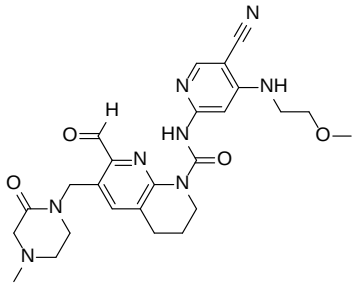
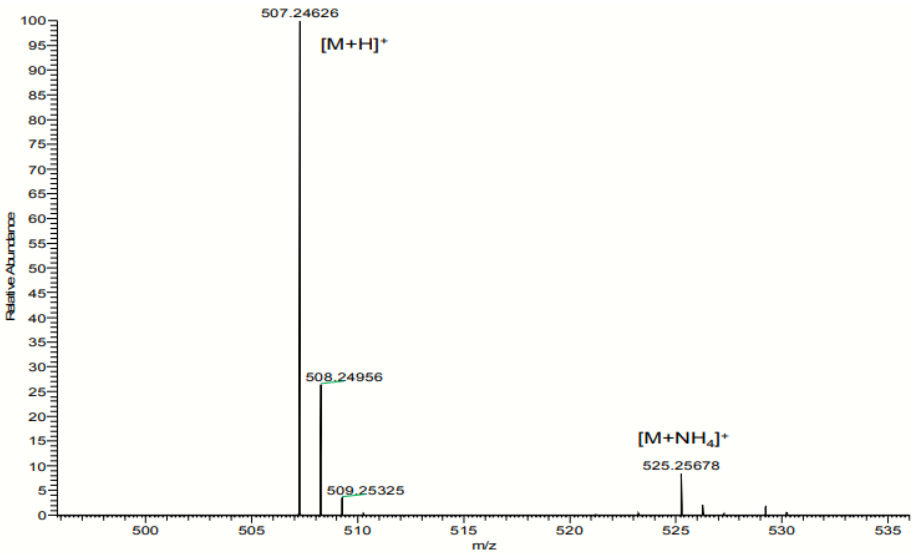
FGF401 characterizing data

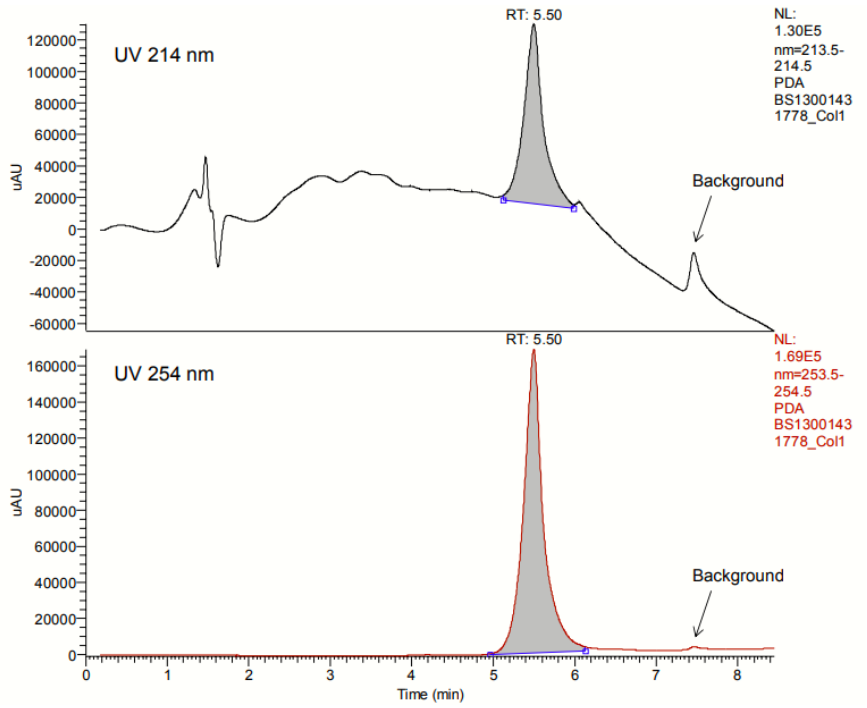


N-(5-cyano-4-((2-methoxyethyl)amino)pyridin-2-yl)-7-formyl-6-((4-methyl-2-oxopiperazin-1-yl)methyl)-3,4-dihydro-1,8-naphthyridine-1(2*H*)-carboxamide
Chemical Formula: $C_{25}H_{30}N_8O_4$
Molecular Weight: 506.57

