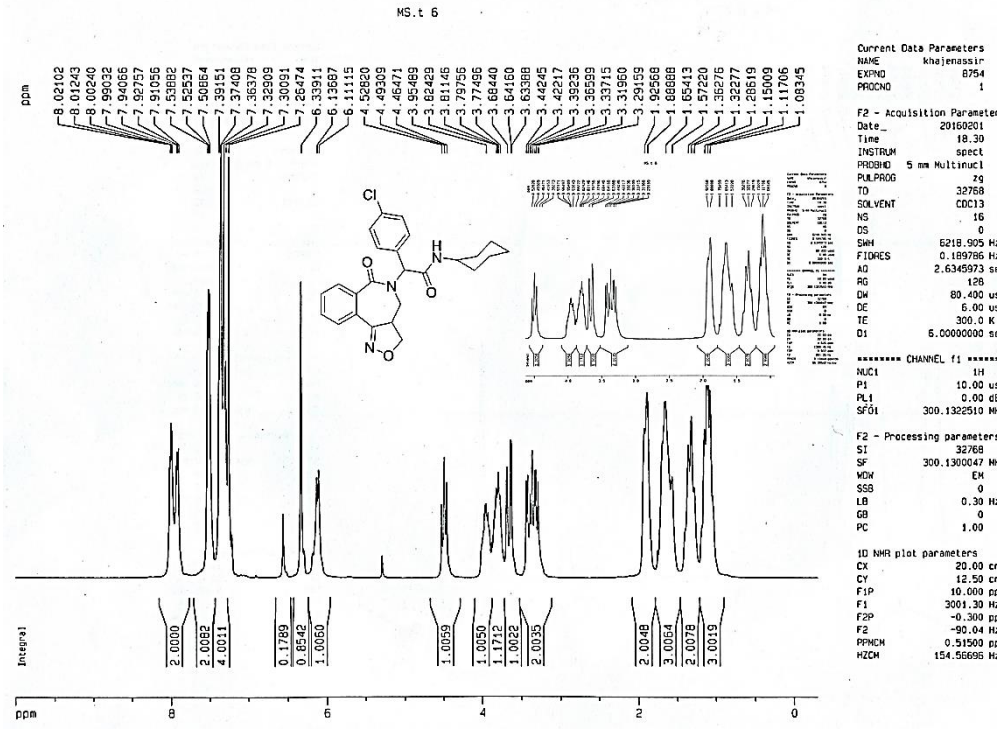
^{13}CNMR (75MHz, $CDCl_3$) (6h)



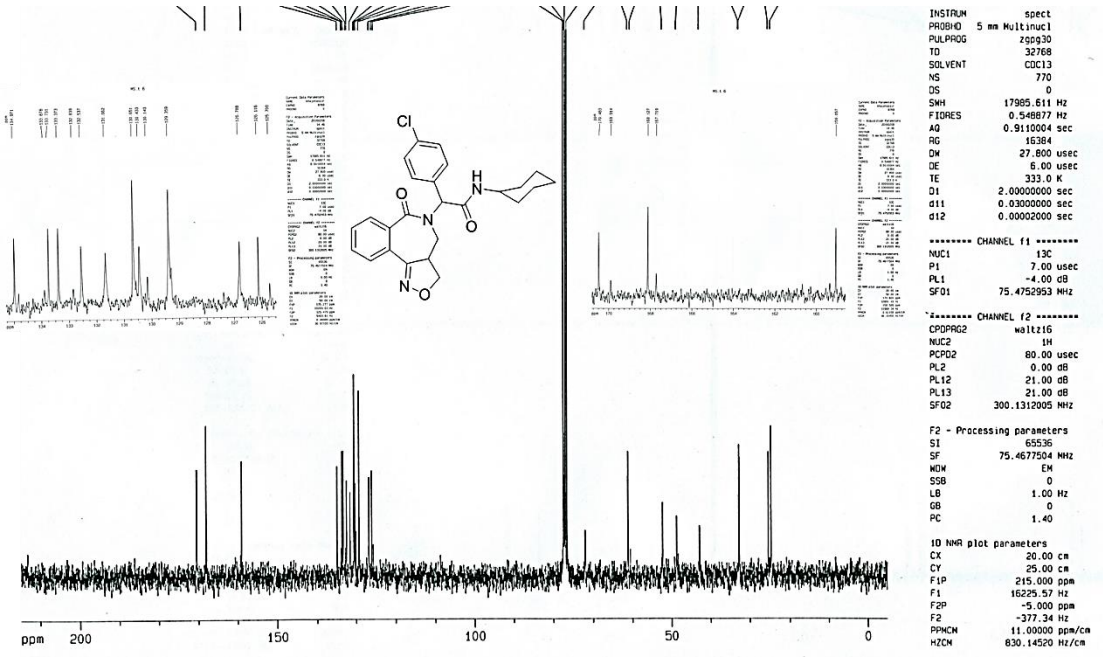
ESI-HRMS (6h)



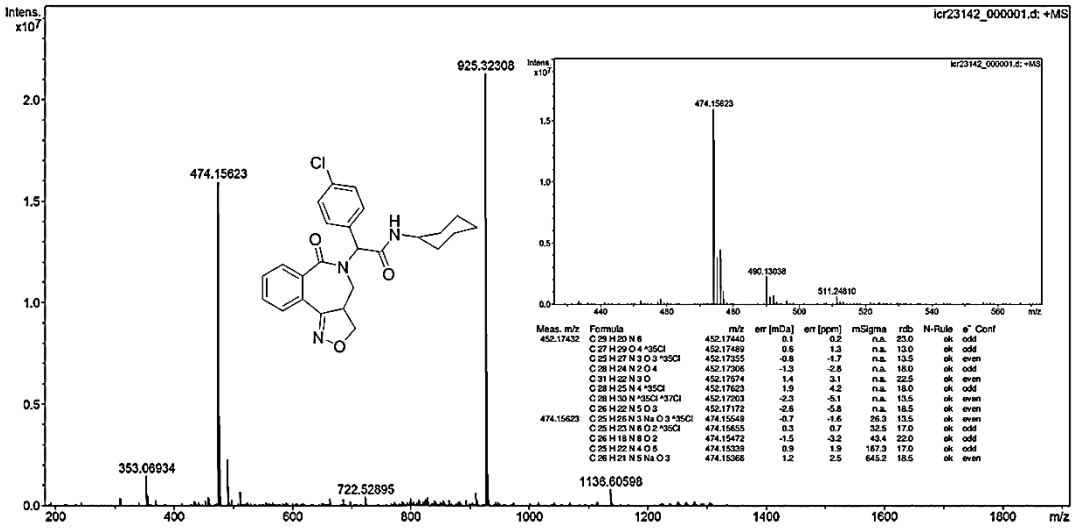
IR (KBr) (6i)



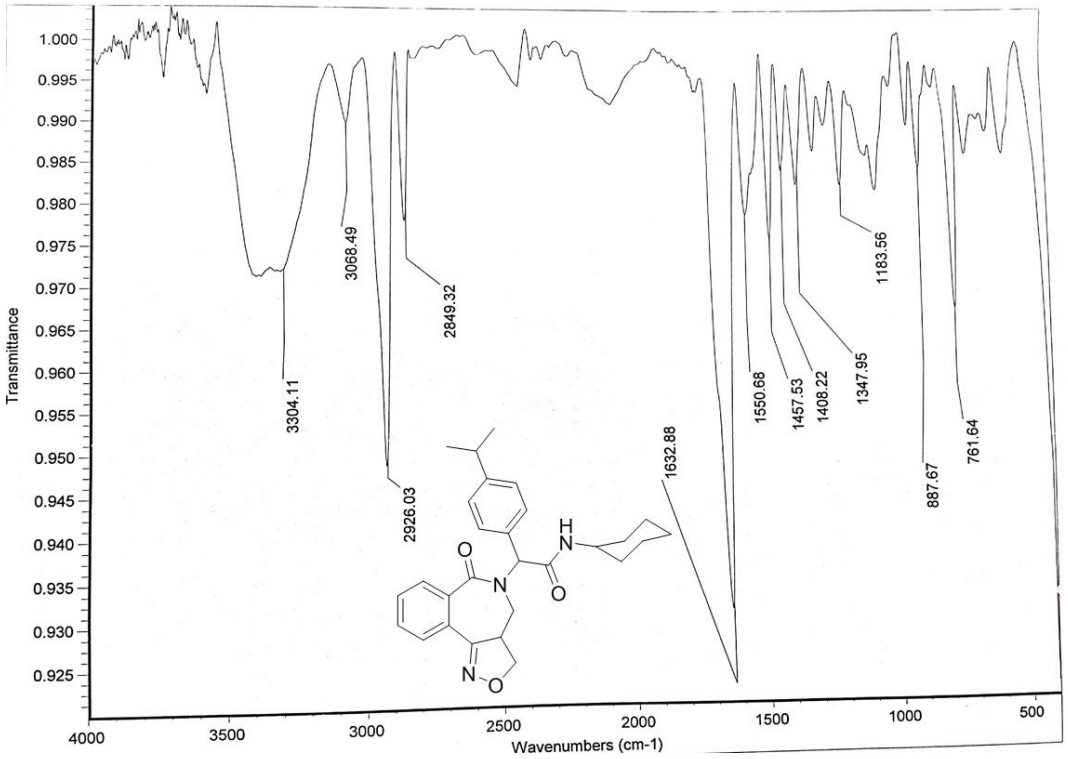
^{1}HNMR (300MHz, $CDCl_3$) (6i)



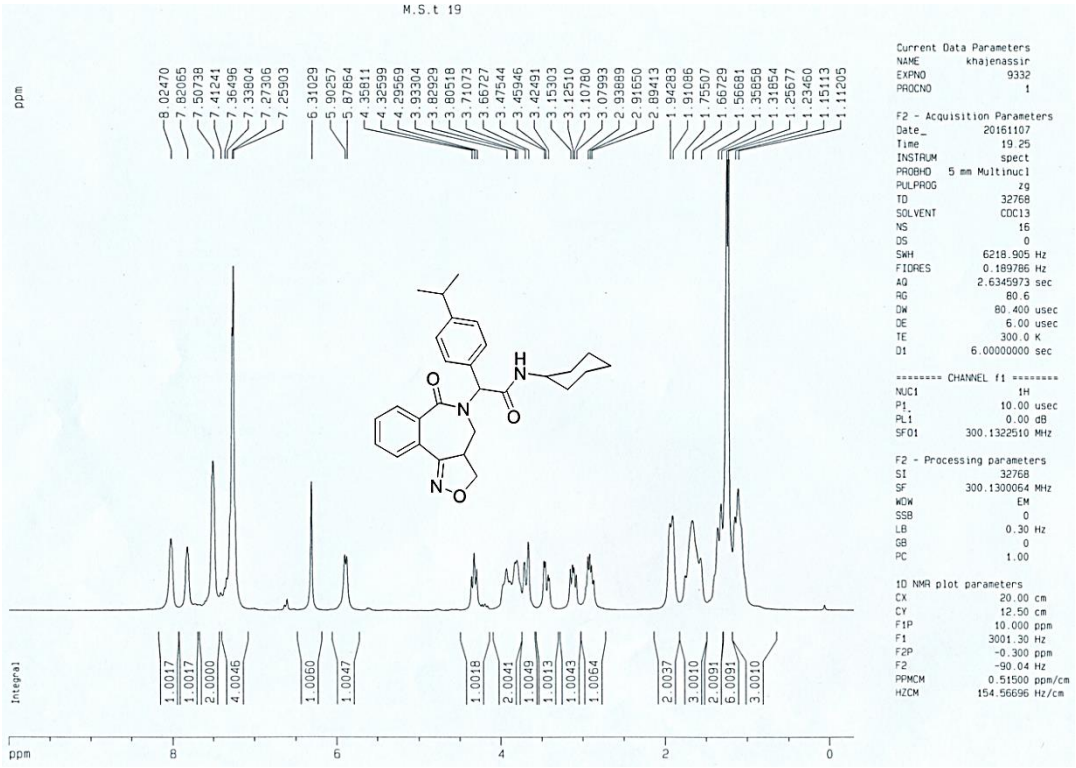
^{13}CNMR (75MHz, $CDCl_3$) (6i)



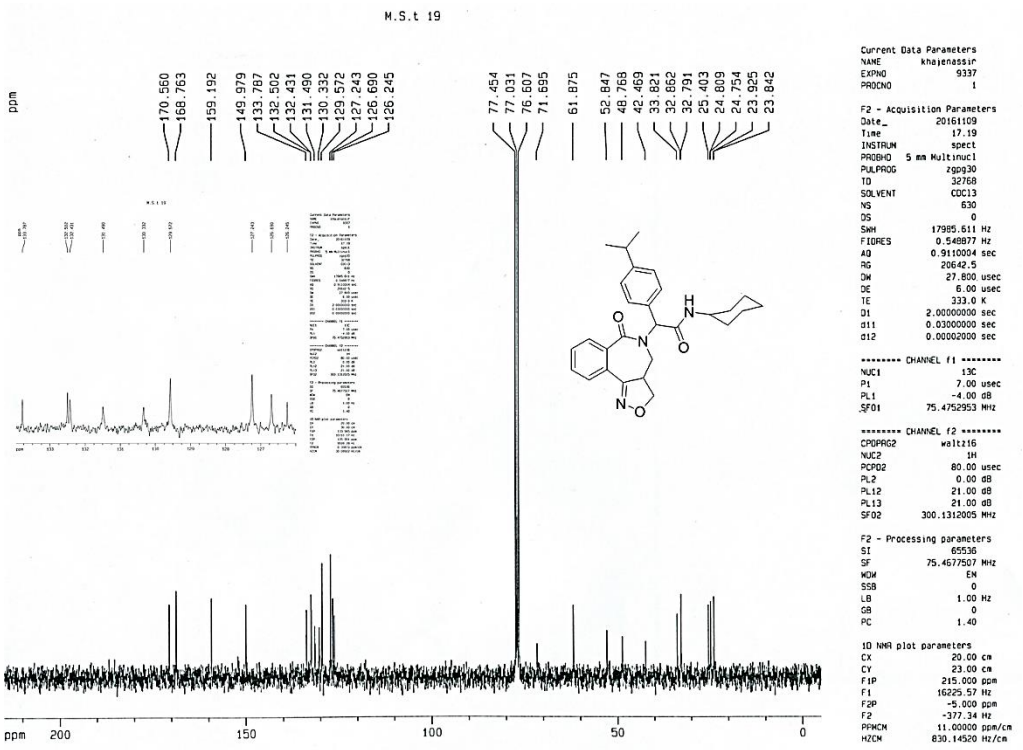
ESI-HRMS (6i)



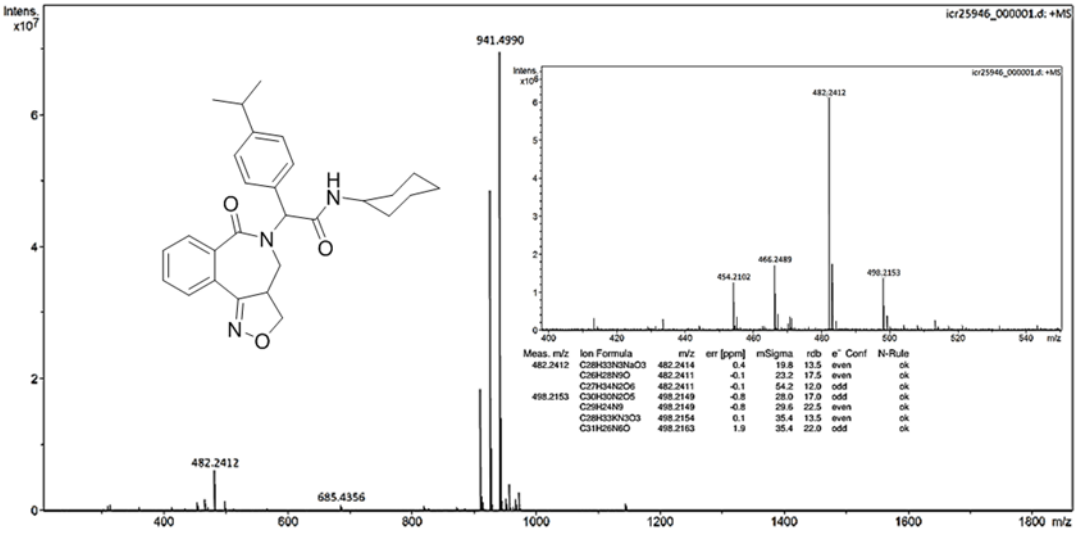
IR (KBr) (6j)



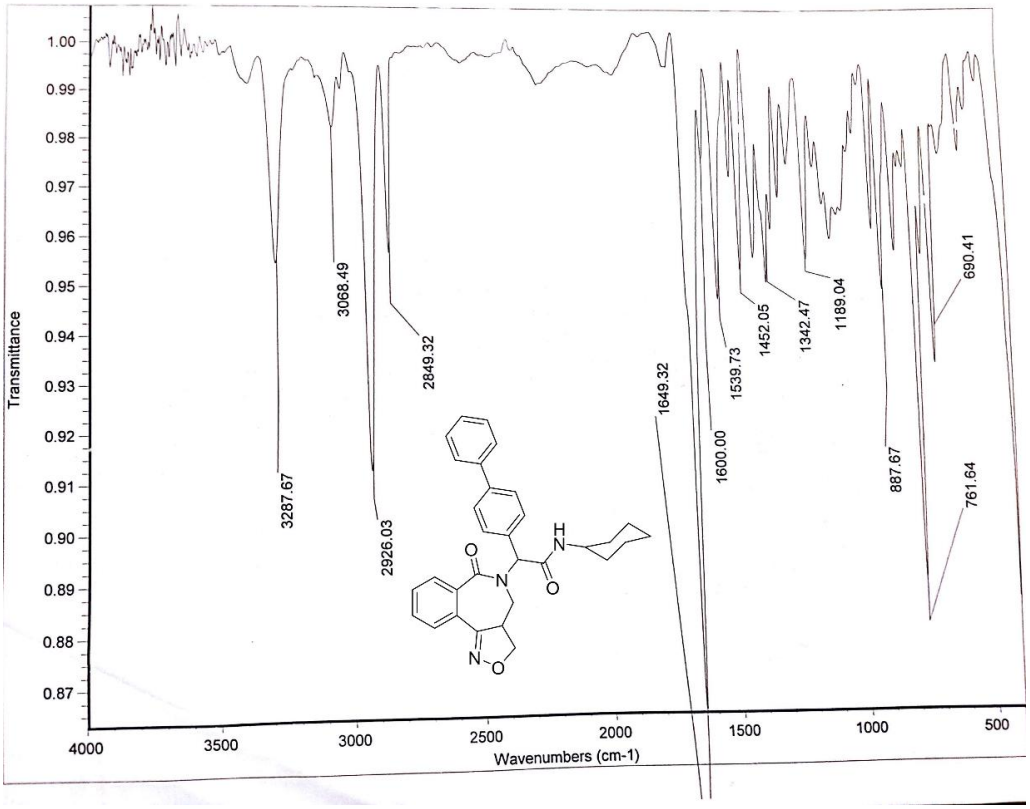
^{1}HNMR (300MHz, $CDCl_3$) (6j)



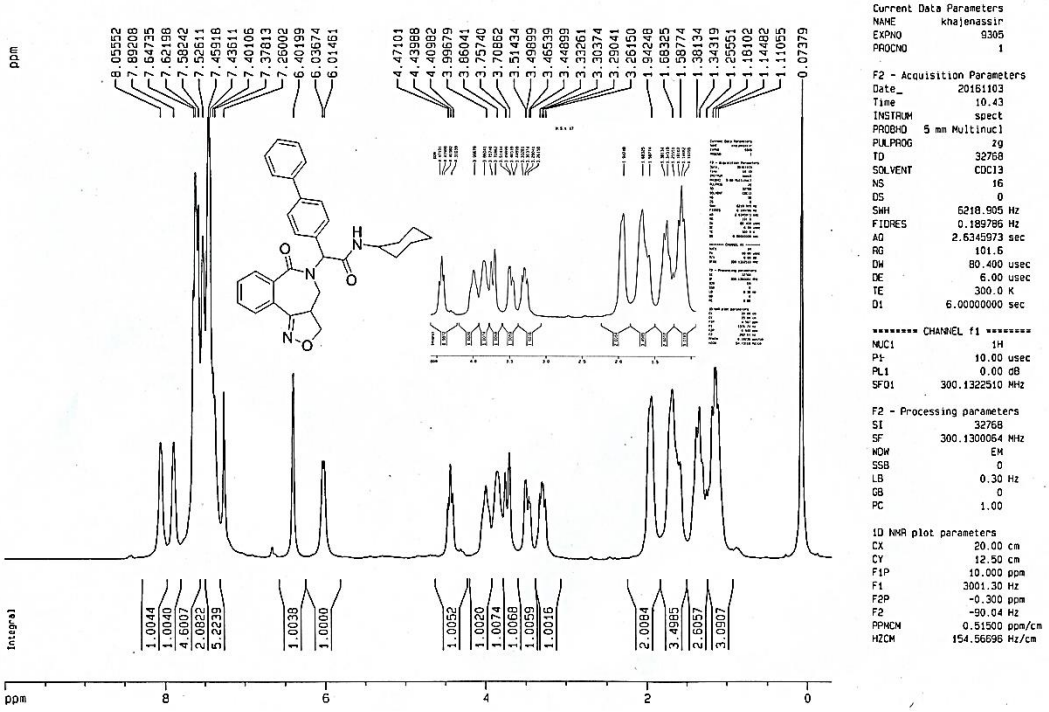
^{13}CNMR (75MHz, $CDCl_3$) (6j)



ESI-HRMS (6j)



IR (KBr) (6k)

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EXPNO               9305
PROCNO                 1

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Time               10.43
INSTRUM            spect
PROBHD  5 mm Multinucl
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           6218.905 Hz
FIDRES        0.189786 Hz
AQ            2.6345973 sec
RG                101.6
DW              80.400 usec
DE               6.00 usec
TE              300.0 K
D1            6.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1              10.00 usec
PL1              0.00 dB
SFO1      300.1322510 MHz

