

In(OTf)₃ Catalysed an Expeditious Synthesis of β -Carboline- imidazo[1,2-*a*]pyridine and imidazo[1,2-*a*]pyrazine Conjugates

Nisha Devi,^a Dharmender Singh,^a Honey,^a Satbir Mor,^b Sandeep Chaudhary^c, Ravindra Kumar Rawal^d, Vipin Kumar,^a Asim Kumar Chowdhury^a and Virender Singh^{a*}

^aDepartment of Chemistry, Dr B R Ambedkar National Institute of Technology, Jalandhar, 144011, Punjab, India

^bDepartment of Chemistry, GJ University of Science and Technology, Hisar, 125001, India

^cDepartment of Chemistry, Malaviya National Institute of Technology, Jaipur, 302017, India

^dDepartment of Pharmaceutical Chemistry, Indo-Soviet Friendship College of Pharmacy, Moga, 142001, Punjab, India

E mail:- singhv@nitj.ac.in; singhvirender010@gmail.com; Fax: (91) 172 2214692

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General Procedure for preparation of 1-(dimethoxymethyl)-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indole (2a-b)^{21a} derivatives as exemplified for 2a: To a stirred solution of L-tryptophan methyl ester **1a** (18.53 g, 85.0 mmol) in anhydrous dichloromethane (120 mL), 2,2-dimethoxy acetaldehyde (60% aqueous solution in water) (14.7 mL, 102.0 mmol) was added at room temperature. Thereafter a solution of 4.5 mL TFA (3 % TFA in CH₂Cl₂) in 30 mL dichloromethane was added in small portions and the reaction mixture was further stirred at room temperature for 16 h. After completion of the reaction as confirmed by TLC, the reaction was quenched by slow addition of 10% aqueous NaHCO₃ solution under stirring. After the pH was set to slight alkaline (ca 7.5), the organic layer was pooled and the aqueous layer was further extracted with chloroform (3 × 30 mL). The organic layers were combined and washed with brine (50 mL), dried over anhydrous Na₂SO₄ and concentrated *in vacuo* to yield the product as yellow oil; **2a** (24.80 g, 96%; R_f = 0.50 (CHCl₃/MeOH, 95:05, v/v) which was utilized for the next step without further purification.

General Procedure for preparation of 1-(dimethoxymethyl)-9H-pyrido[3,4-b]indole derivatives (3a-b)²¹ as exemplified for 3a: To a stirred solution of **2a** (24.5 g, 81.7 mmol) in dry DMF (150 mL), KMnO₄ (49.0 g) was added in small portions over a period of 30 min and stirred vigorously at room temperature for 2 h. After completion of the reaction as monitored by TLC, the viscous blackish content was poured in ice cold water and extracted with ethyl acetate (4 x 50 mL). The organic layer was further washed with 10% aq. NaHCO₃ solution (50 mL) and subsequently by brine (50 mL). The organic layer was concentrated *in vacuo* to yield the light yellow viscous product which was further triturated with hexane/EtOAc, (90:10, v/v) to obtain a pure white solid product, **3a** (21.98 g, 91%, R_f = 0.62 (CHCl₃/MeOH, 95:05, v/v). It deserves attention that KMnO₄ must be added in small portions as the reaction is highly exothermic.

General Procedure for preparation of N-alkylated 1-(dimethoxymethyl)-9H-pyrido[3,4-b]indole (4a-e)²¹ derivatives as exemplified by 4a: To a stirred solution of **3a** (12.0 g, 40.0 mmol) in dry DMF (70 mL), Cs₂CO₃ (19.6 g, 60.0 mmol) was added and stirred the reaction mixture at room temperature for 20 min. Thereafter methyl iodide (3.1 mL, 50.0 mmol) in 10 mL dry DMF was added drop wise and the reaction was stirred for additional 1 h at room temperature. On completion of the reaction, as monitored by TLC, the contents were poured into ice cold water (150 mL) under stirring. Thereafter the aqueous layer was extracted with

EtOAc (4 × 50 mL). The organic layers were combined and washed with brine (50 mL), dried over anhydrous Na₂SO₄ and concentrated to yield the yellow solid product, **4a** (12.05 g, 96%) which was utilized for the next step without any purification. Similarly *N*-benzyl, -allyl and -propargyl derivatives (**4b-e**) were prepared by using benzyl bromide, allyl bromide and propargyl bromide in presence of Cs₂CO₃ in dry DMF.