HR-MS

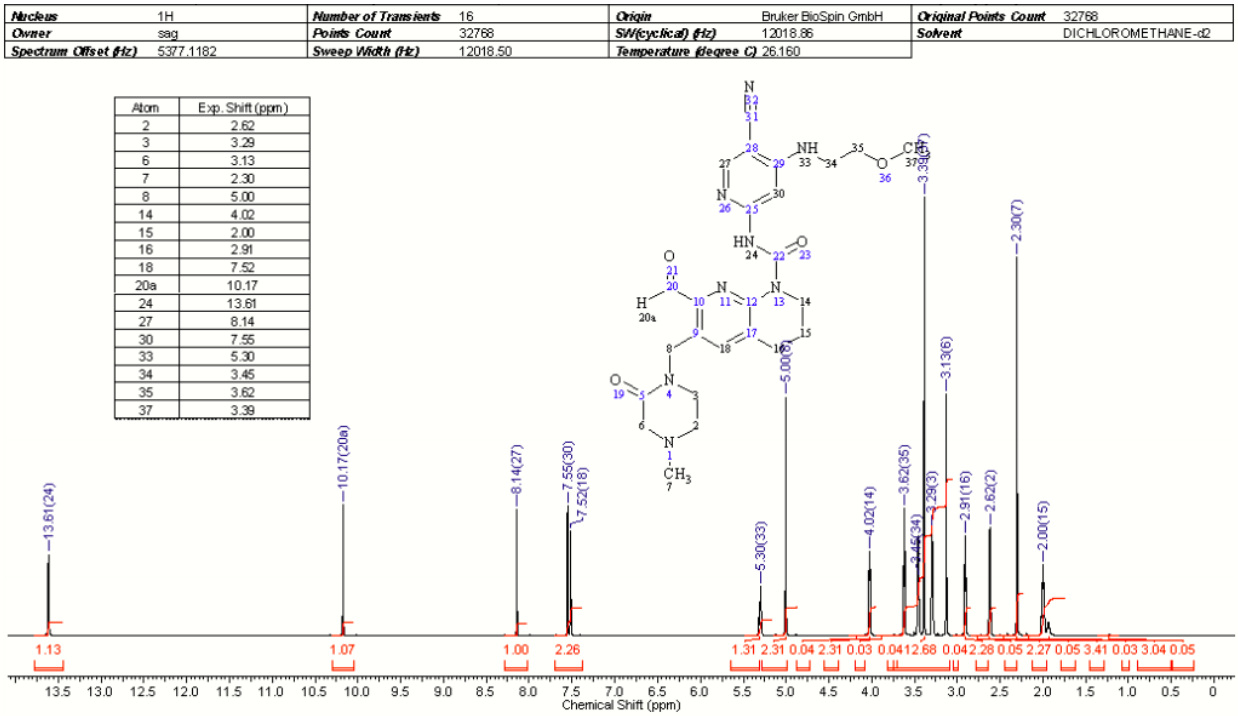
LC/ESI-MS and LC-UV data were recorded using a Thermo Scientific LTQ Orbitrap XL mass spectrometer with an electrospray ionization source and a Shimadzu Nexera liquid chromatograph equipped with a diode array detector. The instrument was lock mass calibrated with the ammonium-adduct ion of a polysiloxane derivative (m/z 536.16537). The accurate mass was obtained by averaging 15 scans at a mass resolution of ca 29500 (FWHM definition). The mass accuracy of the system has been found to be better than 1 ppm. The chromatography was performed at 100 µL/min flow rate (1 mm C_{18}-column) with a polar gradient from 5% to 100% acetonitrile in 8 min. 0.04% formic acid was used as the modifier additive in both mobile phases, in addition to 3.75 mM ammonium acetate in the aqueous phase. The UV traces at the two wavelengths 214 and 254 nm were extracted from the DAD chromatogram.