F2 - Processing parameters
SI                32768
SF        300.1300064 MHz
WDW                  EM
SSB                   0
LB              0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               20.00 cm
CY               12.50 cm
F1P             10.000 ppm
F1             3001.30 Hz
F2P             -0.300 ppm
F2             -90.04 Hz
PPMCM          0.51500 ppm/cm
HZCM         154.56696 Hz/cm
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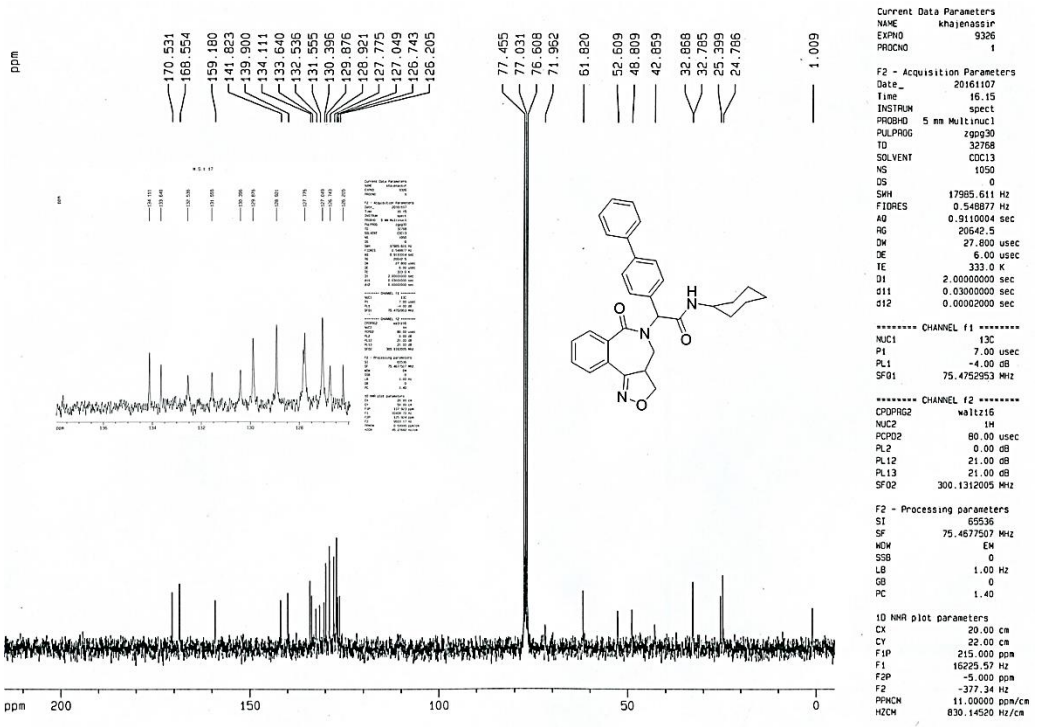
^{1}HNMR (300MHz, $CDCl_3$) (6k)



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NAME khajenassir
EXPNO 9326
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1050
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 20642.5
DW 27.800 usec
DE 6.00 usec
TE 333.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

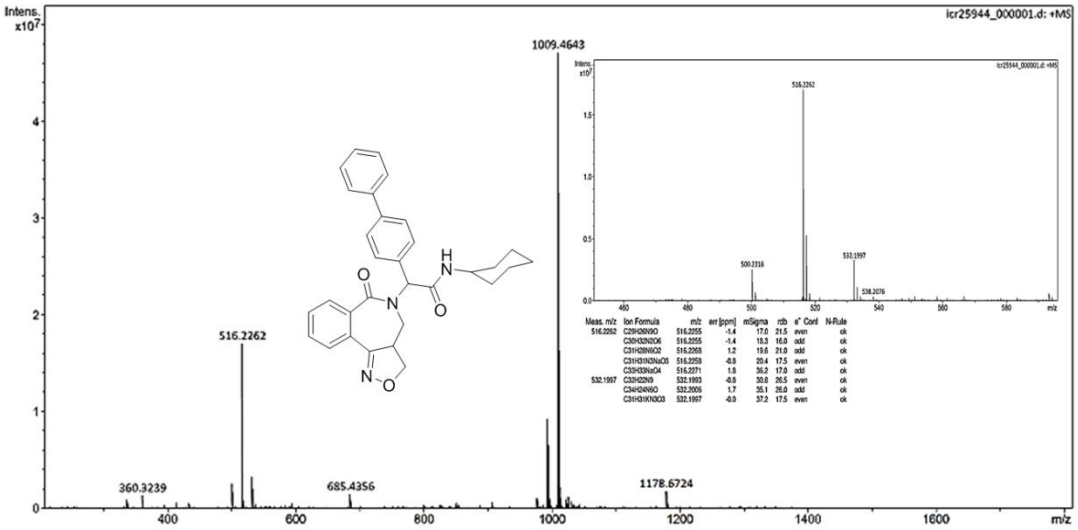
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SF01 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 300.1312005 MHz

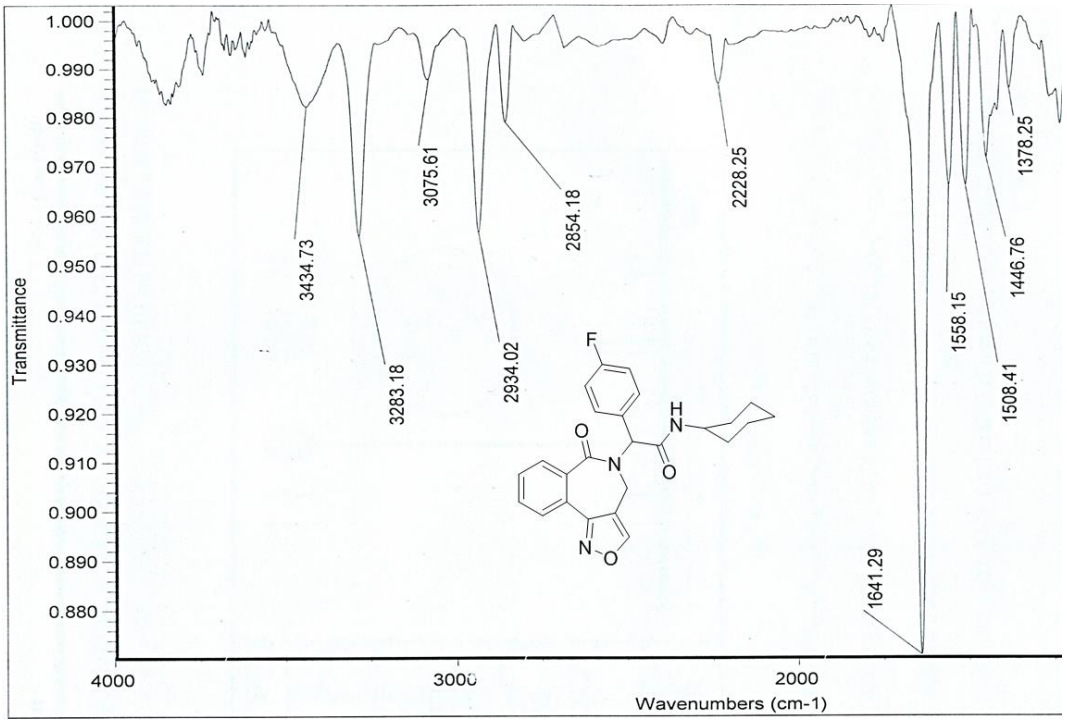
F2 - Processing parameters
SI 65536
SF 75.4677507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 22.00 cm
F1P 215.000 ppm
F1 16225.57 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

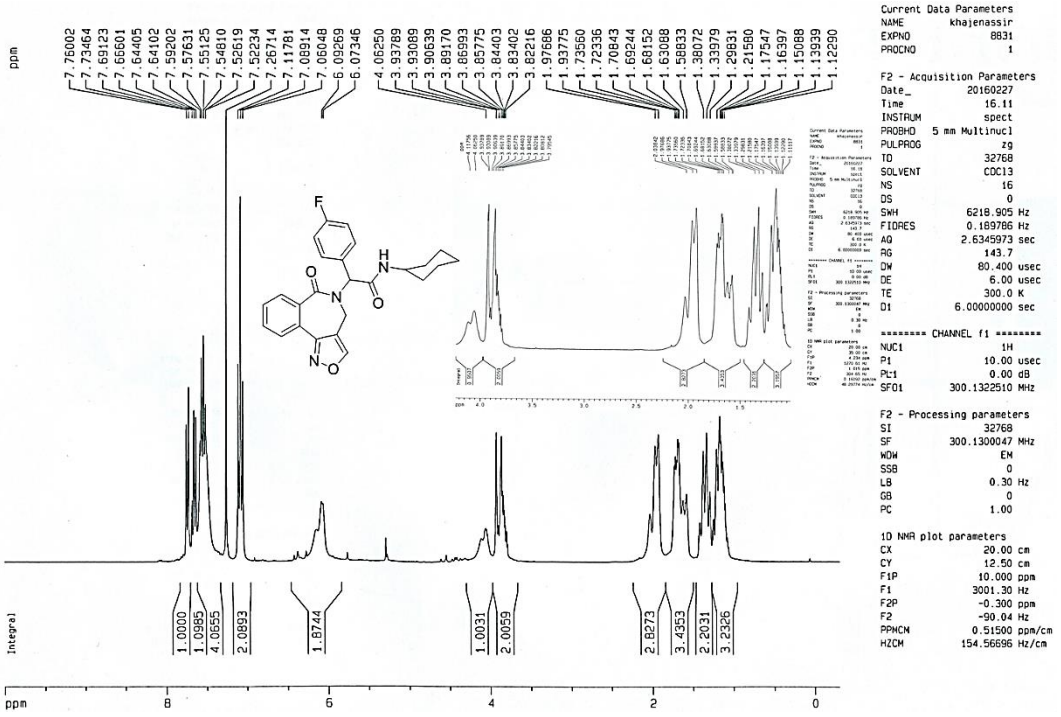
^{13}CNMR (75MHz, $CDCl_3$) (6k)



ESI-HRMS (6k)



IR (KBr) (9a)



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NAME khajenassir
EXPNO 8831
PROCNO 1

F2 - Acquisition Parameters
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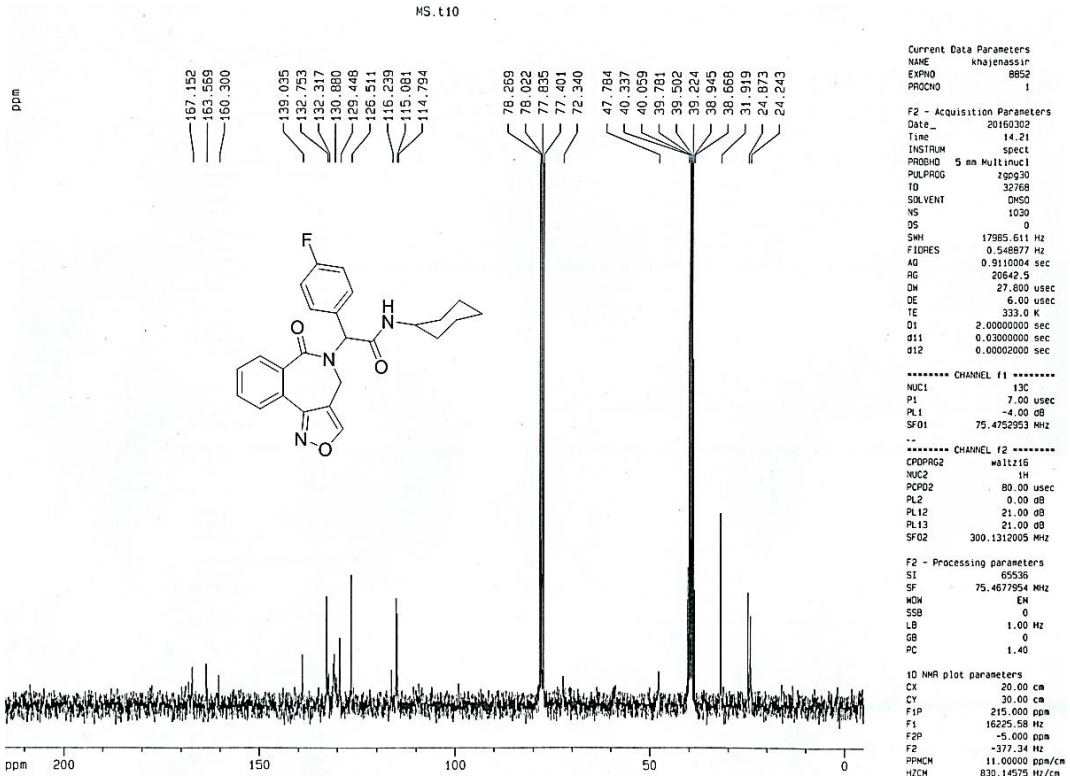
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6218.905 Hz
FIDRES 0.189786 Hz
AQ 2.6345973 sec
RG 143.7
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1322510 MHz

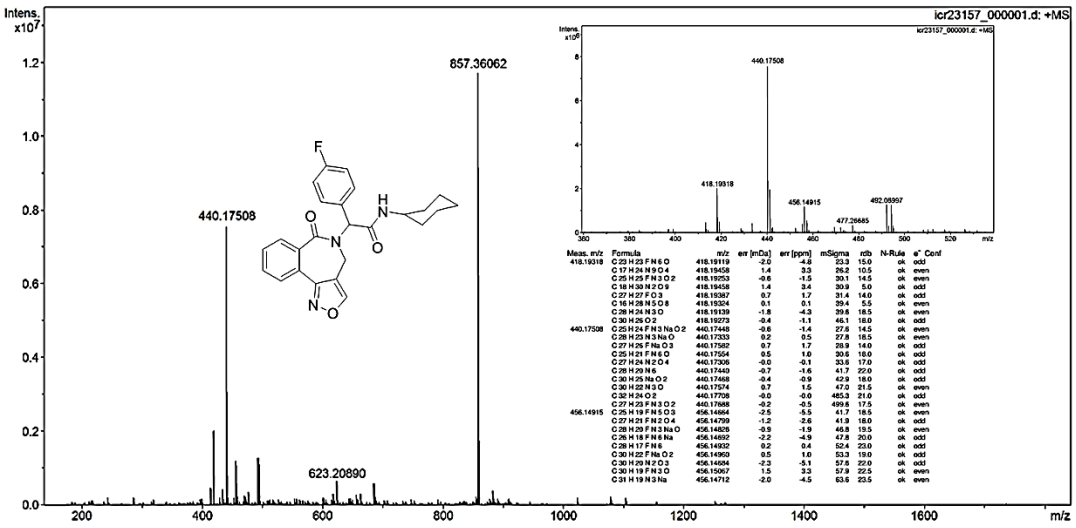
F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.51500 ppm/cm
HZCM 154.56696 Hz/cm

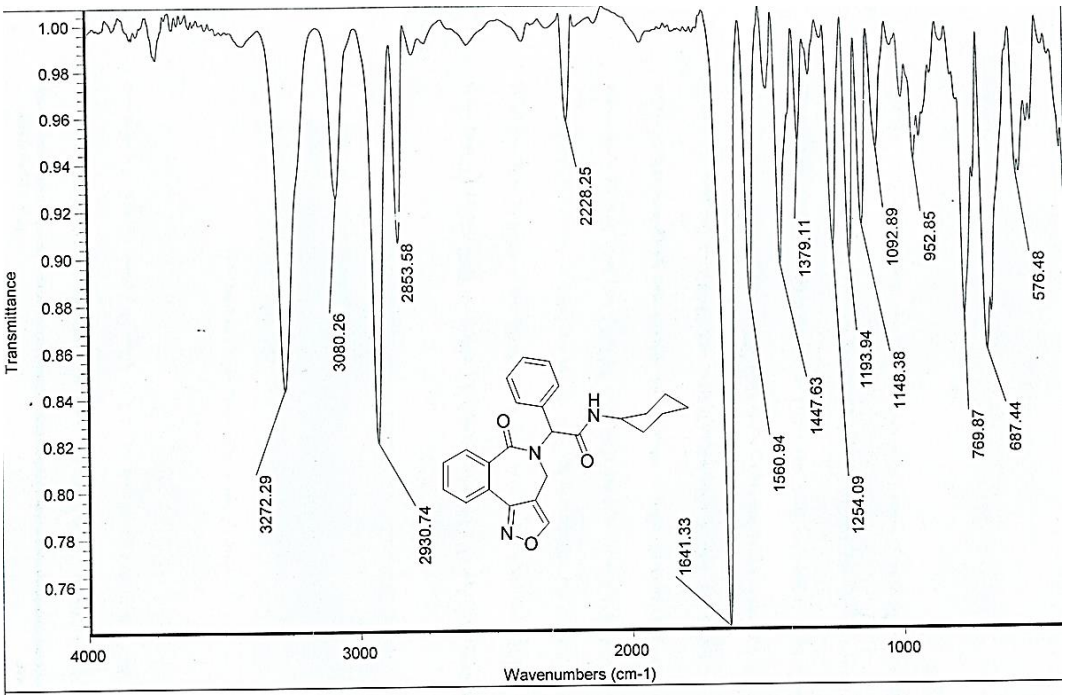
^{1}HNMR (300MHz, $CDCl_3$) (9a)



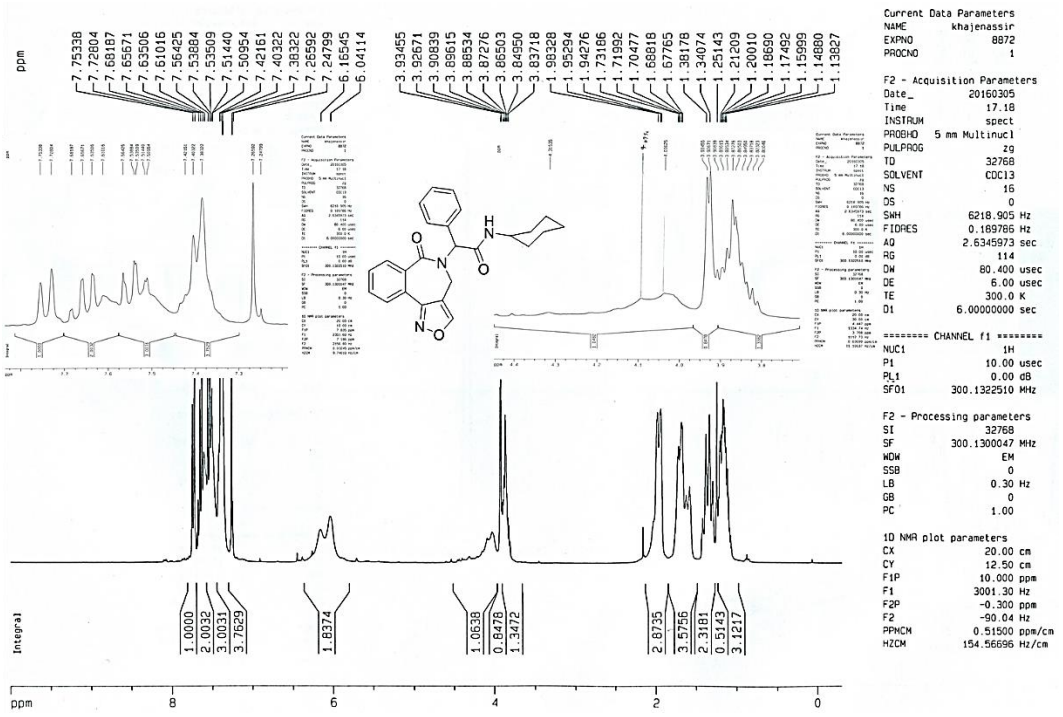
^{13}CNMR (75MHz, $CDCl_3$) (9a)



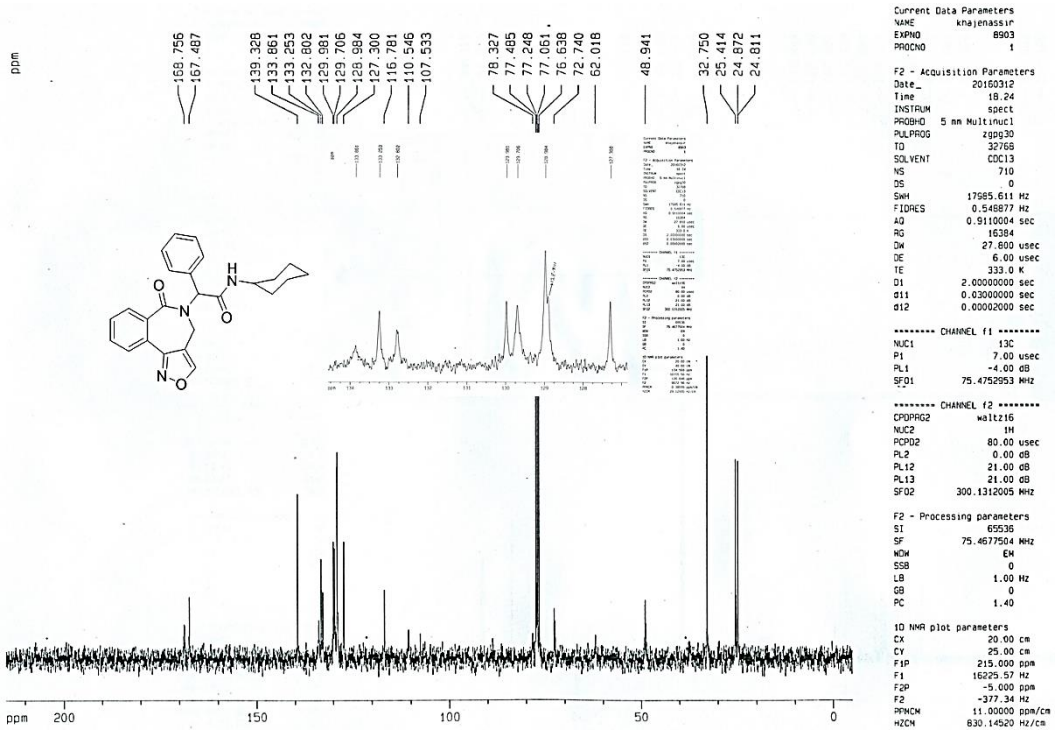
ESI-HRMS (9a)