General Procedure for preparation of *N*-substituted 1-formyl-9*H*-pyrido[3,4-*b*]indole derivatives (5a-e) as exemplified for 5a: To a stirred solution of **4a** (12.0 g, 38.2 mmol) in glacial AcOH (48 mL); water (72 mL) was added and heated the content at 100 °C for 45 min. During this period, yellow solid precipitated out from the reaction mixture and the content was further diluted with ice cold water (100 mL) and yellow solid was filtered under suction and further washed with 10% aqueous NaHCO₃ solution (100 mL). The yellow solid was further air dried and triturated with 5% ethyl acetate: hexane (v/v, 40 mL) to yield the analytically pure yellow solid product, **5a** (10.1 g, 99%) which was subsequently used for GBB multicomponent strategy to generate β-carboline-Imidazo[1,2-*a*]pyridine and β-carboline-imidazo[1,2-*a*]pyrazine conjugates (**6-7**). It deserves attention here that if during deprotection of acetal (**4a-e**), heating is performed at 120 °C or reaction is stirred at 100 °C for longer duration (>1 h), it result in change in color of compound from yellow to dark brown solid and decrease in yield (99% to 90%).

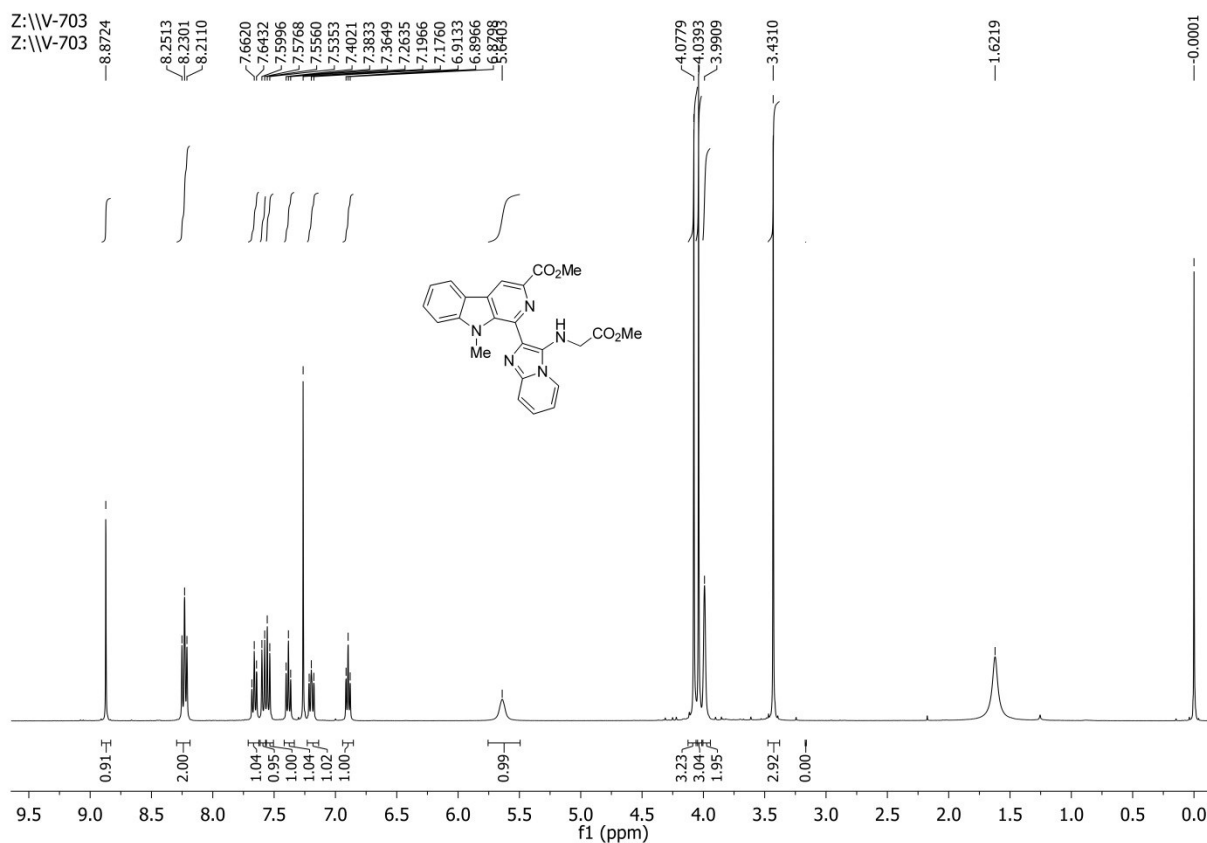


Fig. S 1. ¹H-NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aAX**).

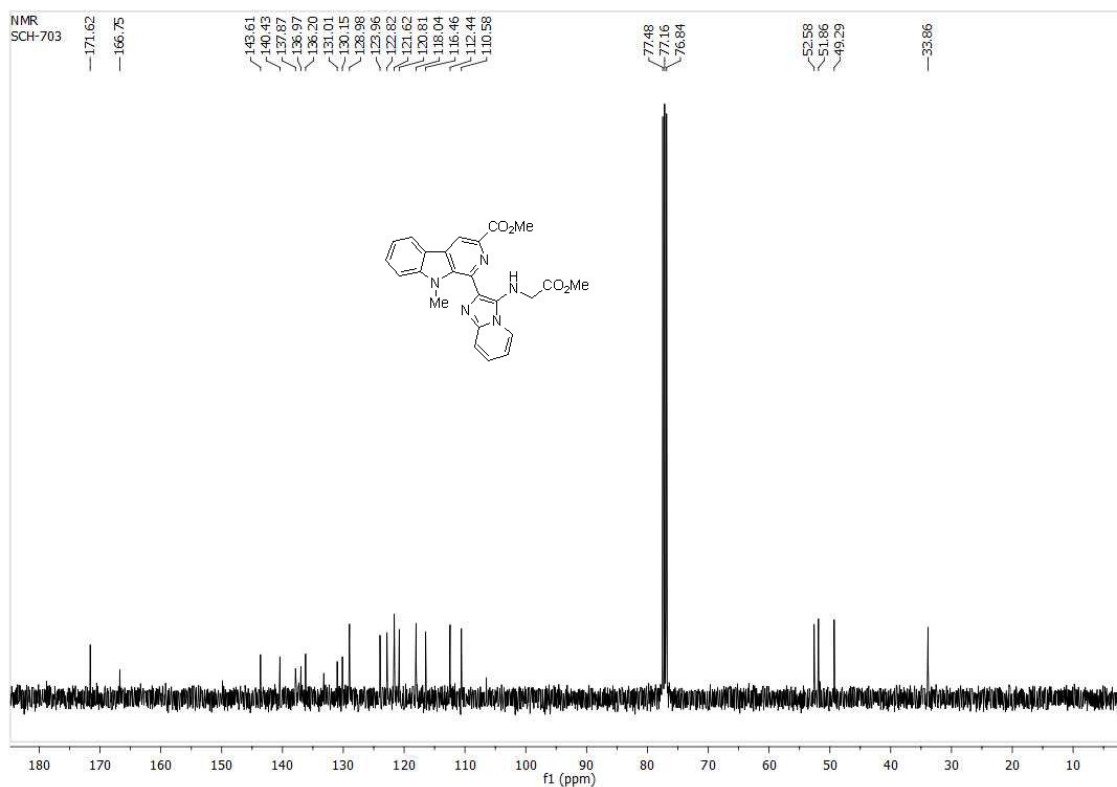


Fig. S 2. ¹³C-NMR of ethyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aAX**).