^{1}H NMR

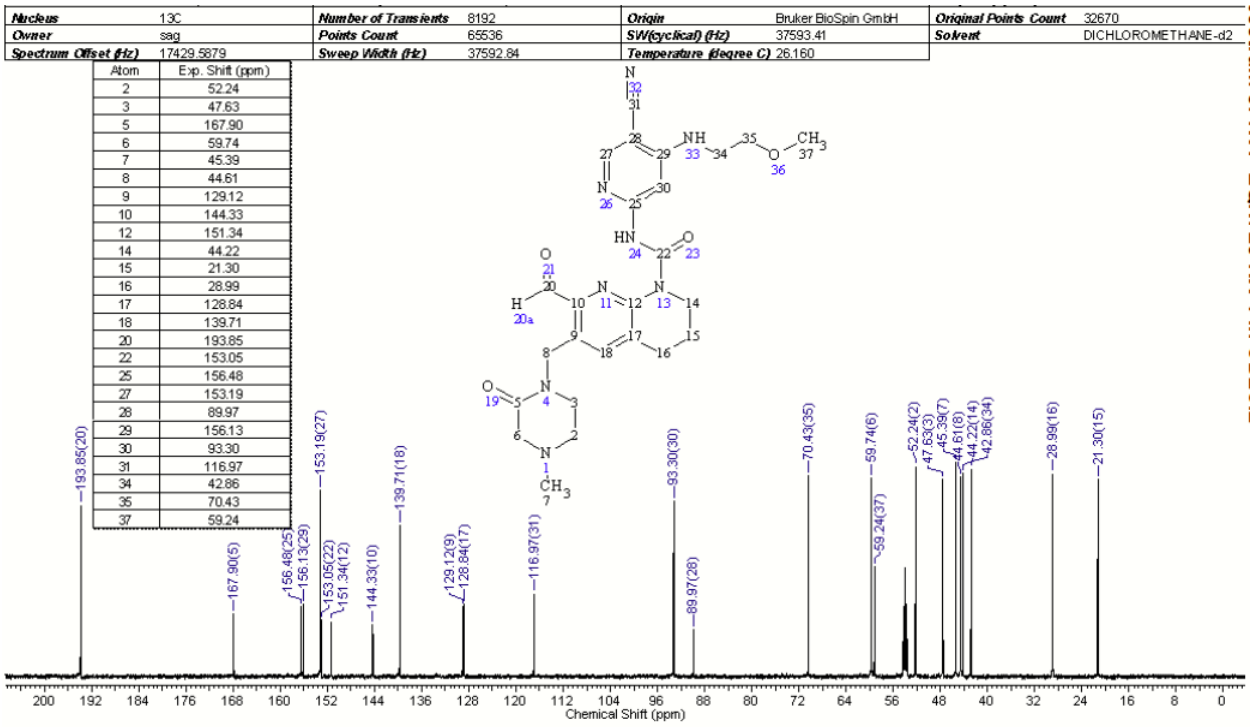
The NMR sample was prepared by dissolving approx 1.5 mg of FGF401 in ca 40 µl DMSO-d_6. The NMR spectra (^{1}H and ^{13}C) were measured at 26.2 ºC on a Bruker AVANCE spectrometer (600 MHz proton frequency) equipped with a 1.7 mm ^{1}H{^{13}C, ^{15}N} CryoProbe™. ^{1}H and ^{13}C shifts were referenced internally to the solvent (CD_2Cl_2) signals at 5.32 ppm and 54 ppm, respectively.



Nucleus	1H	Number of Transients	16	Origin	Bruker BioSpin GmbH	Original Points Count	32768
Owner	sag	Points Count	32768	SW(cyclical) (Hz)	12018.86	Solvent	DICHLOROMETHANE-d2
Spectrum Offset (Hz)	5377.1182	Sweep Width (Hz)	12018.50	Temperature (degree C)	26.160		