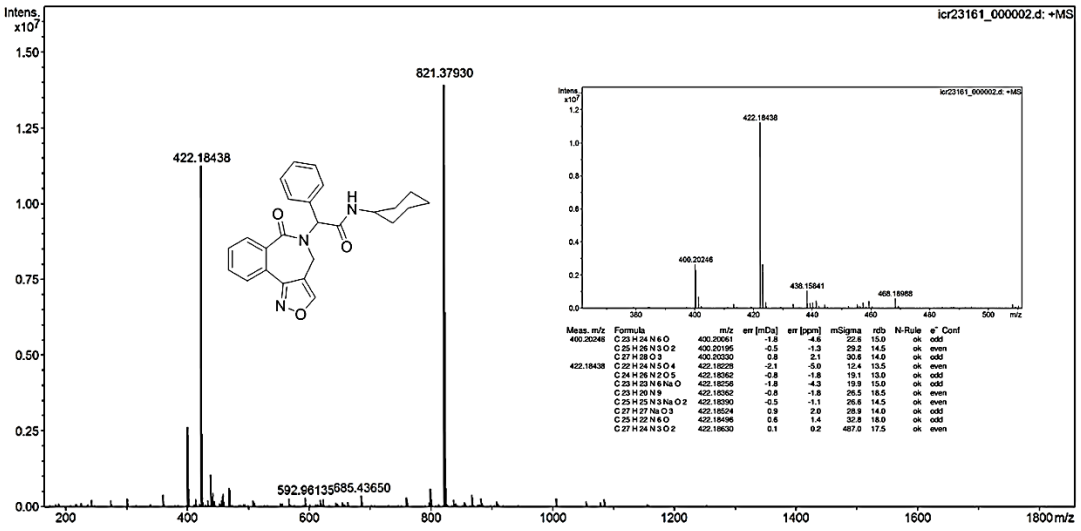
IR (KBr) (9b)



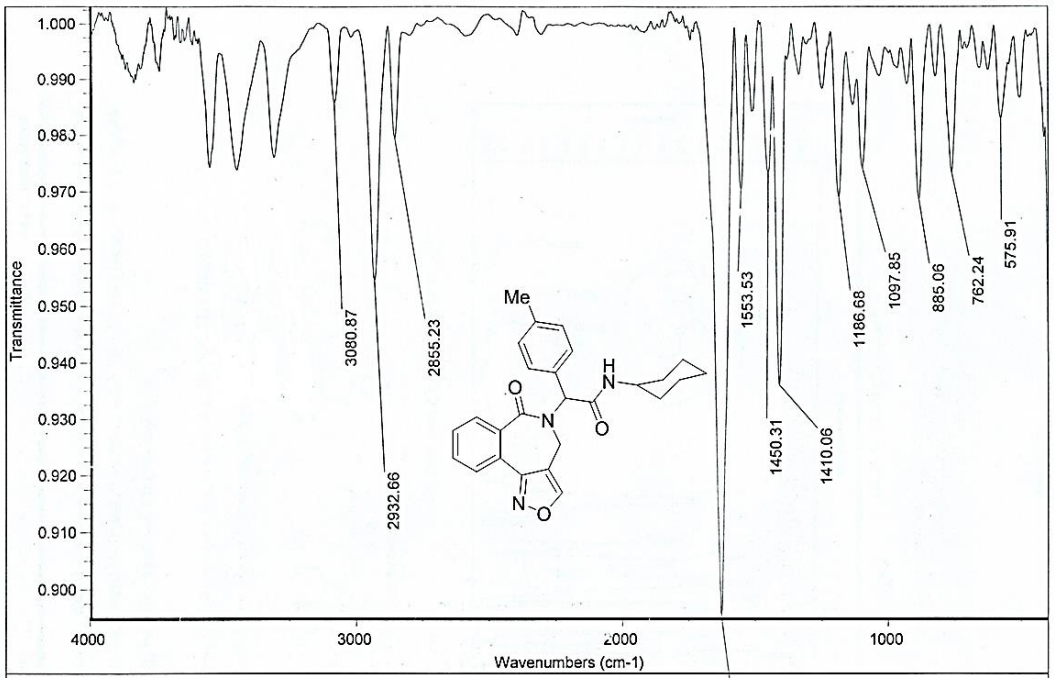
^{1}HNMR (300MHz, $CDCl_3$) (9b)



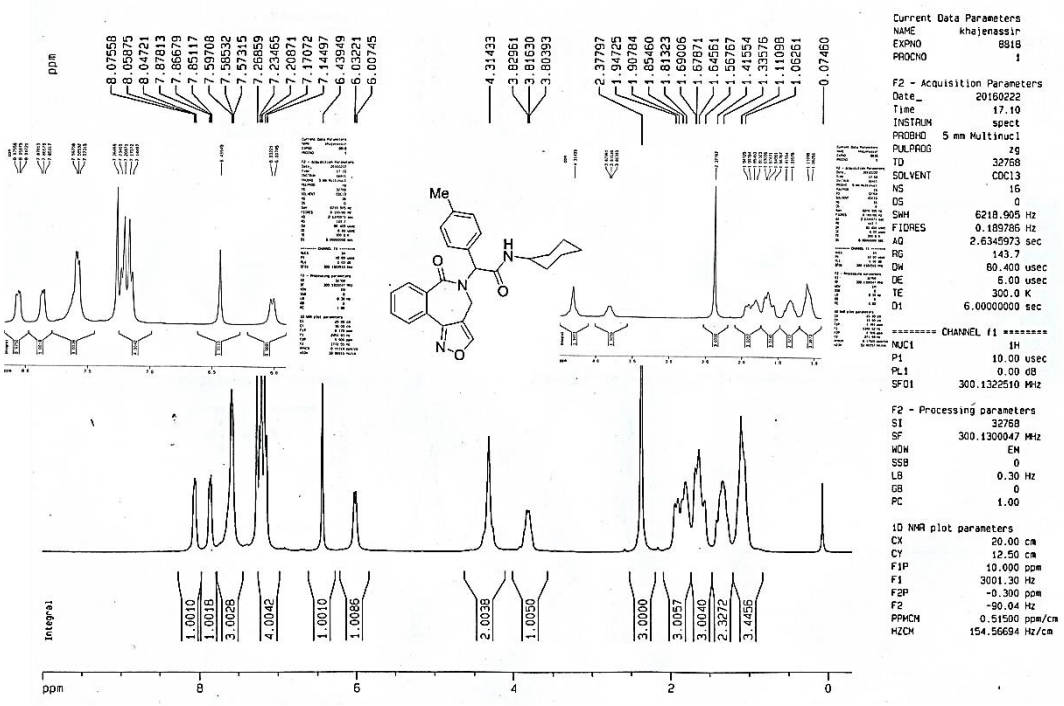
^{13}CNMR (75MHz, $CDCl_3$) (9b)



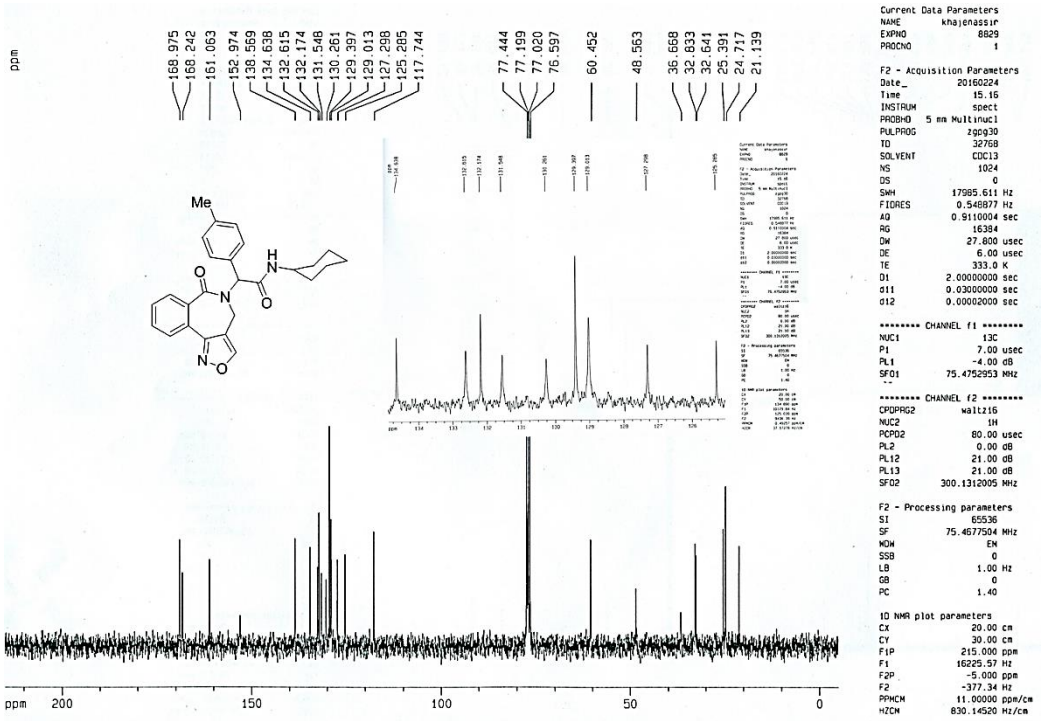
ESI-HRMS (9b)



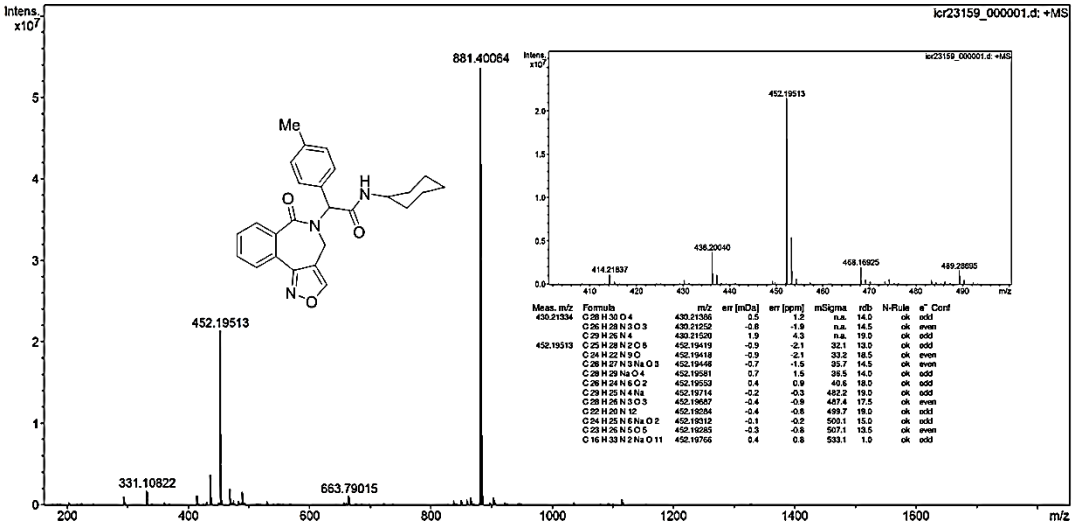
IR (KBr) (9c)



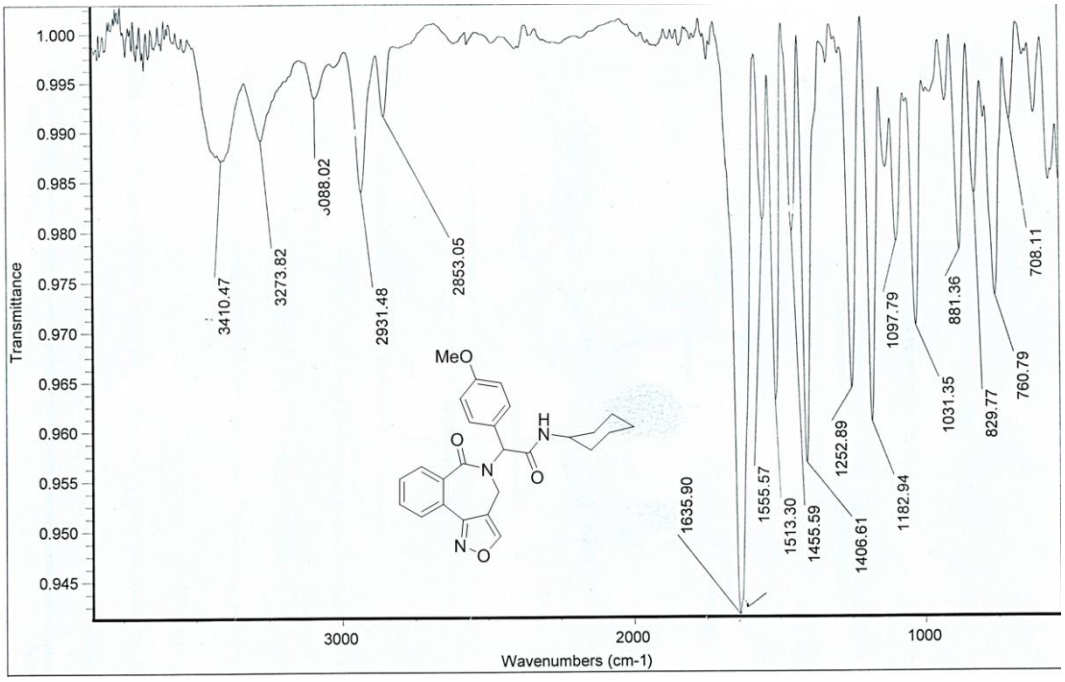
^{1}HNMR (300MHz, $CDCl_3$) (9c)



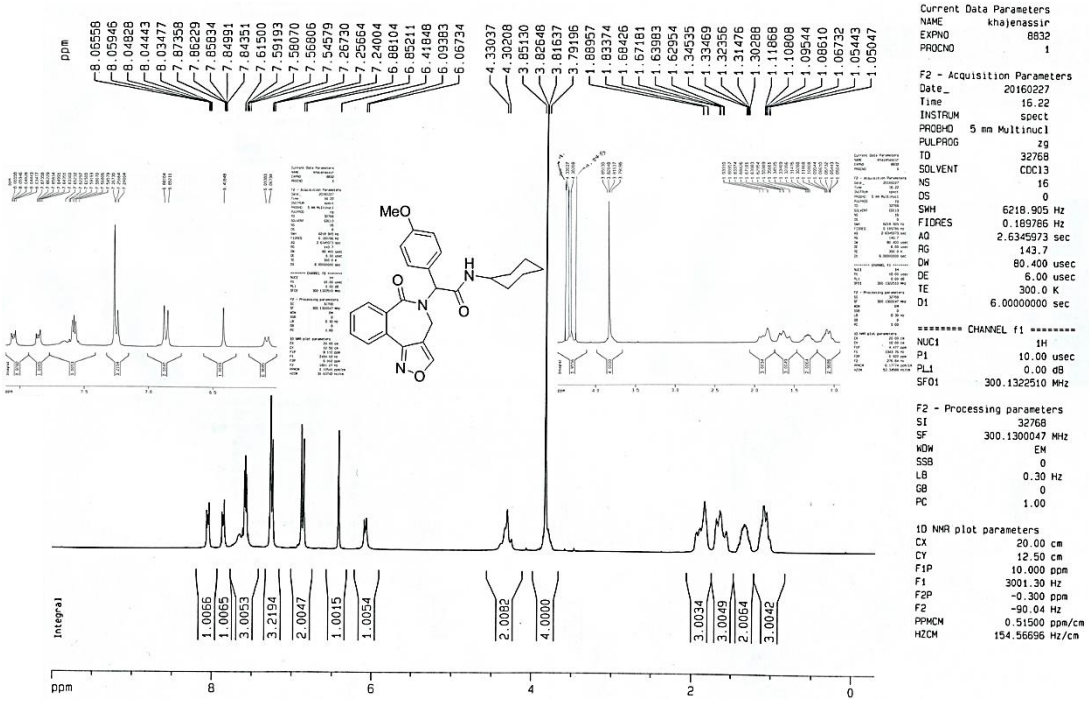
^{13}CNMR (75MHz, $CDCl_3$) (9c)



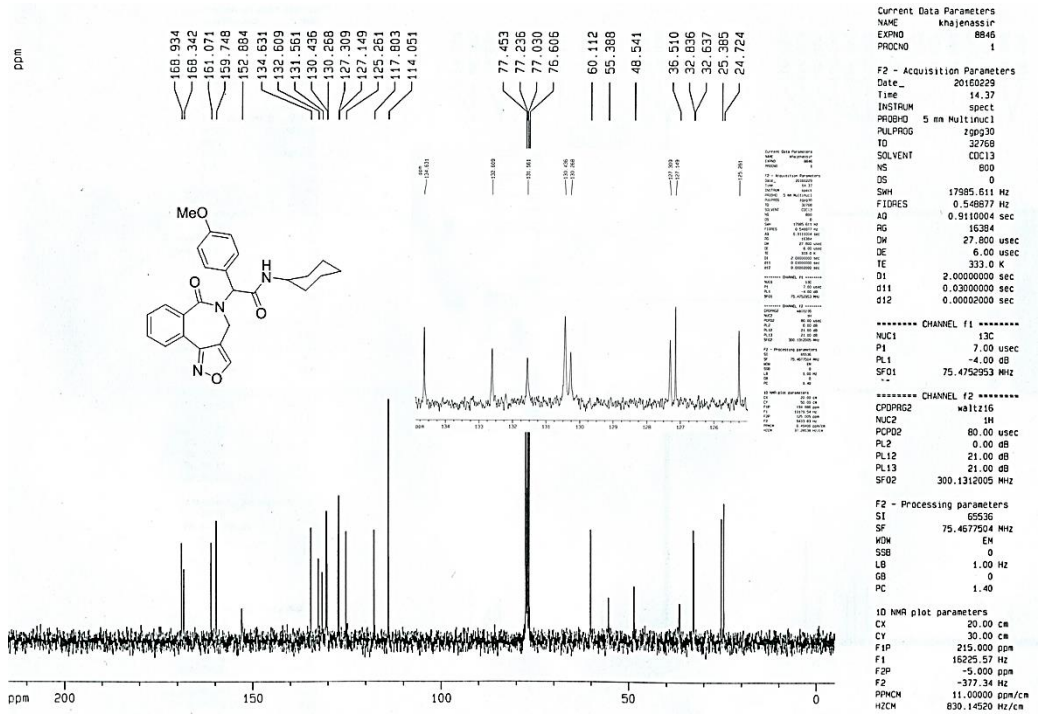
ESI-HRMS (9c)



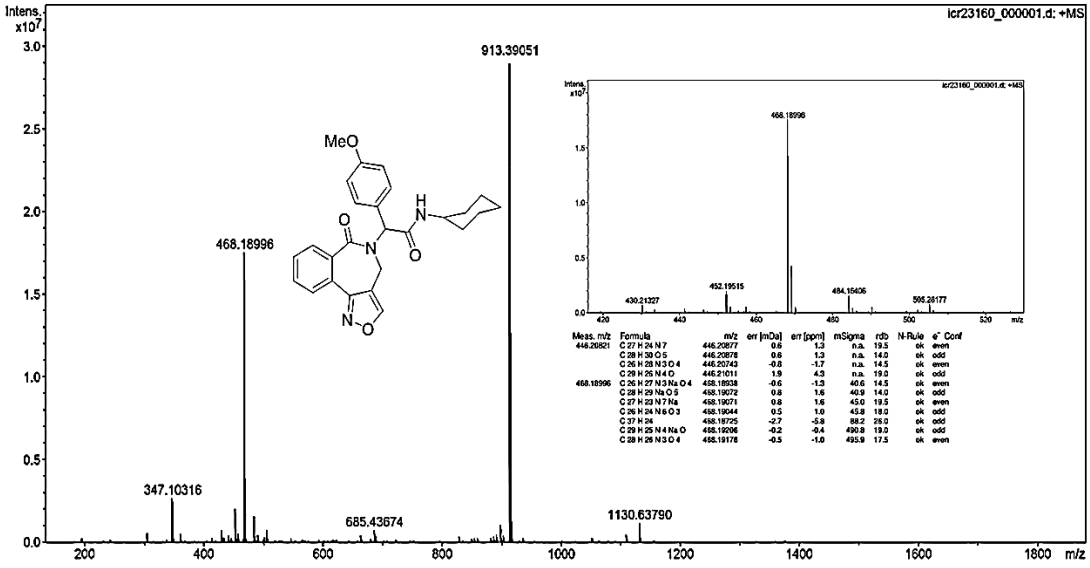
IR (KBr) (9d)



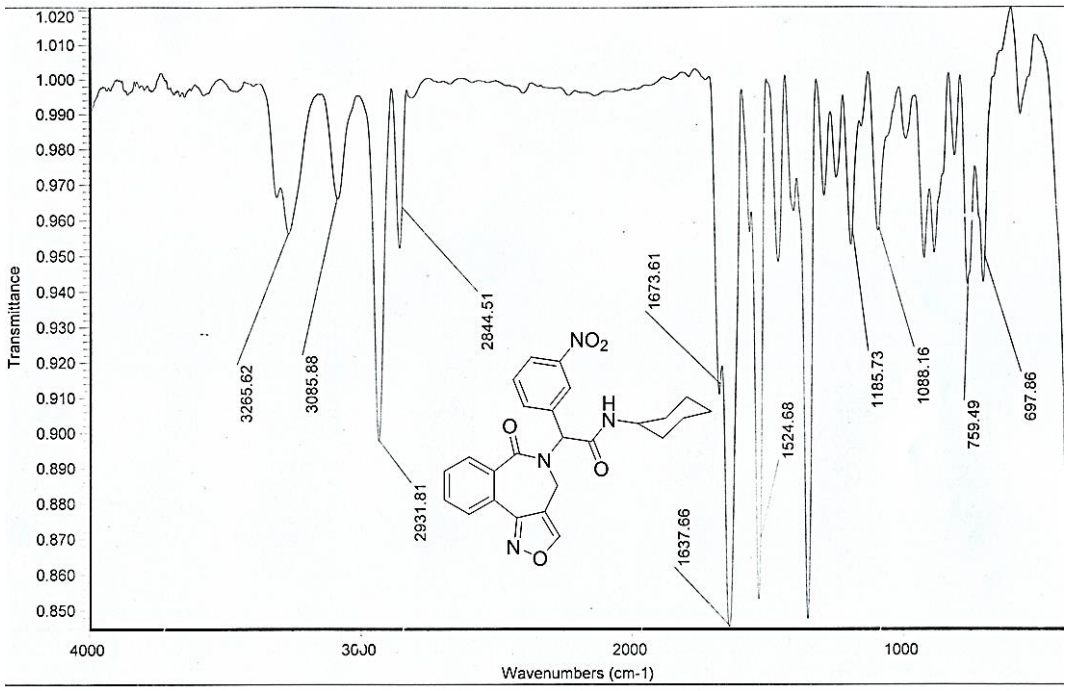
^{1}HNMR (300MHz, $CDCl_3$) (9d)