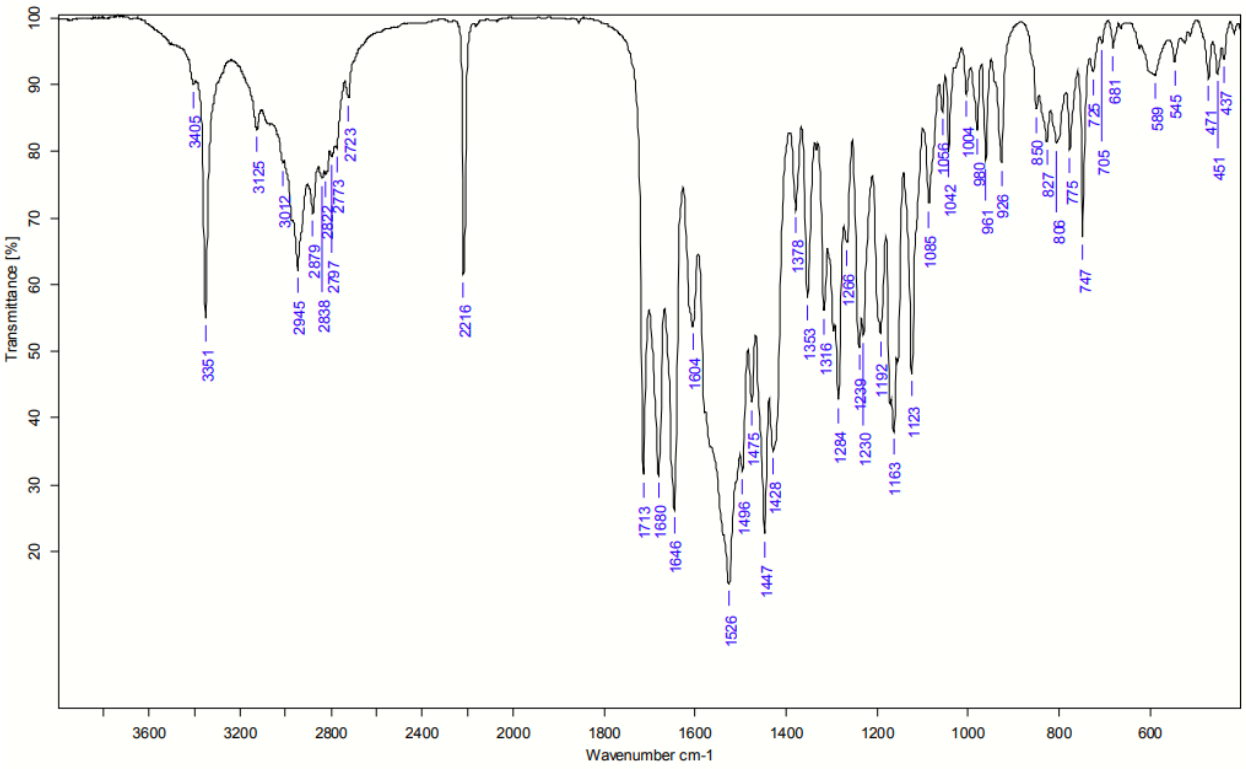
Atom	Exp. Shift (ppm)
2	2.62
3	3.29
6	3.13
7	2.30
8	5.00
14	4.02
15	2.00
16	2.91
18	7.52
20a	10.17
24	13.61
27	8.14
30	7.55
33	5.30
34	3.45
35	3.62
37	3.39

^{13}C NMR


Nucleus 13C
Number of Transients 8192
Origin Bruker BioSpin GmbH
Original Points Count 32670
Owner sag
Points Count 65536
SW(cyclical) (Hz) 37593.41
Solvent DICHLOROMETHANE-d2
Spectrum Offset (Hz) 17429.5879
Sweep Width (Hz) 37592.84
Temperature (degree C) 26.160
Atom | Exp. Shift (ppm)
2 | 52.24
3 | 47.63
5 | 167.90
6 | 59.74
7 | 45.39
8 | 44.61
9 | 129.12
10 | 144.33
12 | 151.34
14 | 44.22
15 | 21.30
16 | 28.99
17 | 128.84
18 | 139.71
20 | 193.85
22 | 153.05
25 | 156.48
27 | 153.19
28 | 89.97
29 | 156.13
30 | 93.30
31 | 116.97
34 | 42.86
35 | 70.43
37 | 59.24
193.85(20)
167.90(5)
156.48(25)
156.13(29)
153.19(27)
153.05(22)
151.34(12)
144.33(10)
139.71(18)
129.12(9)
128.84(17)
116.97(31)
93.30(30)
89.97(28)
70.43(35)
59.74(6)
59.24(37)
52.24(2)
47.63(3)
45.39(7)
44.61(8)
44.22(14)
42.86(34)
28.99(16)
21.30(15)
Chemical Shift (ppm)

IR spectrum

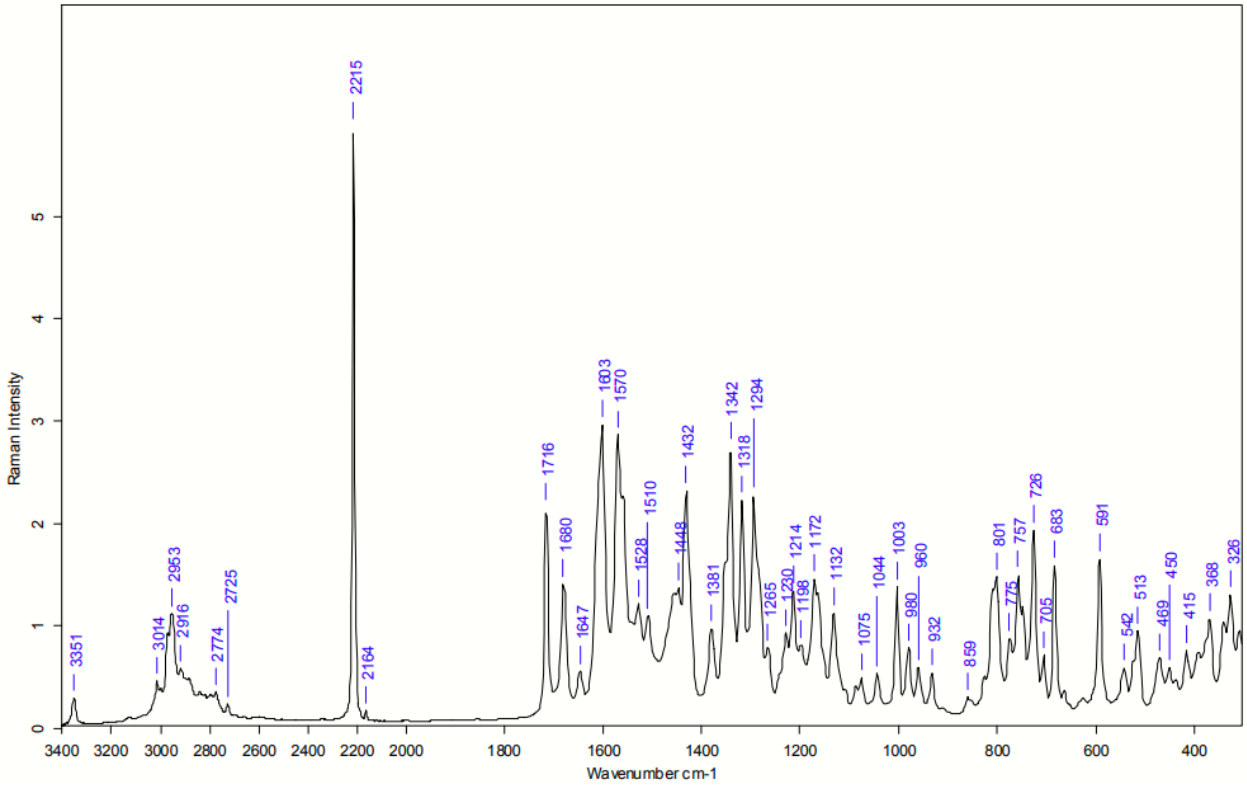
The infrared spectrum of FGF401 was acquired as a KBr pellet on a Vector 22 FT-IR spectrometer (Bruker Optics, Fällanden, Switzerland) over a wave number range of 4000-400 cm^{-1} with a resolution of 2 cm^{-1}.