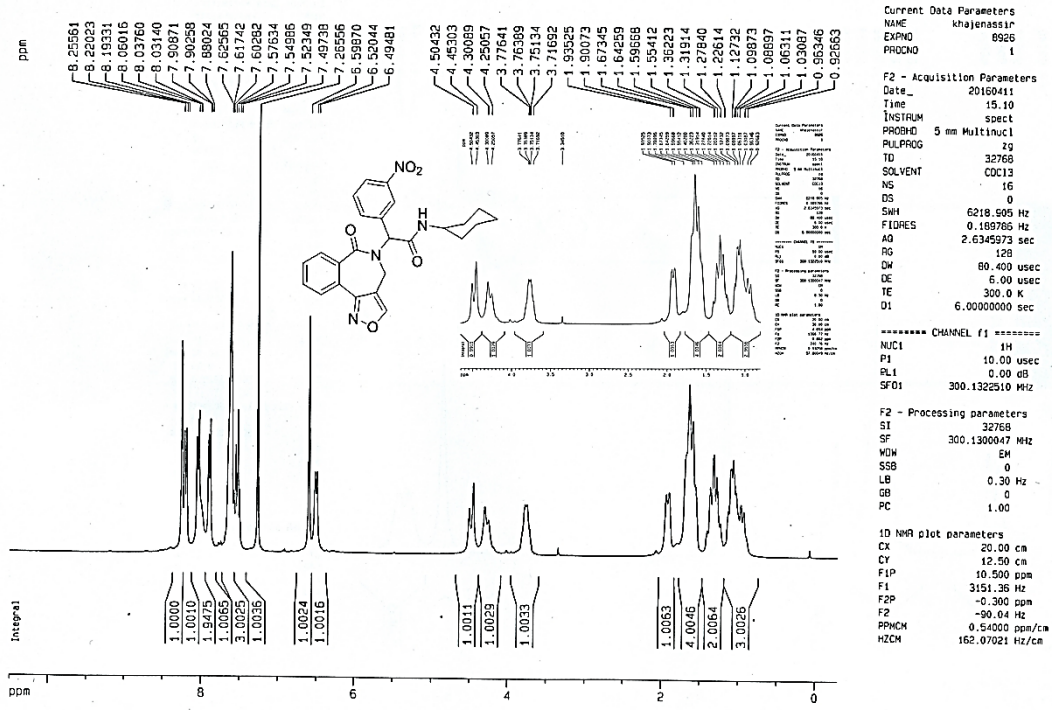
^{13}CNMR (75MHz, $CDCl_3$) (9d)



ESI-HRMS (9d)



IR (KBr) (9e)



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EXPNO 8926
PROCNO 1

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PULPROG zg
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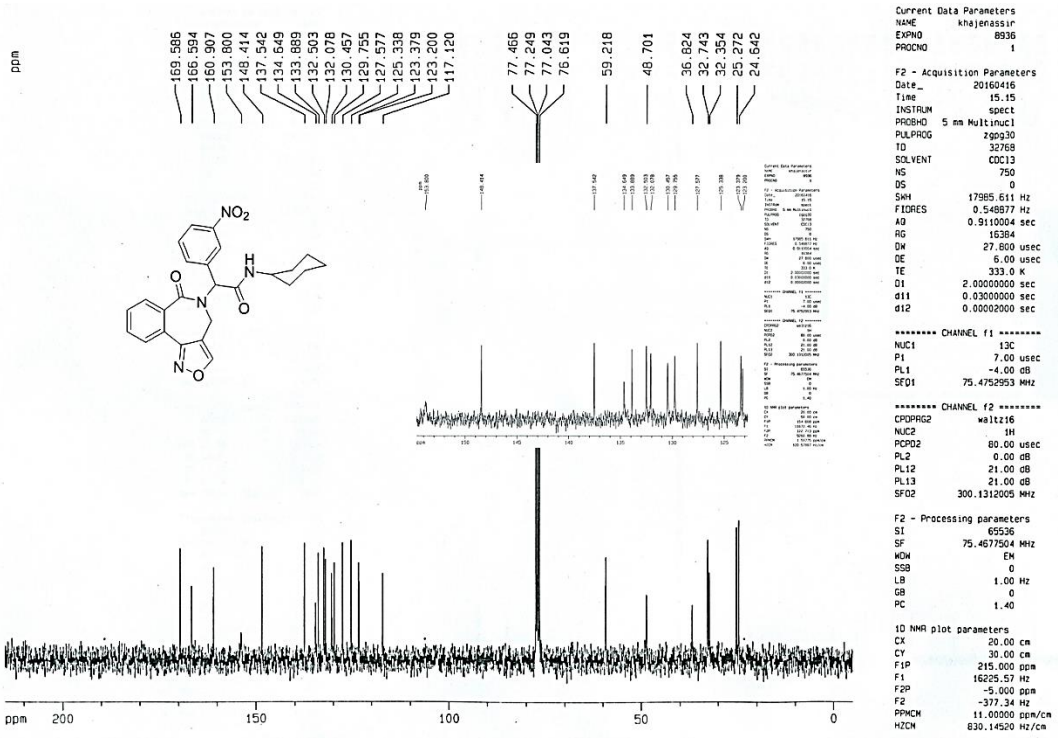
SWH 6218.905 Hz
FIDRES 0.189786 Hz
AQ 2.6345973 sec
RG 128
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1322510 MHz

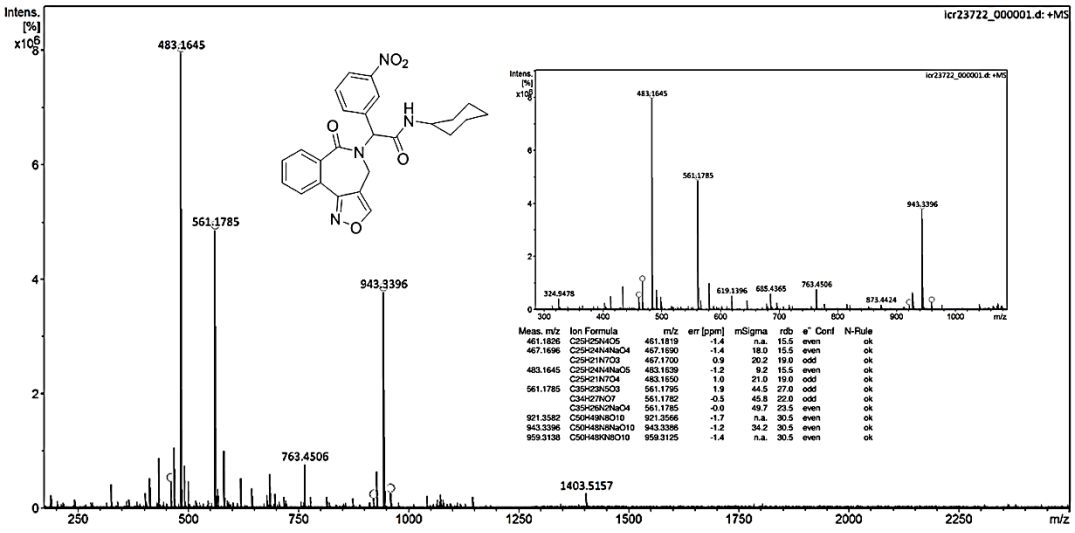
F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.54000 ppm/cm
HZCM 162.07021 Hz/cm

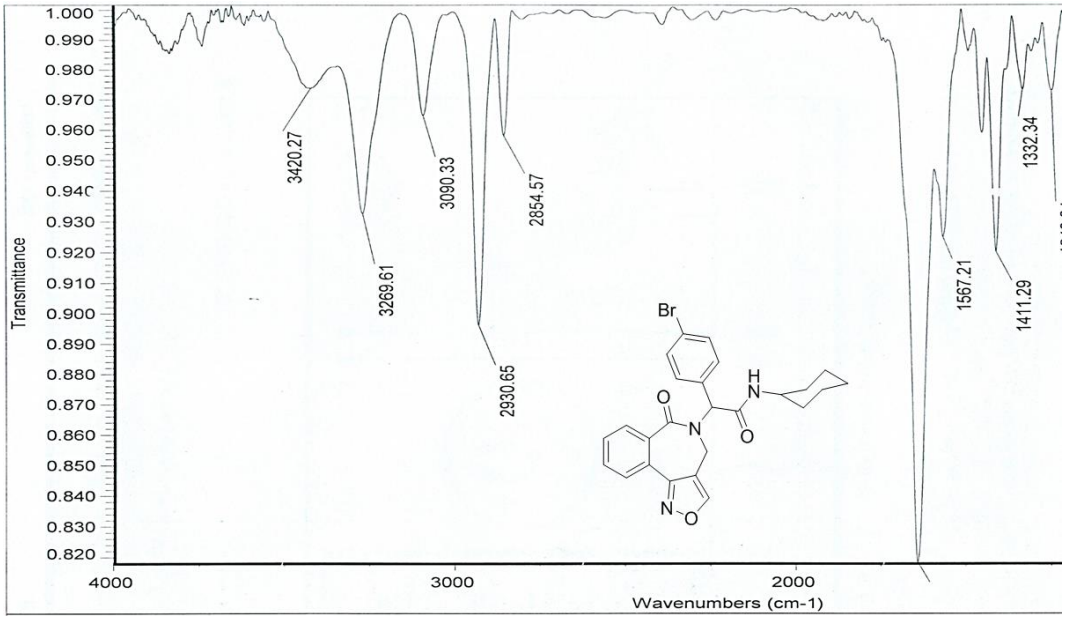
^{1}HNMR (300MHz, $CDCl_3$) (9e)



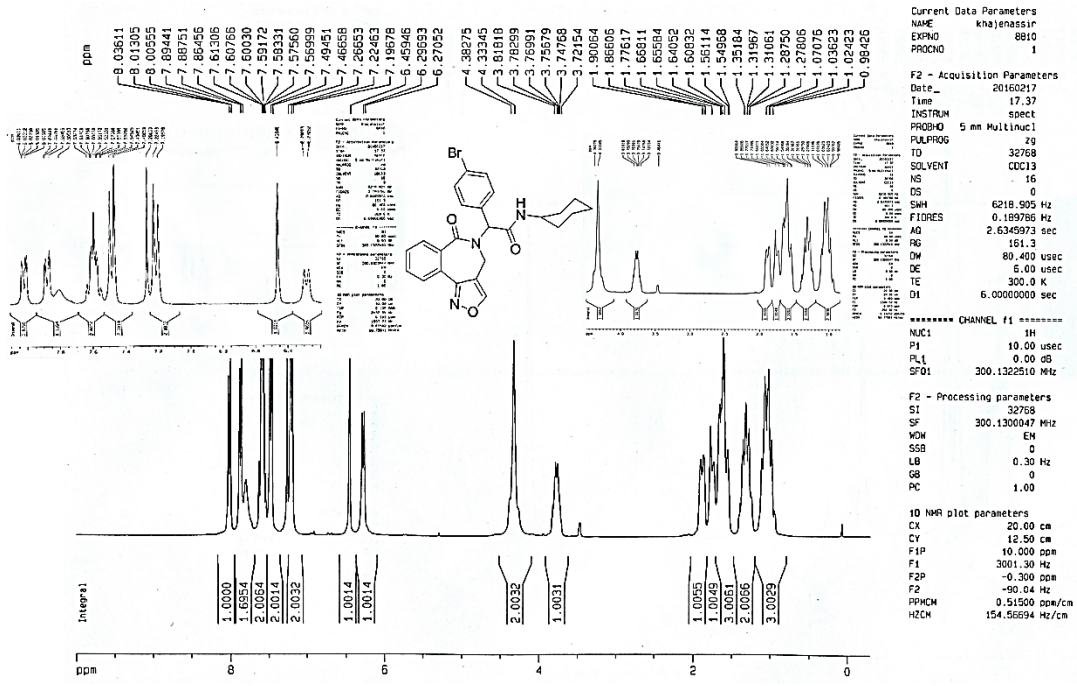
^{13}CNMR (75MHz, $CDCl_3$) (9e)



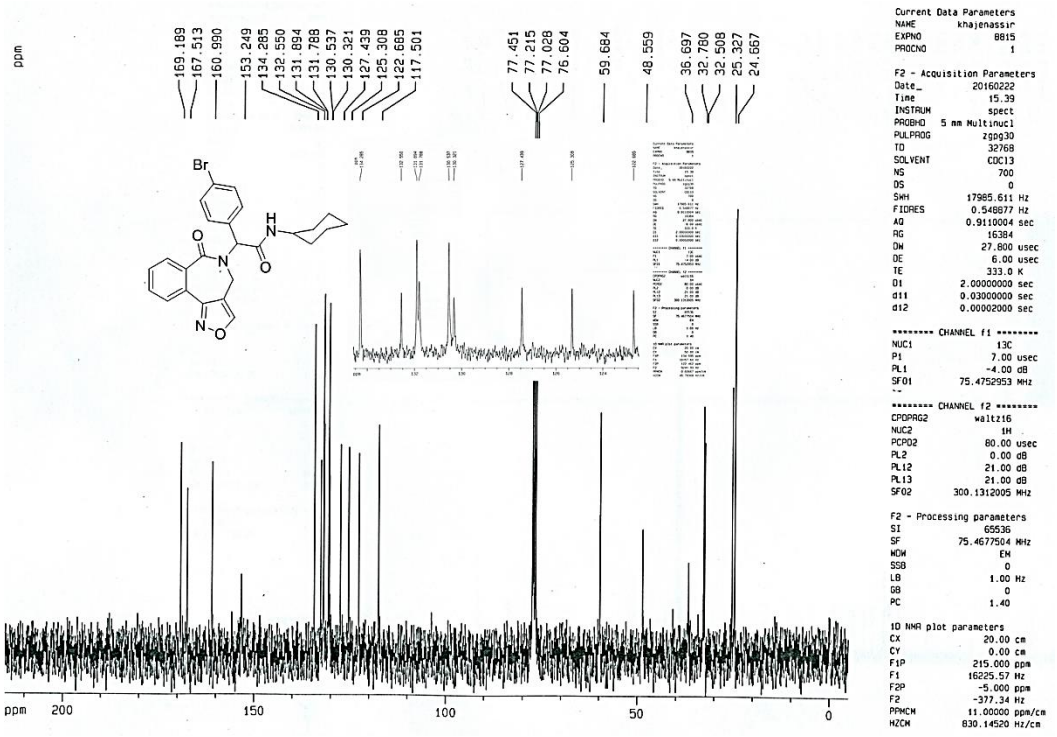
ESI-HRMS (9e)



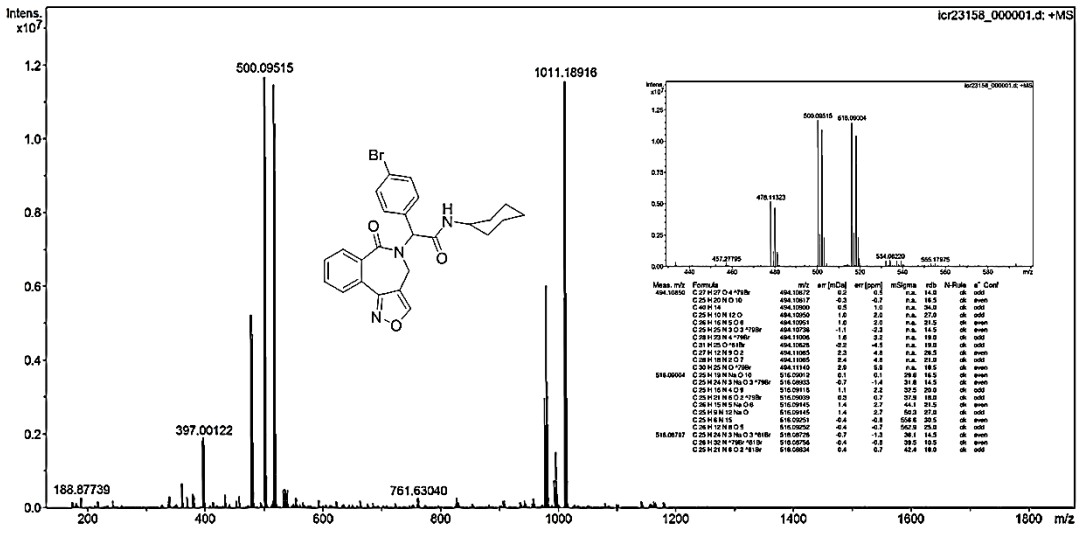
IR (KBr) (9f)



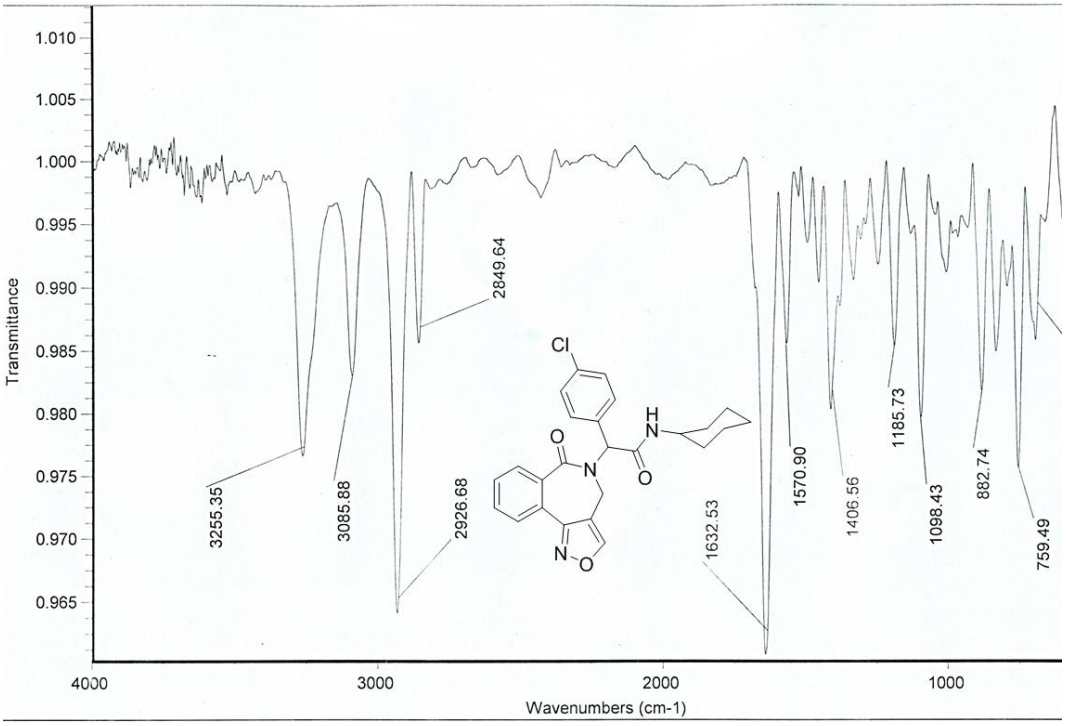
^{1}HNMR (300MHz, $CDCl_3$) (9f)



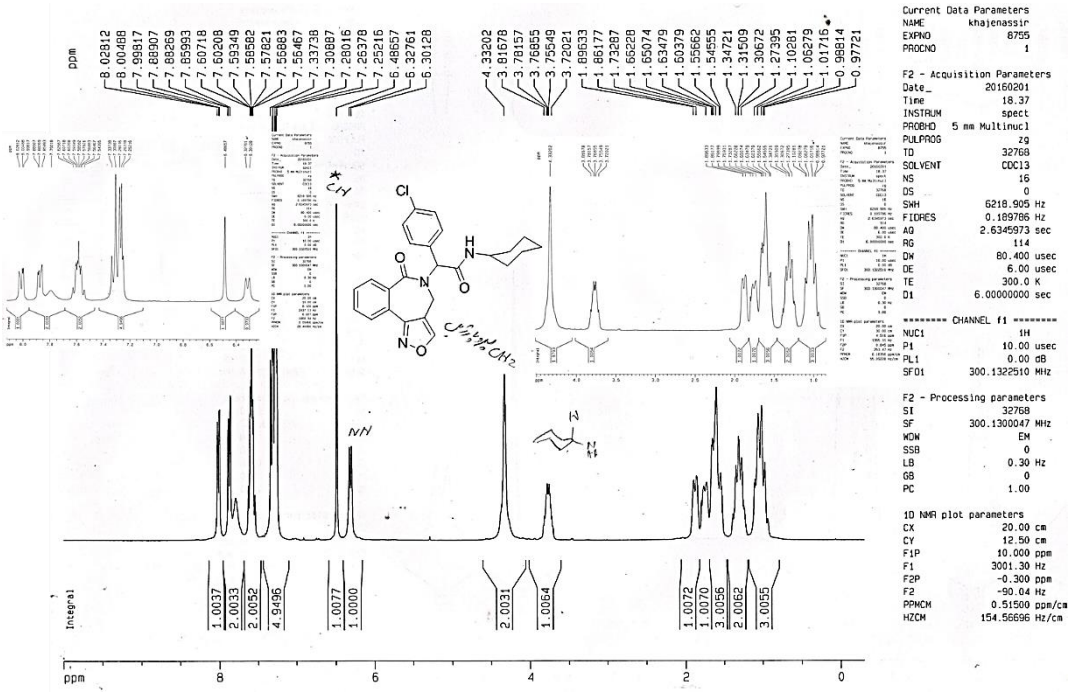
^{13}CNMR (75MHz, $CDCl_3$) (9f)



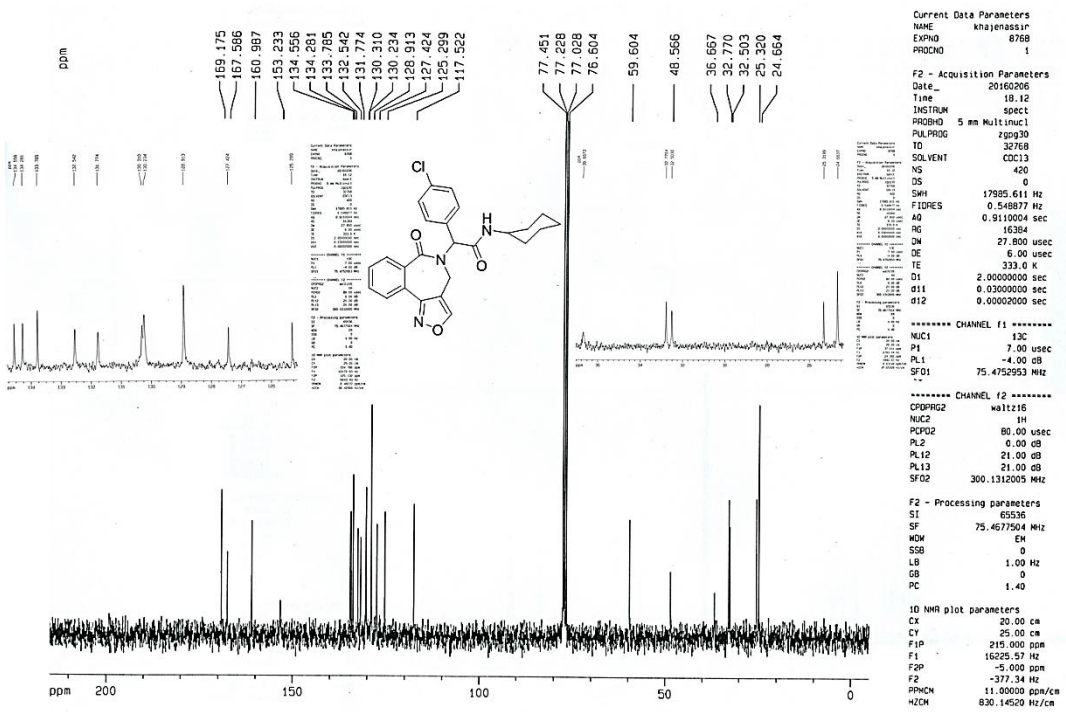
ESI-HRMS (9f)



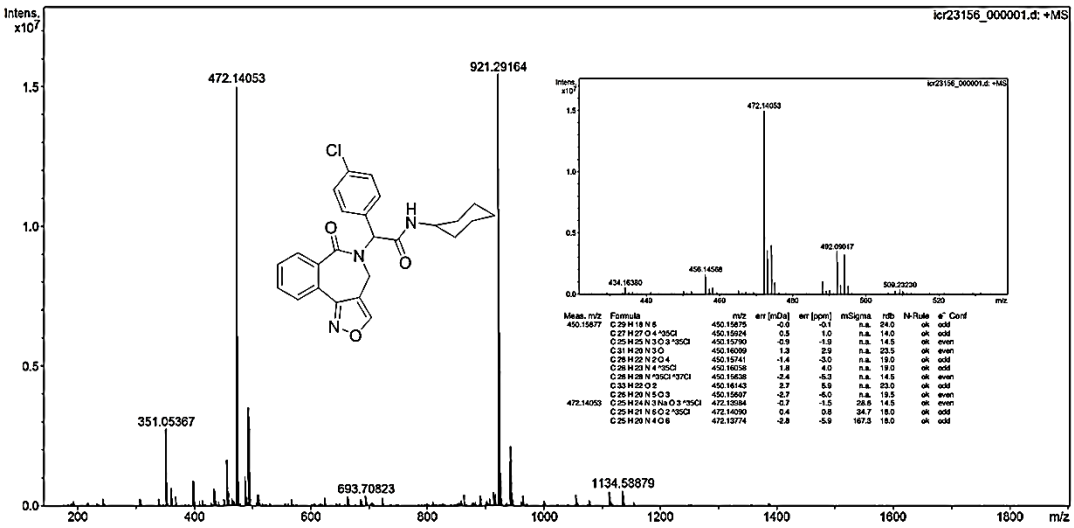
IR (KBr) (9g)



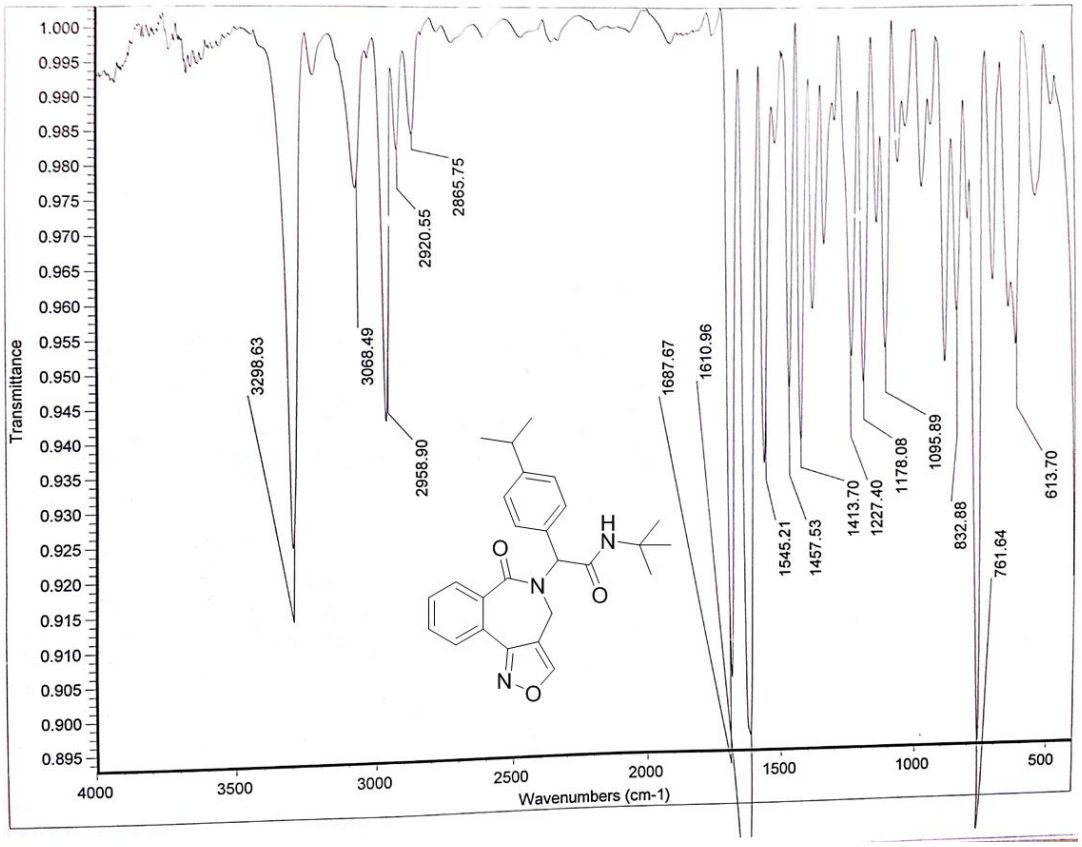
^{1}HNMR (300MHz, $CDCl_3$) (9g)



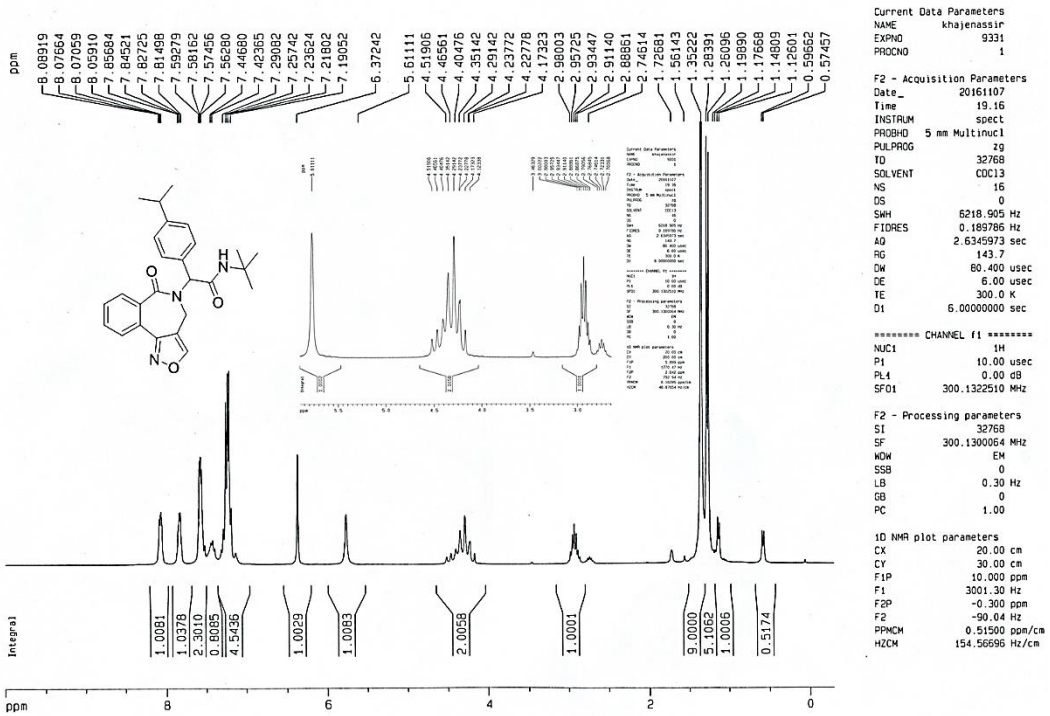
^{13}CNMR (75MHz, $CDCl_3$) (9g)



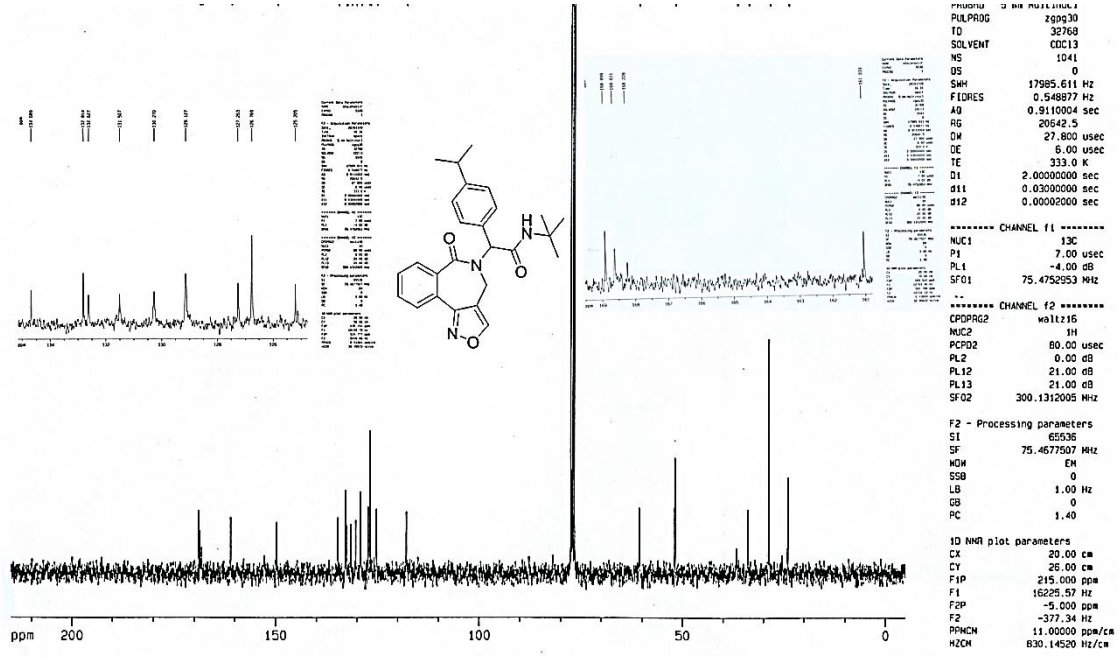
ESI-HRMS (9g)



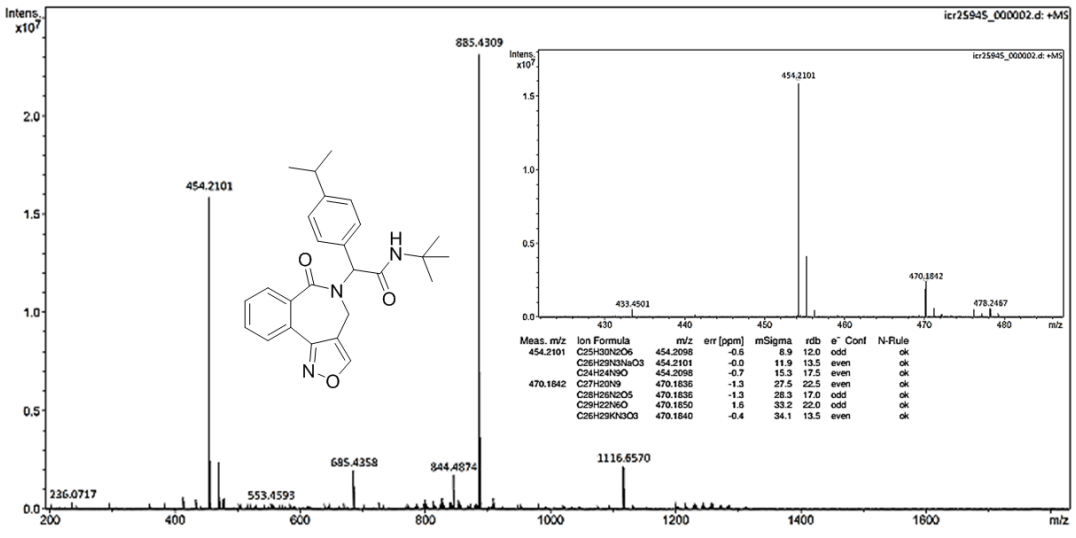
IR (KBr) (9h)



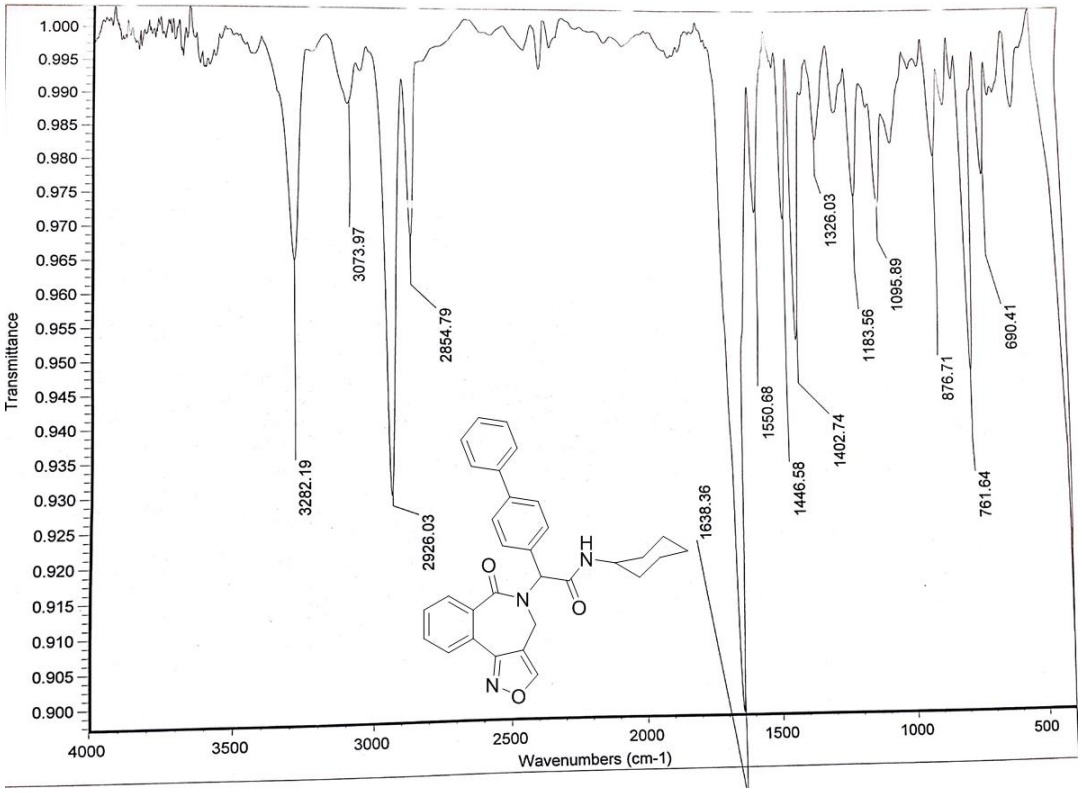
^{1}HNMR (300MHz, $CDCl_3$) (9h)



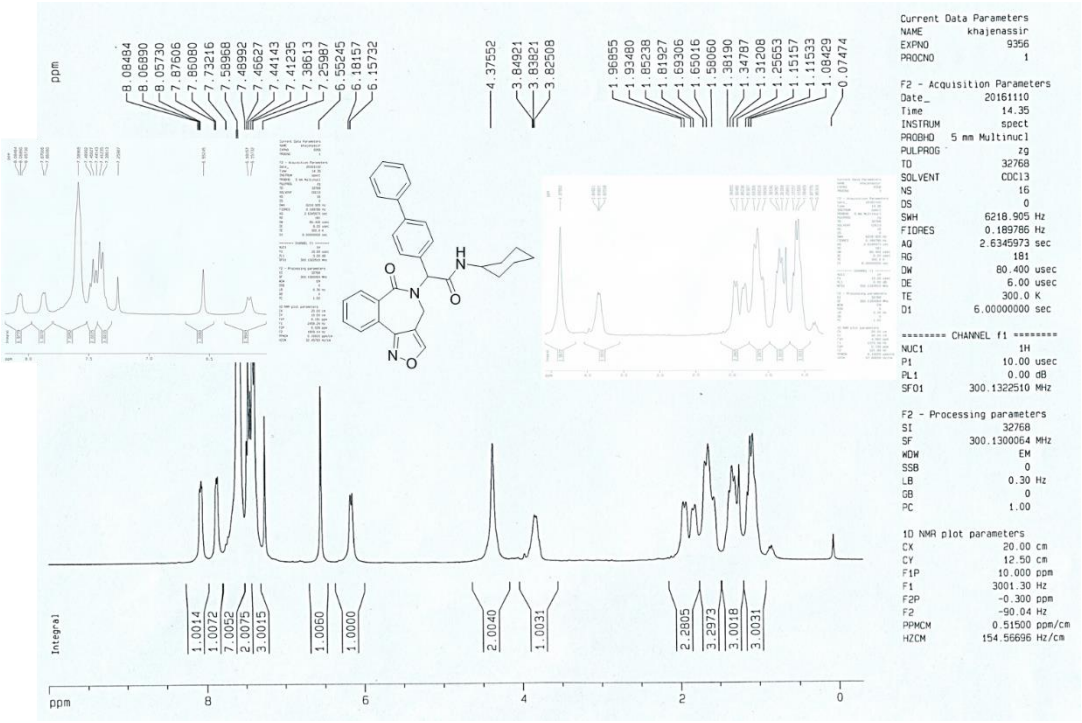
^{13}CNMR (75MHz, $CDCl_3$) (9h)



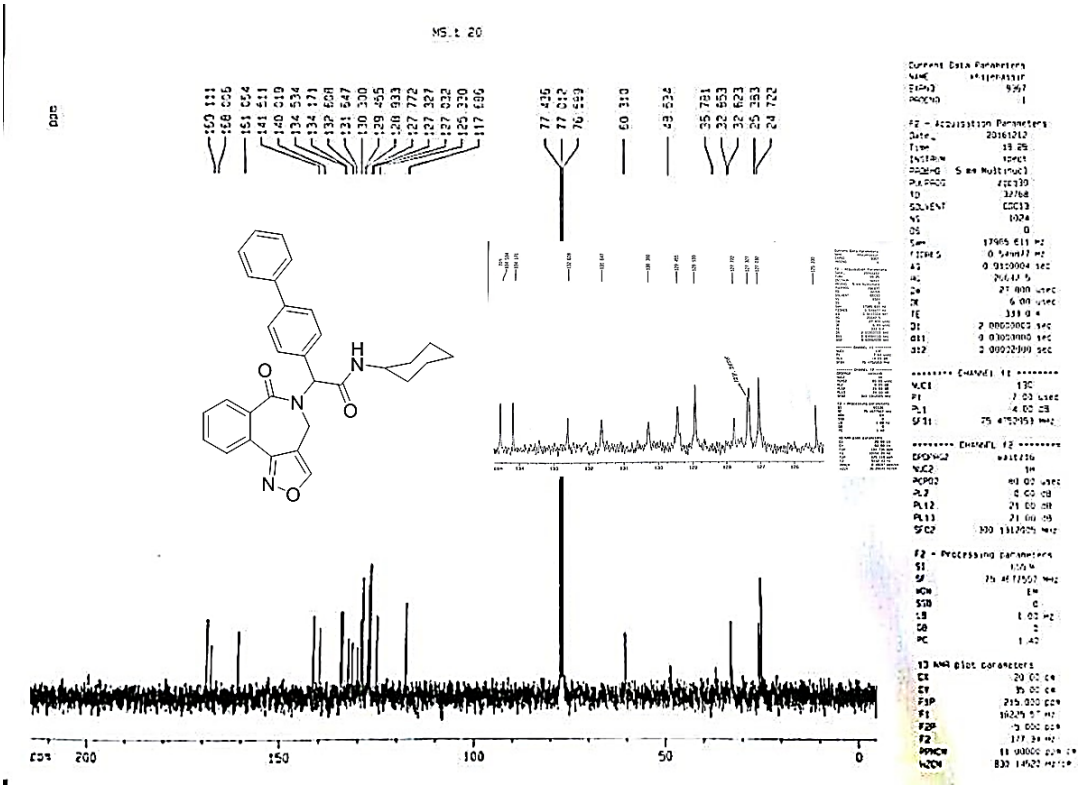
ESI-HRMS (9h)