Wave number (cm^{-1})	Assignments
3351, 3200 ~ 2700	NH stretching
3012	Aromatic C-H stretching
2945, 2879	Aliphatic C-H stretching
2838	Methoxy C-H stretching
2822, 2797	NC-H_3 and NC-H_2 stretching
2723	Aldehyde CH Fermi resonance
2216	C≡N stretching
1713	Aldehyde C=O stretching
1680	Urea C=O stretching
1646	Tertiary amide C=O stretching
1604, 1526, 1496, 1475, 1447	Aromatic rings stretching
1526	Includes Amide II (CON-H bending)
1428	Tertiary amide C-N stretching
1123	Ether C-O-C stretching

Raman spectrum

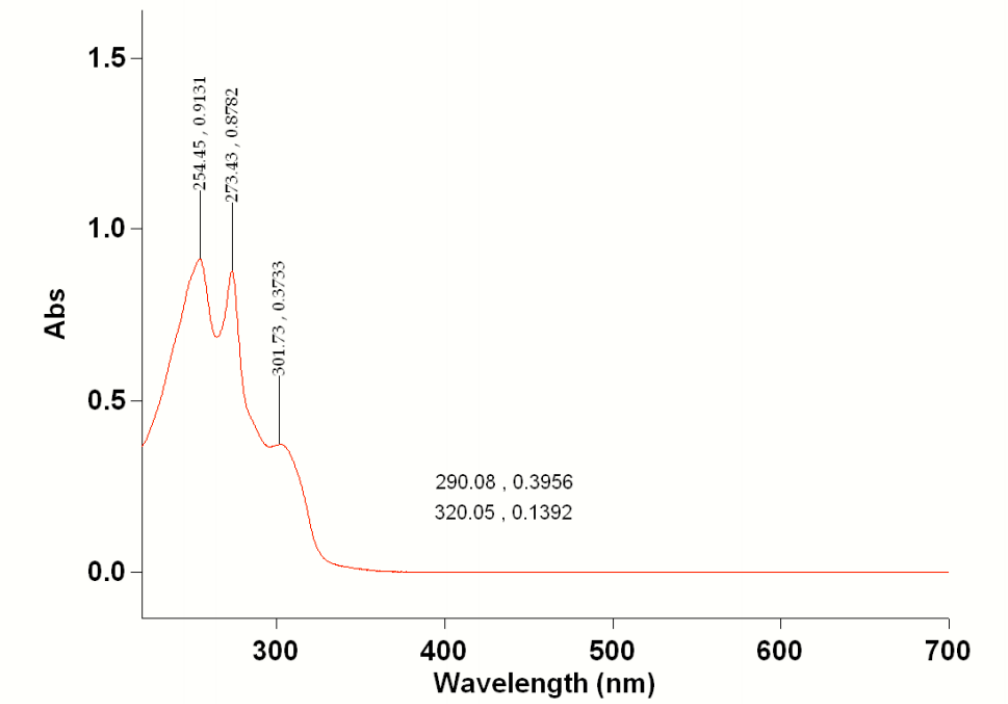
The FT Raman spectrum of solid FGF401 was recorded with a MultiRam Raman spectrometer (Bruker Optics, Fällanden, Switzerland) equipped with a liquid nitrogen cooled germanium detector. The resolution was 4 cm^{-1} and 160 scans were accumulated using a laser output of 400 mW. The spectrum was corrected for instrumental response.



Wave number (cm^{-1})	Assignments
3351	NH stretching
3014	Aromatic C-H stretching
2953, 2916	Aliphatic C-H stretching
2725	Aldehyde CH Fermi resonance
2215	C≡N stretching
1716	Aldehyde C=O stretching
1680	Urea C=O stretching
1647	Tertiary amide C=O stretching
1603, 1570, 1432	Aromatic rings stretching

UV spectrum

The ultraviolet-visible spectrum of FGF401 was acquired in methanol solution at a concentration of C=0,0116 g/L or 2,29.10^{-5}M over a wavelength range of 220 to 700 nm using a Cary 300 Scan spectrometer (Varian) and a UV cuvette cell of 1.0 cm path length.



Molar absorptivity

λ(nm)	A	ε(L/mol/cm)
254.45	0.9131	39869
273.43	0.8782	38346
301.73	0.3733	16300
290.08	*0.3956*	*17273*
320.05	*0.1392*	*6078*