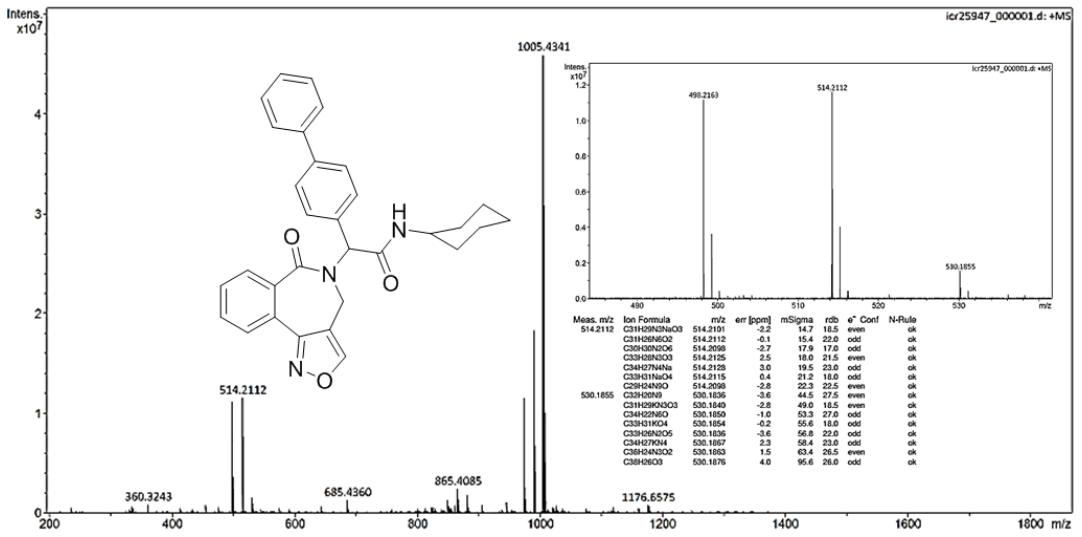
IR (KBr) (9i)



^{1}HNMR (300MHz, $CDCl_3$) (9i)



^{13}CNMR (75MHz, $CDCl_3$) (9i)



ESI-HRMS (9i)

Crystallographic data for compound 9e

Tabelle 1: Kristalldaten und Strukturverfeinerung für sba138

Strukturkennzeichen	sba138	
Summenformel	$C_{25}H_{24}N_4O_5$	
Molmasse	460.48	
Temperatur	200(2) K	
Wellenlänge	0.71073 Å	
Kristallsystem	monoklin	
Raumgruppe	$P2_1/c$	
Z	4	
Gitterkonstanten	a = 15.0879(14) Å	α = 90 °
	b = 17.9838(17) Å	β = 103.191(3) °
	c = 8.8566(9) Å	γ = 90 °
Zellvolumen	2339.7(4) $Å^3$	
Dichte (berechnet)	1.307 g/cm^3	
Absorptionskoeffizient µ	0.093 mm^{-1}	
Kristallform	needle	
Kristallgröße	0.610 x 0.060 x 0.030 mm^3	
Kristallfarbe	colourless	
Gemessener Theta-Bereich	1.386 bis 25.060 °	
Indexgrenzen	$-17 \le h \le 16$, $-21 \le k \le 21$, $-10 \le l \le 10$	
Gemessene Reflexe	14592	
Unabhängige Reflexe	4147 (R(int) = 0.0406)	
Beobachtete Reflexe	2754 ($I > 2\sigma(I)$)	
Absorptionskorrektur	Semi-empirical from equivalents	
Max/min Transmission	0.96 and 0.85	
Strukturverfeinerung	Full-matrix least-squares an F^2	
Daten/Restraints/Parameter	4147 / 316 / 335	
Goodness-of-fit an F^2	1.09	
R-Werte (I>2sigma(I))	R1 = 0.055, wR2 = 0.146	
Extinktionskoeffizient	n/a	
Max/min Restelektronendichte	0.18 und -0.21 $eÅ^{-3}$	

Table 2: Crystal data and structure refinement for sba138.

Identification code	sba138	
Empirical formula	$C_{25}H_{24}N_4O_5$	
Formula weight	460.48	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/c$	
Z	4	
Unit cell dimensions	a = 15.0879(14) Å	α = 90 deg.
	b = 17.9838(17) Å	β = 103.191(3) deg.
	c = 8.8566(9) Å	γ = 90 deg.
Volume	2339.7(4) $Å^3$	
Density (calculated)	1.31 g/cm^3	
Absorption coefficient	0.09 mm^{-1}	
Crystal shape	needle	
Crystal size	0.610 x 0.060 x 0.030 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.4 to 25.1 deg.	
Index ranges	$-17 \le h \le 16$, $-21 \le k \le 21$, $-10 \le l \le 10$	
Reflections collected	14592	
Independent reflections	4147 (R(int) = 0.0406)	
Observed reflections	2754 ($I > 2\sigma(I)$)	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.96 and 0.85
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4147 / 316 / 335
Goodness-of-fit on F^2	1.09
Final R indices (I>2sigma(I))	R1 = 0.055, wR2 = 0.146
Largest diff. peak and hole	0.18 and -0.21 $eÅ^{-3}$

Tabelle 3: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter ($Å^2$) für sba138. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for sba138. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.4403(2)	0.7390(2)	0.6218(3)	0.0388(6)
O1	0.4271(1)	0.7981(1)	0.6829(2)	0.0517(5)
N2	0.3752(1)	0.6858(1)	0.5902(2)	0.0361(5)
C3	0.3857(2)	0.6176(1)	0.5042(3)	0.0385(6)
H3A	0.4054	0.6302	0.4081	0.046
H3B	0.3268	0.5909	0.4752	0.046
C4	0.4552(2)	0.5696(2)	0.6054(3)	0.0412(6)
C5	0.5473(2)	0.5933(2)	0.6499(3)	0.0439(7)
N6	0.5989(2)	0.5450(2)	0.7406(3)	0.0588(7)
C6	0.2912(2)	0.6973(1)	0.6447(3)	0.0337(6)
H6	0.3041	0.7357	0.7286	0.040
C7	0.2152(2)	0.7270(1)	0.5140(3)	0.0333(6)
O7	0.2087(1)	0.7054(1)	0.3797(2)	0.0440(5)
N8	0.1592(1)	0.7748(1)	0.5574(2)	0.0369(5)
H8	0.1714	0.7885	0.6553	0.044
C8	0.4548(2)	0.5039(2)	0.6761(4)	0.0540(8)
H8A	0.4025	0.4736	0.6698	0.065
O9	0.5394(2)	0.4871(1)	0.7578(3)	0.0658(6)
C11	0.5304(2)	0.7277(2)	0.5773(3)	0.0420(6)
C12	0.5820(2)	0.6621(2)	0.5975(3)	0.0446(7)
C13	0.6687(2)	0.6622(2)	0.5648(3)	0.0594(8)
H13	0.7045	0.6183	0.5801	0.071
C14	0.7023(2)	0.7258(2)	0.5106(4)	0.0688(10)
H14	0.7605	0.7250	0.4864	0.083
C15	0.6524(2)	0.7899(2)	0.4917(4)	0.0657(9)
H15	0.6759	0.8334	0.4541	0.079
C16	0.5676(2)	0.7916(2)	0.5271(3)	0.0543(8)
H16	0.5344	0.8369	0.5171	0.065
C21	0.2619(2)	0.6264(1)	0.7136(3)	0.0356(6)
C22	0.1998(2)	0.5775(2)	0.6276(3)	0.0486(7)
H22	0.1716	0.5881	0.5225	0.058
C24	0.2173(3)	0.4961(2)	0.8500(4)	0.0687(9)
H24	0.2017	0.4516	0.8955	0.082
C25	0.2778(2)	0.5450(2)	0.9340(4)	0.0631(9)
H25	0.3045	0.5347	1.0398	0.076
C26	0.3007(2)	0.6092(2)	0.8677(3)	0.0470(7)
H26	0.3436	0.6423	0.9280	0.056
C23	0.1795(2)	0.5128(2)	0.6974(4)	0.0655(9)
N27	0.1136(11)	0.4632(8)	0.5941(17)	0.151(9)
O28	0.1072(15)	0.3992(8)	0.652(3)	0.137(7)
O29	0.0674(16)	0.4854(12)	0.4644(11)	0.103(5)
N27B	0.1200(8)	0.4570(6)	0.6099(11)	0.109(5)
O28B	0.0730(18)	0.4186(12)	0.6827(17)	0.149(7)
O29B	0.1164(18)	0.4563(10)	0.4666(10)	0.104(5)
C31	0.0783(2)	0.8065(1)	0.4536(3)	0.0372(6)

H31	0.0944	0.8215	0.3545	0.045
C32	0.0017(2)	0.7506(2)	0.4172(3)	0.0465(7)
H32A	-0.0101	0.7312	0.5153	0.056
H32B	0.0203	0.7082	0.3600	0.056
C33	-0.0853(2)	0.7844(2)	0.3206(4)	0.0575(8)
H33A	-0.0756	0.7987	0.2177	0.069
H33B	-0.1346	0.7469	0.3046	0.069
C34	-0.1135(2)	0.8520(2)	0.3997(4)	0.0542(8)
H34A	-0.1682	0.8745	0.3320	0.065
H34B	-0.1293	0.8367	0.4977	0.065
C35	-0.0379(2)	0.9089(2)	0.4342(4)	0.0579(8)
H35A	-0.0567	0.9511	0.4913	0.069
H35B	-0.0268	0.9282	0.3354	0.069
C36	0.0497(2)	0.8755(2)	0.5301(4)	0.0476(7)
H36A	0.0407	0.8622	0.6341	0.057
H36B	0.0988	0.9130	0.5438	0.057

Tabelle 4: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba138.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba138.)

Atom	x	y	z	U_{eq}
H3A	0.4054	0.6302	0.4081	0.046
H3B	0.3268	0.5909	0.4752	0.046
H6	0.3041	0.7357	0.7286	0.040
H8	0.1714	0.7885	0.6553	0.044
H8A	0.4025	0.4736	0.6698	0.065
H13	0.7045	0.6183	0.5801	0.071
H14	0.7605	0.7250	0.4864	0.083
H15	0.6759	0.8334	0.4541	0.079
H16	0.5344	0.8369	0.5171	0.065
H22	0.1716	0.5881	0.5225	0.058
H24	0.2017	0.4516	0.8955	0.082
H25	0.3045	0.5347	1.0398	0.076
H26	0.3436	0.6423	0.9280	0.056
H31	0.0944	0.8215	0.3545	0.045
H32A	-0.0101	0.7312	0.5153	0.056
H32B	0.0203	0.7082	0.3600	0.056
H33A	-0.0756	0.7987	0.2177	0.069
H33B	-0.1346	0.7469	0.3046	0.069
H34A	-0.1682	0.8745	0.3320	0.065
H34B	-0.1293	0.8367	0.4977	0.065
H35A	-0.0567	0.9511	0.4913	0.069
H35B	-0.0268	0.9282	0.3354	0.069
H36A	0.0407	0.8622	0.6341	0.057
H36B	0.0988	0.9130	0.5438	0.057

Tabelle 5: Anisotrope Auslenkungsparameter ($Å^2$) für sba138. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba138. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0370(14)	0.0413(15)	0.0354(14)	0.0042(12)	0.0028(11)	-0.0015(12)

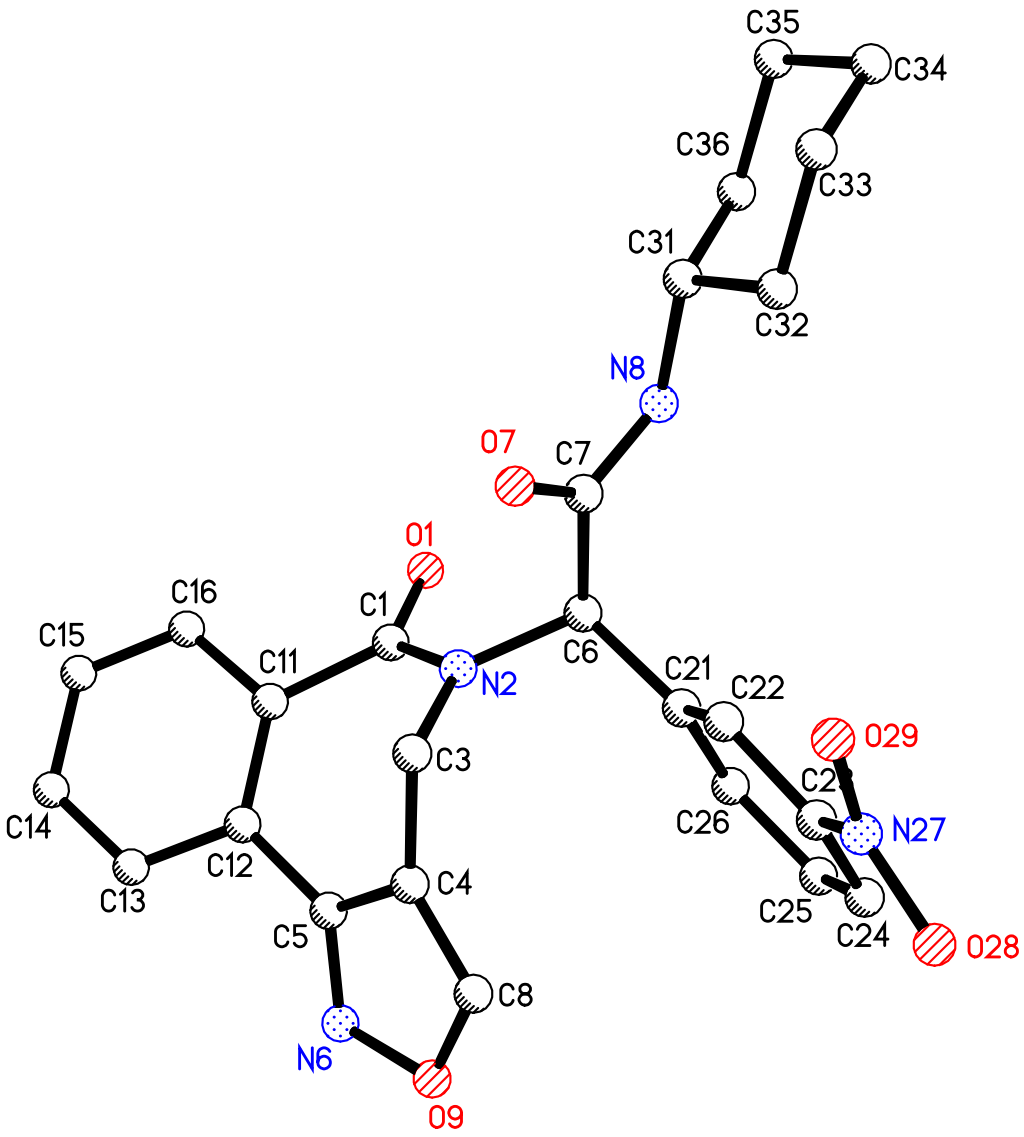
O1	0.0565(12)	0.0379(11)	0.0598(13)	-0.0057(10)	0.0116(10)	-0.0030(9)
N2	0.0326(11)	0.0379(12)	0.0375(12)	-0.0048(9)	0.0074(9)	-0.0002(9)
C3	0.0377(14)	0.0405(15)	0.0377(14)	-0.0055(11)	0.0093(11)	0.0023(11)
C4	0.0467(15)	0.0411(15)	0.0374(14)	-0.0041(12)	0.0131(12)	0.0067(12)
C5	0.0422(15)	0.0501(16)	0.0381(15)	-0.0010(12)	0.0066(12)	0.0108(12)
N6	0.0592(15)	0.0543(15)	0.0579(16)	0.0043(13)	0.0030(13)	0.0134(13)
C6	0.0348(13)	0.0360(14)	0.0287(12)	-0.0022(10)	0.0038(10)	0.0038(11)
C7	0.0359(13)	0.0339(13)	0.0304(13)	0.0048(11)	0.0083(10)	0.0001(11)
O7	0.0479(11)	0.0578(12)	0.0263(9)	0.0029(8)	0.0083(8)	0.0086(9)
N8	0.0385(11)	0.0401(12)	0.0300(11)	-0.0001(9)	0.0039(9)	0.0060(10)
C8	0.0617(19)	0.0471(17)	0.0529(18)	-0.0041(14)	0.0124(15)	0.0078(14)
O9	0.0805(16)	0.0502(13)	0.0630(14)	0.0098(11)	0.0082(12)	0.0166(12)
C11	0.0357(14)	0.0519(16)	0.0346(14)	0.0025(12)	0.0001(11)	-0.0049(12)
C12	0.0373(14)	0.0600(18)	0.0339(14)	-0.0006(12)	0.0027(11)	-0.0004(13)
C13	0.0371(16)	0.090(2)	0.0487(18)	-0.0010(17)	0.0051(13)	0.0031(16)
C14	0.0416(18)	0.107(3)	0.057(2)	0.0009(19)	0.0101(15)	-0.0170(18)
C15	0.0480(18)	0.089(3)	0.055(2)	0.0145(18)	0.0019(15)	-0.0224(17)
C16	0.0454(16)	0.064(2)	0.0491(17)	0.0091(15)	0.0013(13)	-0.0149(14)
C21	0.0380(14)	0.0367(14)	0.0316(13)	0.0037(10)	0.0071(11)	0.0076(11)
C22	0.0590(18)	0.0490(17)	0.0382(15)	0.0020(12)	0.0118(13)	-0.0108(14)
C24	0.090(3)	0.054(2)	0.071(2)	0.0246(17)	0.0341(19)	0.0072(18)
C25	0.065(2)	0.074(2)	0.0507(18)	0.0279(16)	0.0147(15)	0.0179(17)
C26	0.0443(16)	0.0597(18)	0.0350(14)	0.0073(13)	0.0052(12)	0.0091(13)
C23	0.084(2)	0.0489(18)	0.067(2)	0.0003(15)	0.0243(17)	-0.0201(17)
N27	0.209(15)	0.092(8)	0.127(8)	0.015(6)	-0.015(9)	-0.095(9)
O28	0.164(12)	0.080(6)	0.165(12)	0.016(7)	0.031(8)	-0.065(7)
O29	0.114(9)	0.092(8)	0.099(5)	-0.021(4)	0.018(5)	-0.048(7)
N27B	0.165(10)	0.083(7)	0.086(5)	-0.011(4)	0.040(5)	-0.074(7)
O28B	0.228(15)	0.113(10)	0.123(6)	-0.016(6)	0.073(7)	-0.116(11)
O29B	0.137(12)	0.090(7)	0.082(4)	-0.030(4)	0.022(5)	-0.049(8)
C31	0.0348(13)	0.0450(15)	0.0318(13)	0.0071(11)	0.0075(10)	0.0039(11)
C32	0.0411(15)	0.0494(17)	0.0500(17)	-0.0102(13)	0.0127(12)	-0.0032(13)
C33	0.0385(16)	0.077(2)	0.0554(18)	-0.0172(16)	0.0067(13)	0.0025(15)
C34	0.0374(15)	0.068(2)	0.0561(18)	-0.0038(15)	0.0080(13)	0.0066(14)
C35	0.0467(17)	0.0494(18)	0.076(2)	0.0132(16)	0.0101(15)	0.0113(14)
C36	0.0413(15)	0.0373(15)	0.0617(18)	0.0021(13)	0.0064(13)	0.0020(12)

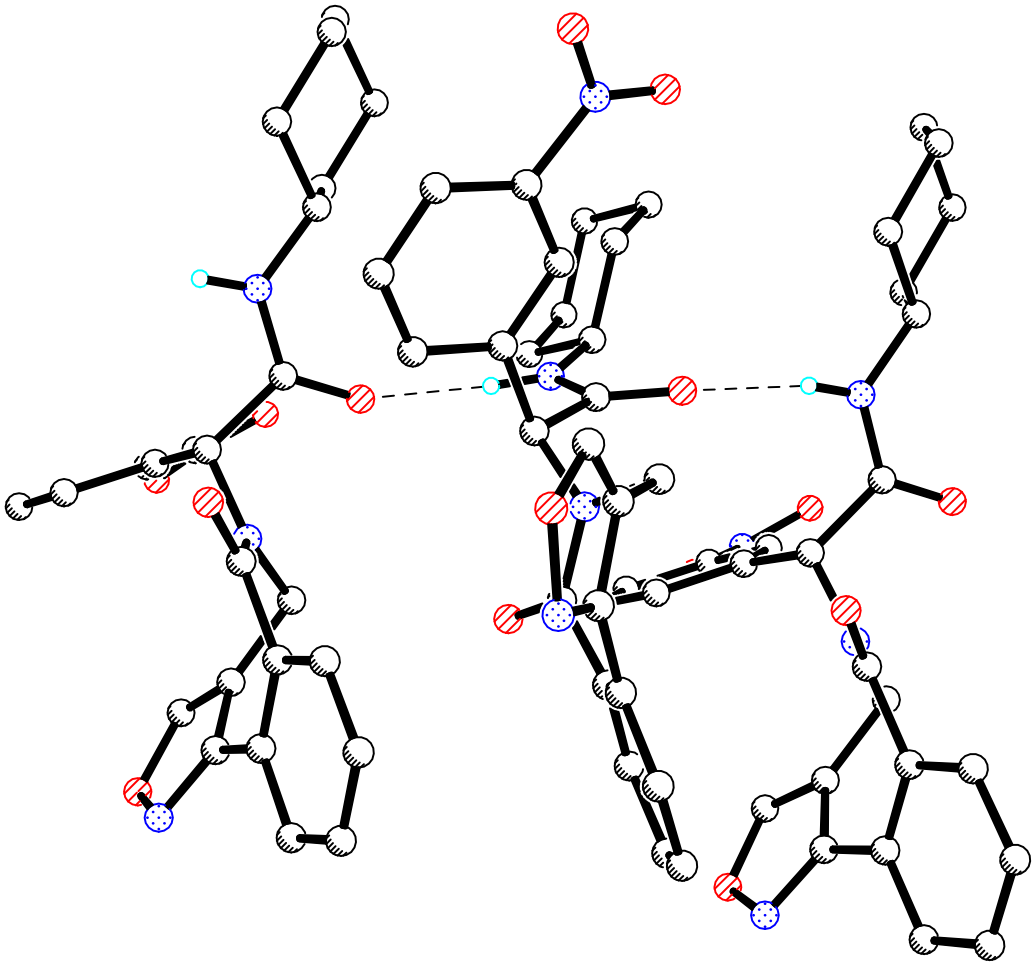
Tabelle 6: Bindungslängen (Å) und -winkel (°) für sba138.
(Bond lengths (Å) and angles (deg) for sba138.)

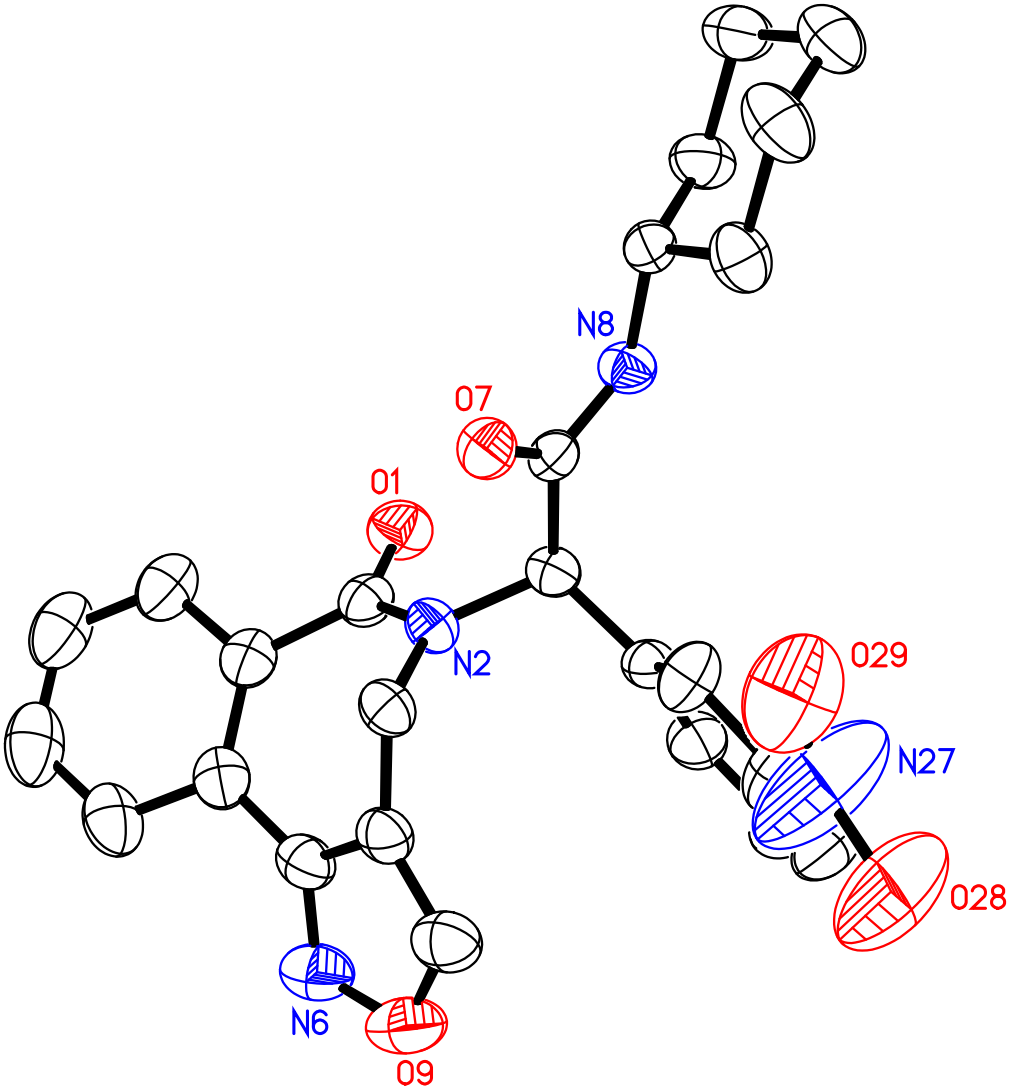
C1-O1	1.229(3)	C11-C16	1.396(4)
C1-N2	1.354(3)	C11-C12	1.403(4)
C1-C11	1.512(4)	C12-C13	1.403(4)
N2-C6	1.469(3)	C13-C14	1.380(5)
N2-C3	1.472(3)	C13-H13	0.9500
C3-C4	1.489(4)	C14-C15	1.366(5)
C3-H3A	0.9900	C14-H14	0.9500
C3-H3B	0.9900	C15-C16	1.385(5)
C4-C8	1.338(4)	C15-H15	0.9500
C4-C5	1.421(4)	C16-H16	0.9500
C5-N6	1.312(4)	C21-C22	1.380(4)
C5-C12	1.459(4)	C21-C26	1.391(3)
N6-O9	1.406(3)	C22-C23	1.386(4)
C6-C21	1.523(3)	C22-H22	0.9500
C6-C7	1.528(3)	C24-C25	1.360(5)
C6-H6	1.0000	C24-C23	1.375(5)
C7-O7	1.234(3)	C24-H24	0.9500
C7-N8	1.322(3)	C25-C26	1.374(4)
N8-C31	1.466(3)	C25-H25	0.9500
N8-H8	0.8800	C26-H26	0.9500
C8-O9	1.351(4)	C23-N27B	1.447(9)
C8-H8A	0.9500	C23-N27	1.484(12)

N27-O29	1.264(11)	C15-C14-C13	120.3(3)
N27-O28	1.273(12)	C15-C14-H14	119.8
N27B-O29B	1.258(10)	C13-C14-H14	119.8
N27B-O28B	1.267(10)	C14-C15-C16	120.2(3)
C31-C32	1.509(4)	C14-C15-H15	119.9
C31-C36	1.523(4)	C16-C15-H15	119.9
C31-H31	1.0000	C15-C16-C11	120.9(3)
C32-C33	1.520(4)	C15-C16-H16	119.6
C32-H32A	0.9900	C11-C16-H16	119.6
C32-H32B	0.9900	C22-C21-C26	118.7(3)
C33-C34	1.513(4)	C22-C21-C6	122.3(2)
C33-H33A	0.9900	C26-C21-C6	118.9(2)
C33-H33B	0.9900	C21-C22-C23	118.7(3)
C34-C35	1.510(4)	C21-C22-H22	120.6
C34-H34A	0.9900	C23-C22-H22	120.6
C34-H34B	0.9900	C25-C24-C23	118.2(3)
C35-C36	1.522(4)	C25-C24-H24	120.9
C35-H35A	0.9900	C23-C24-H24	120.9
C35-H35B	0.9900	C24-C25-C26	120.8(3)
C36-H36A	0.9900	C24-C25-H25	119.6
C36-H36B	0.9900	C26-C25-H25	119.6
O1-C1-N2	121.3(2)	C25-C26-C21	121.0(3)
O1-C1-C11	118.5(2)	C25-C26-H26	119.5
N2-C1-C11	120.3(2)	C21-C26-H26	119.5
C1-N2-C6	118.2(2)	C24-C23-C22	122.5(3)
C1-N2-C3	122.5(2)	C24-C23-N27B	116.3(5)
C6-N2-C3	119.3(2)	C22-C23-N27B	121.1(5)
N2-C3-C4	108.4(2)	C24-C23-N27	123.0(7)
N2-C3-H3A	110.0	C22-C23-N27	114.5(7)
C4-C3-H3A	110.0	O29-N27-O28	124.9(13)
N2-C3-H3B	110.0	O29-N27-C23	121.6(10)
C4-C3-H3B	110.0	O28-N27-C23	113.4(11)
H3A-C3-H3B	108.4	O29B-N27B-O28B	127.4(11)
C8-C4-C5	104.1(3)	O29B-N27B-C23	115.3(8)
C8-C4-C3	135.7(3)	O28B-N27B-C23	117.1(9)
C5-C4-C3	120.2(2)	N8-C31-C32	111.3(2)
N6-C5-C4	112.2(3)	N8-C31-C36	108.5(2)
N6-C5-C12	123.4(3)	C32-C31-C36	110.9(2)
C4-C5-C12	124.4(2)	N8-C31-H31	108.7
C5-N6-O9	104.6(2)	C32-C31-H31	108.7
N2-C6-C21	111.46(19)	C36-C31-H31	108.7
N2-C6-C7	110.77(19)	C31-C32-C33	112.0(2)
C21-C6-C7	111.1(2)	C31-C32-H32A	109.2
N2-C6-H6	107.8	C33-C32-H32A	109.2
C21-C6-H6	107.8	C31-C32-H32B	109.2
C7-C6-H6	107.8	C33-C32-H32B	109.2
O7-C7-N8	124.7(2)	H32A-C32-H32B	107.9
O7-C7-C6	120.0(2)	C34-C33-C32	111.0(2)
N8-C7-C6	115.2(2)	C34-C33-H33A	109.4
C7-N8-C31	124.5(2)	C32-C33-H33A	109.4
C7-N8-H8	117.7	C34-C33-H33B	109.4
C31-N8-H8	117.7	C32-C33-H33B	109.4
C4-C8-O9	110.1(3)	H33A-C33-H33B	108.0
C4-C8-H8A	124.9	C35-C34-C33	111.0(2)
O9-C8-H8A	124.9	C35-C34-H34A	109.4
C8-O9-N6	109.0(2)	C33-C34-H34A	109.4
C16-C11-C12	118.7(3)	C35-C34-H34B	109.4
C16-C11-C1	115.0(3)	C33-C34-H34B	109.4
C12-C11-C1	125.9(2)	H34A-C34-H34B	108.0
C13-C12-C11	119.3(3)	C34-C35-C36	111.4(2)
C13-C12-C5	119.1(3)	C34-C35-H35A	109.3
C11-C12-C5	121.6(2)	C36-C35-H35A	109.3
C14-C13-C12	120.5(3)	C34-C35-H35B	109.3
C14-C13-H13	119.8	C36-C35-H35B	109.3
C12-C13-H13	119.8	H35A-C35-H35B	108.0

C35-C36-C31	111.8(2)
C35-C36-H36A	109.3
C31-C36-H36A	109.3
C35-C36-H36B	109.3
C31-C36-H36B	109.3
H36A-C36-H36B	107.9


C35
C34
C36
C33
C31
C32
N8
O7
C7
O1
C1
C16
C15
C11
N2
C6
C21
C22
O29
C3
C26
N27
C14
C12
C4
C13
C5
C25
C24
O28
C8
N6
O9





Vorschlag für eine stichwortartige Experimentbeschreibung
(suggestion for a short experimental part):

sba138: colourless crystal (needle), dimensions 0.610 x 0.060 x 0.030 mm^3, crystal system monoclinic, space group $P2_1/c$, Z=4, a=15.0879(14) Å, b=17.9838(17) Å, c=8.8566(9) Å, alpha=90 deg, beta=103.191(3) deg, gamma=90 deg, V=2339.7(4) $Å^3$, rho=1.307 g/cm^3, T=200(2) K, $Theta_{max}$= 25.060 deg, radiation Mo Kalpha, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.47and a completeness of 99.9% to a resolution of 0.84 Å, 14592 reflections measured, 4147 unique (R(int)=0.0406), 2754 observed ($I > 2\sigma(I)$), intensities were corrected for Lorentz and polarization effects, an empirical scaling and absorption correction was applied using SADABS[1] based on the Laue symmetry of the reciprocal space, mu=0.09mm^{-1}, T_{min}=0.85, T_{max}=0.96, structure refined against F^2 with a Full-matrix least-squares algorithm using the SHELXL-2014/7 (Sheldrick, 2014) software [2], 335 parameters refined, hydrogen atoms were treated using appropriate riding models, goodness of fit 1.09 for observed reflections, final residual values R1(F)=0.055, $wR(F^2)$=0.146 for observed reflections, residual electron density -0.21 to 0.18 $eÅ^{-3}$. CCDC ..... contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2014/5 for absorption correction)
G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2014

Lit. 2: (program SHELXL-2014/7 (Sheldrick, 2014) for structure refinement)
Acta Cryst. (2015). C71, 3-8

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus:
Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.

Crystallographic data for compound 9e

Tabelle 1: Kristalldaten und Strukturverfeinerung für sba139

Strukturkennzeichen	sba139	
Summenformel	$C_{25}H_{24}N_4O_5$	
Molmasse	460.48	
Temperatur	100(2) K	
Wellenlänge	1.54178 Å	
Kristallsystem	triklin	
Raumgruppe	$P\bar{1}$	
Z	2	
Gitterkonstanten	a = 8.6689(3) Å	α = 77.844(3) °
	b = 10.6916(4) Å	β = 81.090(3) °
	c = 13.0284(4) Å	γ = 71.413(3) °
Zellvolumen	1113.85(7) $Å^3$	
Dichte (berechnet)	1.373 g/cm^3	
Absorptionskoeffizient µ	0.804 mm^{-1}	
Kristallform	polyhedron	
Kristallgröße	0.070 x 0.070 x 0.060 mm^3	
Kristallfarbe	colourless	
Gemessener Theta-Bereich	3.486 bis 72.124 °	
Indexgrenzen	$-8\le h\le 10$, $-13\le k\le 13$, $-13\le l\le 16$	
Gemessene Reflexe	13869	
Unabhängige Reflexe	4256 (R(int) = 0.0376)	
Beobachtete Reflexe	2864 ($I > 2\sigma(I)$)	
Absorptionskorrektur	Semi-empirical from equivalents	
Max/min Transmission	1.39 and 0.70	
Strukturverfeinerung	Full-matrix least-squares an F^2	
Daten/Restraints/Parameter	4256 / 0 / 307	
Goodness-of-fit an F^2	0.85	
R-Werte (I>2sigma(I))	R1 = 0.035, wR2 = 0.078	
Extinktionskoeffizient	n/a	
Max/min Restelektronendichte	0.15 und -0.24 $eÅ^{-3}$	

Table 2: Crystal data and structure refinement for sba139.

Identification code	sba139	
Empirical formula	$C_{25}H_{24}N_4O_5$	
Formula weight	460.48	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	triclinic	
Space group	$P\bar{1}$	
Z	2	
Unit cell dimensions	a = 8.6689(3) Å	α = 77.844(3) deg.
	b = 10.6916(4) Å	β = 81.090(3) deg.
	c = 13.0284(4) Å	γ = 71.413(3) deg.
Volume	1113.85(7) $Å^3$	
Density (calculated)	1.37 g/cm^3	
Absorption coefficient	0.80 mm^{-1}	
Crystal shape	polyhedron	
Crystal size	0.070 x 0.070 x 0.060 mm^3	
Crystal colour	colourless	
Theta range for data collection	3.5 to 72.1 deg.	
Index ranges	$-8\le h\le 10$, $-13\le k\le 13$, $-13\le l\le 16$	
Reflections collected	13869	
Independent reflections	4256 (R(int) = 0.0376)	

Observed reflections	2864 ($I > 2\sigma(I)$)
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.39 and 0.70
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4256 / 0 / 307
Goodness-of-fit on F^2	0.85
Final R indices (I>2sigma(I))	R1 = 0.035, wR2 = 0.078
Largest diff. peak and hole	0.15 and -0.24 $eÅ^{-3}$

Tabelle 3: Atomkoordinaten und äquivalente isotrope Auslenkungsparameter ($Å^2$) für sba139. U_{eq} wird berechnet als ein Drittel der Spur des orthogonalen U_{ij} Tensors. (Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for sba139. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.)

Atom	x	y	z	U_{eq}
C1	0.7785(2)	0.5073(1)	0.3869(1)	0.0238(3)
O1	0.7179(1)	0.4676(1)	0.4755(1)	0.0262(2)
N2	0.6944(2)	0.6194(1)	0.3254(1)	0.0234(3)
C3	0.7719(2)	0.6788(2)	0.2270(1)	0.0266(3)
H3A	0.8800	0.6831	0.2390	0.032
H3B	0.7030	0.7711	0.2030	0.032
C4	0.7917(2)	0.5950(2)	0.1455(1)	0.0275(3)
C5	0.8908(2)	0.4592(2)	0.1585(1)	0.0271(3)
N6	0.8867(2)	0.4000(1)	0.0808(1)	0.0339(3)
C6	0.5177(2)	0.6729(1)	0.3572(1)	0.0228(3)
H6	0.4820	0.5976	0.4032	0.027
C7	0.4877(2)	0.7825(1)	0.4241(1)	0.0224(3)
O7	0.5518(1)	0.8733(1)	0.3956(1)	0.0291(2)
N8	0.3878(2)	0.7683(1)	0.5110(1)	0.0254(3)
H8	0.3495	0.6991	0.5227	0.030
C8	0.7264(2)	0.6133(2)	0.0545(1)	0.0337(4)
H8A	0.6527	0.6945	0.0237	0.040
O9	0.7803(2)	0.4993(1)	0.0131(1)	0.0376(3)
C11	0.9452(2)	0.4226(1)	0.3487(1)	0.0246(3)
C12	0.9909(2)	0.3905(1)	0.2467(1)	0.0267(3)
C13	1.1353(2)	0.2880(2)	0.2286(1)	0.0310(4)
H13	1.1661	0.2653	0.1601	0.037
C14	1.2329(2)	0.2199(2)	0.3084(1)	0.0340(4)
H14	1.3291	0.1494	0.2950	0.041
C15	1.1917(2)	0.2535(2)	0.4084(1)	0.0332(4)
H15	1.2616	0.2091	0.4627	0.040
C16	1.0474(2)	0.3525(2)	0.4281(1)	0.0290(4)
H16	1.0173	0.3731	0.4973	0.035
C21	0.4152(2)	0.7220(1)	0.2645(1)	0.0243(3)
C22	0.3956(2)	0.8484(2)	0.2038(1)	0.0257(3)
H22	0.4455	0.9081	0.2203	0.031
C23	0.3027(2)	0.8865(2)	0.1191(1)	0.0293(3)
C24	0.2233(2)	0.8051(2)	0.0936(1)	0.0337(4)
H24	0.1592	0.8340	0.0353	0.040
C25	0.2403(2)	0.6805(2)	0.1555(1)	0.0345(4)
H25	0.1865	0.6228	0.1402	0.041
C26	0.3353(2)	0.6388(2)	0.2400(1)	0.0295(4)
H26	0.3461	0.5526	0.2817	0.035
N27	0.2909(2)	1.0187(1)	0.0540(1)	0.0338(3)
O28	0.3753(2)	1.0832(1)	0.0727(1)	0.0371(3)
O29	0.1989(2)	1.0585(1)	-0.0163(1)	0.0512(4)
C31	0.3358(2)	0.8578(1)	0.5892(1)	0.0247(3)
H31	0.4049	0.9199	0.5750	0.030
C32	0.1573(2)	0.9402(2)	0.5813(1)	0.0286(3)

H32A	0.0895	0.8792	0.5893	0.034
H32B	0.1454	0.9971	0.5107	0.034
C33	0.0956(2)	1.0293(2)	0.6661(1)	0.0326(4)
H33A	0.1543	1.0976	0.6530	0.039
H33B	-0.0223	1.0765	0.6617	0.039
C34	0.1217(2)	0.9474(2)	0.7758(1)	0.0347(4)
H34A	0.0515	0.8873	0.7925	0.042
H34B	0.0890	1.0083	0.8282	0.042
C35	0.2997(2)	0.8642(2)	0.7837(1)	0.0334(4)
H35A	0.3115	0.8075	0.8544	0.040
H35B	0.3684	0.9246	0.7753	0.040
C36	0.3591(2)	0.7750(2)	0.6992(1)	0.0274(3)
H36A	0.4764	0.7255	0.7042	0.033
H36B	0.2976	0.7086	0.7118	0.033

Tabelle 4: H-Atomkoordinaten und isotrope Auslenkungsparameter ($Å^2$) für sba139.
(Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for sba139.)

Atom	x	y	z	U_{eq}
H3A	0.8800	0.6831	0.2390	0.032
H3B	0.7030	0.7711	0.2030	0.032
H6	0.4820	0.5976	0.4032	0.027
H8	0.3495	0.6991	0.5227	0.030
H8A	0.6527	0.6945	0.0237	0.040
H13	1.1661	0.2653	0.1601	0.037
H14	1.3291	0.1494	0.2950	0.041
H15	1.2616	0.2091	0.4627	0.040
H16	1.0173	0.3731	0.4973	0.035
H22	0.4455	0.9081	0.2203	0.031
H24	0.1592	0.8340	0.0353	0.040
H25	0.1865	0.6228	0.1402	0.041
H26	0.3461	0.5526	0.2817	0.035
H31	0.4049	0.9199	0.5750	0.030
H32A	0.0895	0.8792	0.5893	0.034
H32B	0.1454	0.9971	0.5107	0.034
H33A	0.1543	1.0976	0.6530	0.039
H33B	-0.0223	1.0765	0.6617	0.039
H34A	0.0515	0.8873	0.7925	0.042
H34B	0.0890	1.0083	0.8282	0.042
H35A	0.3115	0.8075	0.8544	0.040
H35B	0.3684	0.9246	0.7753	0.040
H36A	0.4764	0.7255	0.7042	0.033
H36B	0.2976	0.7086	0.7118	0.033

Tabelle 5: Anisotrope Auslenkungsparameter ($Å^2$) für sba139. Der Exponent für den anisotropen Auslenkungsparameter hat die Form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$
(Anisotropic displacement parameters ($Å^2$) for sba139. The anisotropic displacement factor exponent takes the form: $-2\,pi^2\,(h^2\,a^{*2}\,U_{11} + ... + 2\,h\,k\,a^*\,b^*\,U_{12})$)

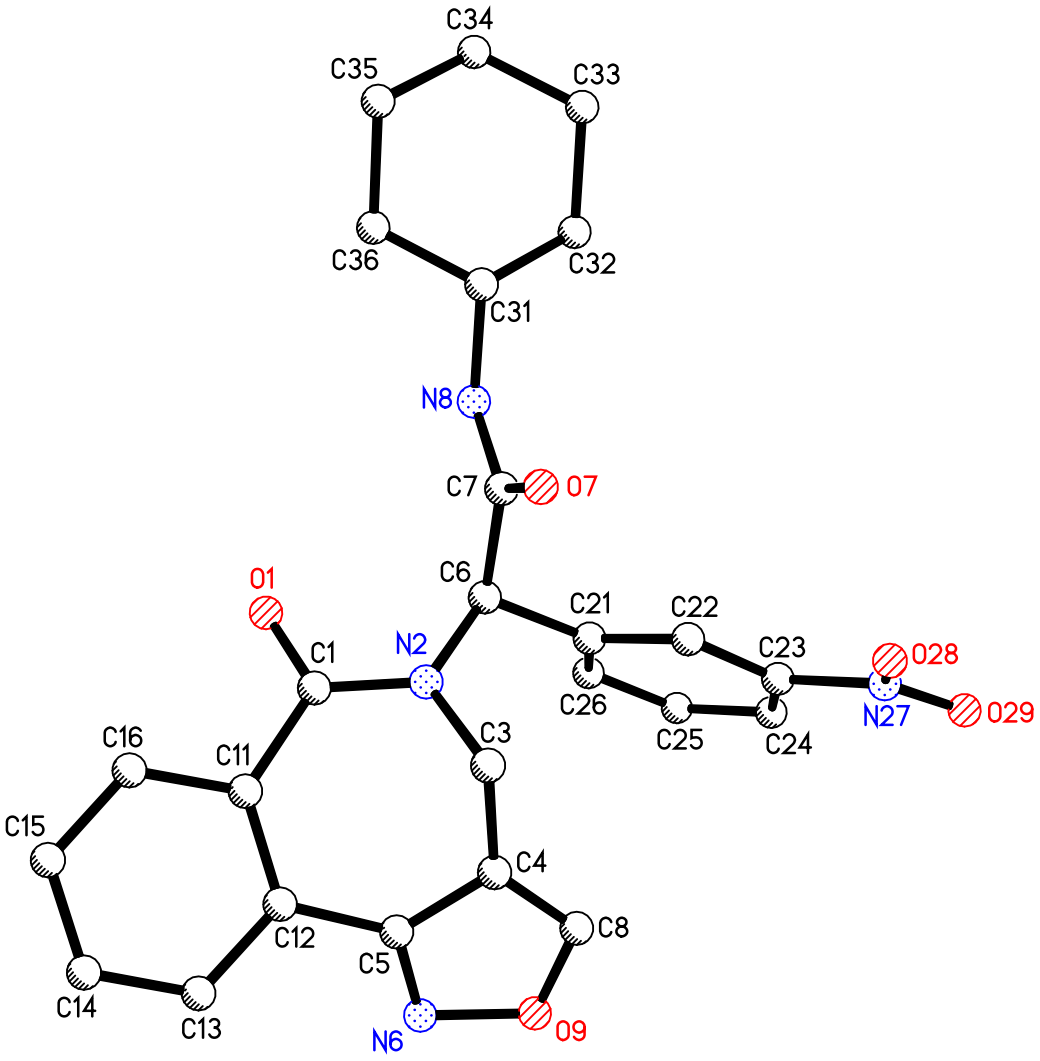
Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0281(8)	0.0201(7)	0.0252(8)	-0.0078(6)	-0.0007(7)	-0.0081(6)
O1	0.0284(6)	0.0236(5)	0.0257(6)	-0.0049(4)	0.0008(5)	-0.0074(4)
N2	0.0232(6)	0.0197(6)	0.0248(6)	-0.0053(5)	0.0030(5)	-0.0045(5)

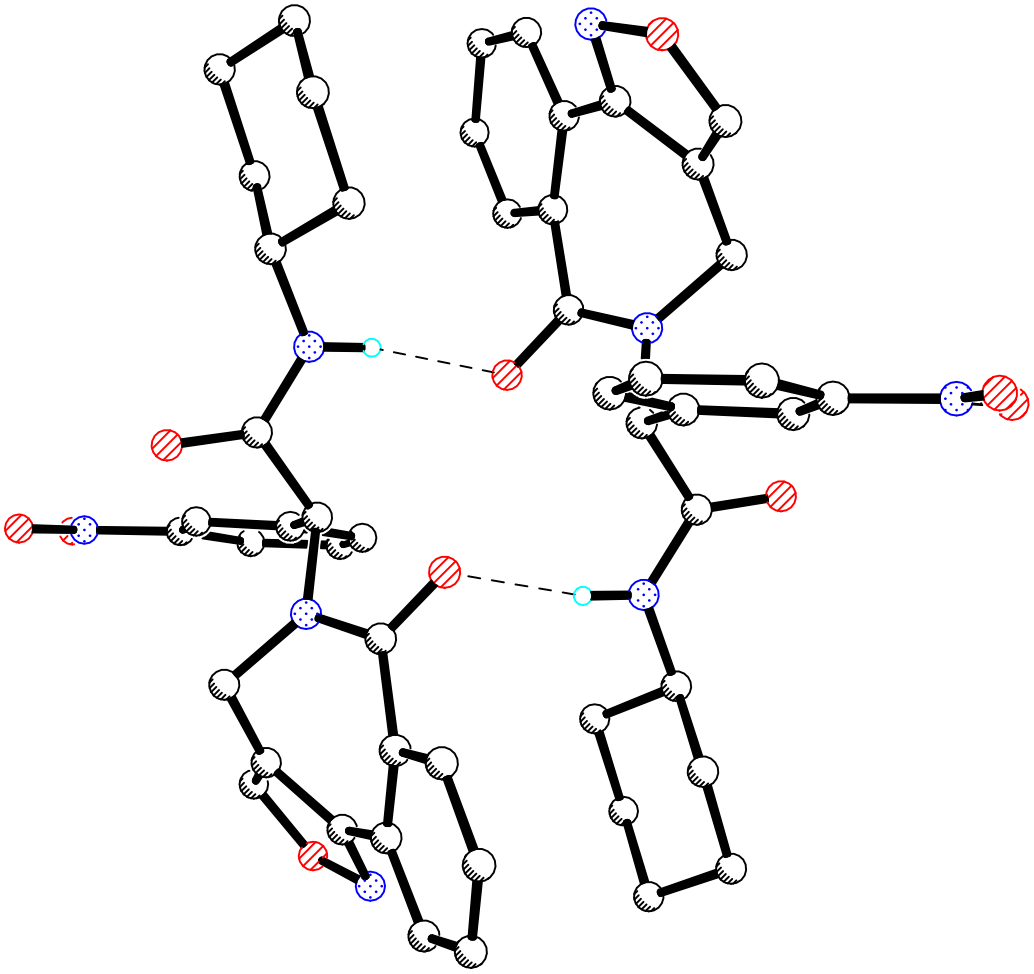
C3	0.0279(8)	0.0201(7)	0.0288(8)	-0.0040(6)	0.0047(7)	-0.0068(6)
C4	0.0303(8)	0.0240(8)	0.0258(8)	-0.0051(6)	0.0055(7)	-0.0081(7)
C5	0.0292(8)	0.0238(8)	0.0273(8)	-0.0070(6)	0.0049(7)	-0.0083(6)
N6	0.0418(8)	0.0278(7)	0.0297(7)	-0.0076(6)	0.0010(6)	-0.0075(6)
C6	0.0226(8)	0.0205(7)	0.0238(7)	-0.0067(6)	0.0017(6)	-0.0044(6)
C7	0.0234(8)	0.0195(7)	0.0230(7)	-0.0050(6)	-0.0025(6)	-0.0033(6)
O7	0.0360(6)	0.0264(6)	0.0288(5)	-0.0091(4)	0.0034(5)	-0.0147(5)
N8	0.0307(7)	0.0212(6)	0.0258(7)	-0.0100(5)	0.0049(6)	-0.0096(5)
C8	0.0404(10)	0.0270(8)	0.0288(9)	-0.0062(7)	0.0046(7)	-0.0062(7)
O9	0.0506(7)	0.0329(6)	0.0269(6)	-0.0089(5)	-0.0010(5)	-0.0081(5)
C11	0.0252(8)	0.0189(7)	0.0307(8)	-0.0062(6)	0.0003(7)	-0.0077(6)
C12	0.0263(8)	0.0210(7)	0.0330(8)	-0.0075(6)	0.0038(7)	-0.0088(6)
C13	0.0279(9)	0.0263(8)	0.0383(9)	-0.0117(7)	0.0060(7)	-0.0075(7)
C14	0.0230(8)	0.0246(8)	0.0524(11)	-0.0120(7)	0.0010(8)	-0.0031(7)
C15	0.0267(9)	0.0269(8)	0.0462(10)	-0.0068(7)	-0.0067(8)	-0.0065(7)
C16	0.0295(9)	0.0244(8)	0.0354(9)	-0.0086(7)	-0.0034(7)	-0.0085(7)
C21	0.0232(8)	0.0238(8)	0.0257(8)	-0.0116(6)	0.0022(6)	-0.0040(6)
C22	0.0254(8)	0.0244(8)	0.0256(8)	-0.0107(6)	0.0012(6)	-0.0027(6)
C23	0.0292(8)	0.0284(8)	0.0258(8)	-0.0095(6)	-0.0005(7)	-0.0002(7)
C24	0.0285(9)	0.0445(10)	0.0278(8)	-0.0153(7)	-0.0029(7)	-0.0042(7)
C25	0.0322(9)	0.0424(10)	0.0343(9)	-0.0186(8)	0.0008(7)	-0.0129(8)
C26	0.0315(9)	0.0285(8)	0.0301(8)	-0.0137(7)	0.0030(7)	-0.0085(7)
N27	0.0369(8)	0.0317(7)	0.0248(7)	-0.0081(6)	-0.0023(6)	0.0026(6)
O28	0.0453(7)	0.0258(6)	0.0358(6)	-0.0067(5)	-0.0032(5)	-0.0039(5)
O29	0.0606(9)	0.0493(8)	0.0357(7)	-0.0014(6)	-0.0205(7)	-0.0017(6)
C31	0.0275(8)	0.0225(7)	0.0265(8)	-0.0119(6)	0.0021(6)	-0.0079(6)
C32	0.0288(8)	0.0255(8)	0.0328(8)	-0.0094(6)	-0.0033(7)	-0.0067(7)
C33	0.0260(8)	0.0291(8)	0.0427(10)	-0.0168(7)	0.0015(7)	-0.0037(7)
C34	0.0318(9)	0.0405(10)	0.0354(9)	-0.0211(7)	0.0079(7)	-0.0118(7)
C35	0.0352(9)	0.0395(9)	0.0274(8)	-0.0113(7)	-0.0004(7)	-0.0113(8)
C36	0.0257(8)	0.0274(8)	0.0297(8)	-0.0081(6)	-0.0006(7)	-0.0074(7)

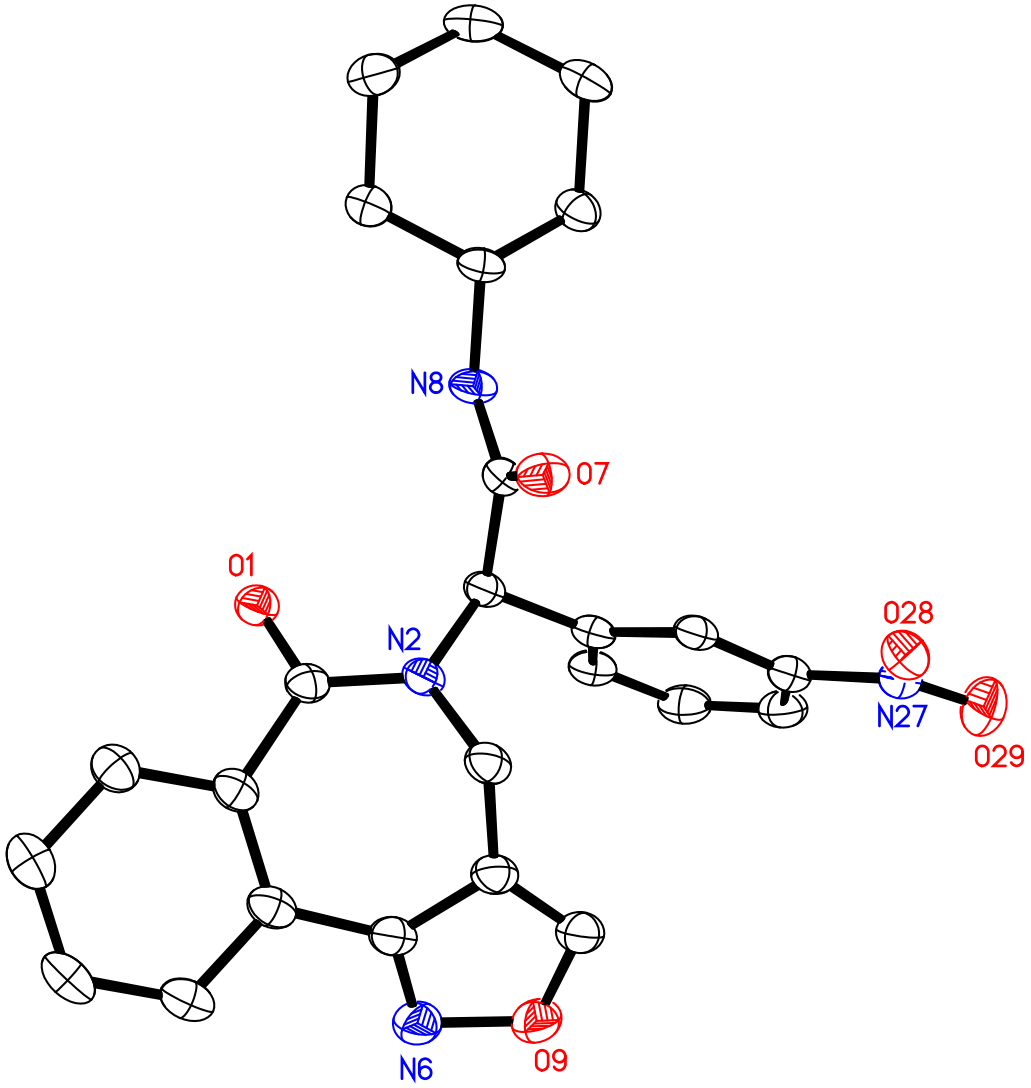
Tabelle 6: Bindungslängen (Å) und -winkel (°) für sba139.
(Bond lengths (Å) and angles (deg) for sba139.)

C1-O1	1.2400(18)	C14-C15	1.387(2)
C1-N2	1.3522(19)	C14-H14	0.9500
C1-C11	1.513(2)	C15-C16	1.387(2)
N2-C3	1.4700(19)	C15-H15	0.9500
N2-C6	1.4803(19)	C16-H16	0.9500
C3-C4	1.485(2)	C21-C22	1.388(2)
C3-H3A	0.9900	C21-C26	1.398(2)
C3-H3B	0.9900	C22-C23	1.383(2)
C4-C8	1.342(2)	C22-H22	0.9500
C4-C5	1.422(2)	C23-C24	1.384(2)
C5-N6	1.313(2)	C23-N27	1.469(2)
C5-C12	1.469(2)	C24-C25	1.381(3)
N6-O9	1.4104(18)	C24-H24	0.9500
C6-C21	1.514(2)	C25-C26	1.389(2)
C6-C7	1.5375(19)	C25-H25	0.9500
C6-H6	1.0000	C26-H26	0.9500
C7-O7	1.2324(17)	N27-O29	1.2256(18)
C7-N8	1.3273(19)	N27-O28	1.2319(18)
N8-C31	1.4667(18)	C31-C36	1.524(2)
N8-H8	0.8800	C31-C32	1.524(2)
C8-O9	1.3532(19)	C31-H31	1.0000
C8-H8A	0.9500	C32-C33	1.532(2)
C11-C16	1.396(2)	C32-H32A	0.9900
C11-C12	1.409(2)	C32-H32B	0.9900
C12-C13	1.402(2)	C33-C34	1.519(2)
C13-C14	1.374(2)	C33-H33A	0.9900
C13-H13	0.9500	C33-H33B	0.9900

C34-C35	1.523(2)
C34-H34A	0.9900
C34-H34B	0.9900
C35-C36	1.529(2)
C35-H35A	0.9900
C35-H35B	0.9900
C36-H36A	0.9900
C36-H36B	0.9900
O1-C1-N2	120.67(13)
O1-C1-C11	117.61(13)
N2-C1-C11	121.66(13)
C1-N2-C3	121.52(12)
C1-N2-C6	116.82(12)
C3-N2-C6	121.48(12)
N2-C3-C4	108.88(12)
N2-C3-H3A	109.9
C4-C3-H3A	109.9
N2-C3-H3B	109.9
C4-C3-H3B	109.9
H3A-C3-H3B	108.3
C8-C4-C5	103.90(13)
C8-C4-C3	134.60(15)
C5-C4-C3	121.37(14)
N6-C5-C4	112.40(14)
N6-C5-C12	122.46(13)
C4-C5-C12	125.12(13)
C5-N6-O9	104.66(12)
N2-C6-C21	112.91(11)
N2-C6-C7	110.41(11)
C21-C6-C7	111.53(11)
N2-C6-H6	107.2
C21-C6-H6	107.2
C7-C6-H6	107.2
O7-C7-N8	125.65(13)
O7-C7-C6	120.91(13)
N8-C7-C6	113.44(12)
C7-N8-C31	125.71(12)
C7-N8-H8	117.1
C31-N8-H8	117.1
C4-C8-O9	110.29(14)
C4-C8-H8A	124.9
O9-C8-H8A	124.9
C8-O9-N6	108.75(11)
C16-C11-C12	118.62(14)
C16-C11-C1	114.88(13)
C12-C11-C1	125.35(13)
C13-C12-C11	119.12(14)
C13-C12-C5	118.61(14)
C11-C12-C5	122.26(13)
C14-C13-C12	121.03(15)
C14-C13-H13	119.5
C12-C13-H13	119.5
C13-C14-C15	120.30(14)
C13-C14-H14	119.9
C15-C14-H14	119.9
C16-C15-C14	119.34(15)
C16-C15-H15	120.3
C14-C15-H15	120.3
C15-C16-C11	121.54(15)
C15-C16-H16	119.2
C11-C16-H16	119.2
C22-C21-C26	118.68(14)
C22-C21-C6	122.04(13)
C26-C21-C6	119.27(13)
C23-C22-C21	119.24(14)
C23-C22-H22	120.4
C21-C22-H22	120.4
C22-C23-C24	122.72(15)
C22-C23-N27	117.53(14)
C24-C23-N27	119.74(14)
C25-C24-C23	117.85(15)
C25-C24-H24	121.1
C23-C24-H24	121.1
C24-C25-C26	120.61(15)
C24-C25-H25	119.7
C26-C25-H25	119.7
C25-C26-C21	120.86(15)
C25-C26-H26	119.6
C21-C26-H26	119.6
O29-N27-O28	123.52(14)
O29-N27-C23	118.42(14)
O28-N27-C23	118.05(13)
N8-C31-C36	109.39(12)
N8-C31-C32	110.15(12)
C36-C31-C32	110.35(13)
N8-C31-H31	109.0
C36-C31-H31	109.0
C32-C31-H31	109.0
C31-C32-C33	111.67(12)
C31-C32-H32A	109.3
C33-C32-H32A	109.3
C31-C32-H32B	109.3
C33-C32-H32B	109.3
H32A-C32-H32B	107.9
C34-C33-C32	111.28(13)
C34-C33-H33A	109.4
C32-C33-H33A	109.4
C34-C33-H33B	109.4
C32-C33-H33B	109.4
H33A-C33-H33B	108.0
C33-C34-C35	111.24(13)
C33-C34-H34A	109.4
C35-C34-H34A	109.4
C33-C34-H34B	109.4
C35-C34-H34B	109.4
H34A-C34-H34B	108.0
C34-C35-C36	111.29(13)
C34-C35-H35A	109.4
C36-C35-H35A	109.4
C34-C35-H35B	109.4
C36-C35-H35B	109.4
H35A-C35-H35B	108.0
C31-C36-C35	111.10(13)
C31-C36-H36A	109.4
C35-C36-H36A	109.4
C31-C36-H36B	109.4
C35-C36-H36B	109.4
H36A-C36-H36B	108.0


C34
C35
C33
C36
C32
C31
N8
C7
O7
O1
C6
C21
C22
C1
N2
C23
O28
C26
N27
O29
C25
C24
C16
C11
C3
C15
C4
C8
C12
C5
C14
C13
N6
O9





Vorschlag für eine stichwortartige Experimentbeschreibung
(suggestion for a short experimental part):

sba139: colourless crystal (polyhedron), dimensions 0.070 x 0.070 x 0.060 mm^3, crystal system triclinic, space group $P\bar{1}$, Z=2, a=8.6689(3) Å, b=10.6916(4) Å, c=13.0284(4) Å, alpha=77.844(3) deg, beta=81.090(3) deg, gamma=71.413(3) deg, V=1113.85(7) $Å^3$, rho=1.373 g/cm^3, T=100(2) K, $Theta_{max}$= 72.124 deg, radiation Mo Kalpha, lambda=1.54178 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 3.15and a completeness of 96.8% to a resolution of 0.81 Å, 13869 reflections measured, 4256 unique (R(int)=0.0376), 2864 observed

(I > 2σ(I)), intensities were corrected for Lorentz and polarization effects, an empirical scaling and absorption correction was applied using STOE X-AREA Laue Analyzer based on the Laue symmetry of the mu=0.80mm^{-1}, T_{min}=0.70, T_{max}=1.39, structure refined against F^2 with a Full-matrix least-squares algorithm using the SHELXL-2014/7 (Sheldrick, 2014) software [2], 307 parameters refined, hydrogen atoms were treated using appropriate riding models, goodness of fit 0.85 for observed reflections, final residual values R1(F)=0.035, wR(F^2)=0.078 for observed reflections, residual electron density -0.24 to 0.15 eÅ^{-3}. CCDC ..... contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Lit. 1: (program SADABS 2014/5 for absorption correction)
G. M. Sheldrick, Bruker Analytical X-ray-Division, Madison, Wisconsin 2014

Lit. 2: (program SHELXL-2014/7 (Sheldrick, 2014) for structure refinement)
Acta Cryst. (2015). C71, 3-8

Lit. APEX, APEX2, SMART, SAINT, SAINT-Plus:
Bruker (2007). "Program name(s)". Bruker AXS Inc., Madison, Wisconsin, USA.