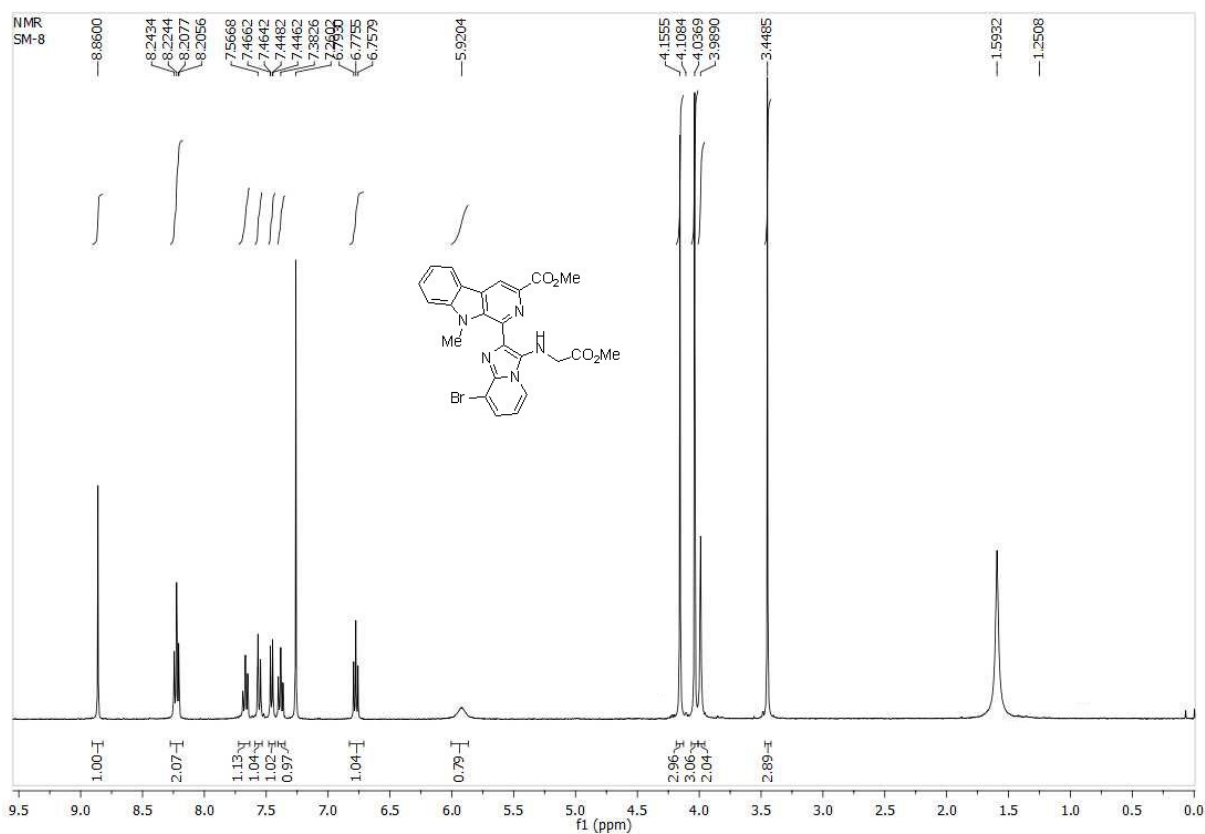


Fig. S 3. ¹H-NMR of Methyl 1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6aBX**).

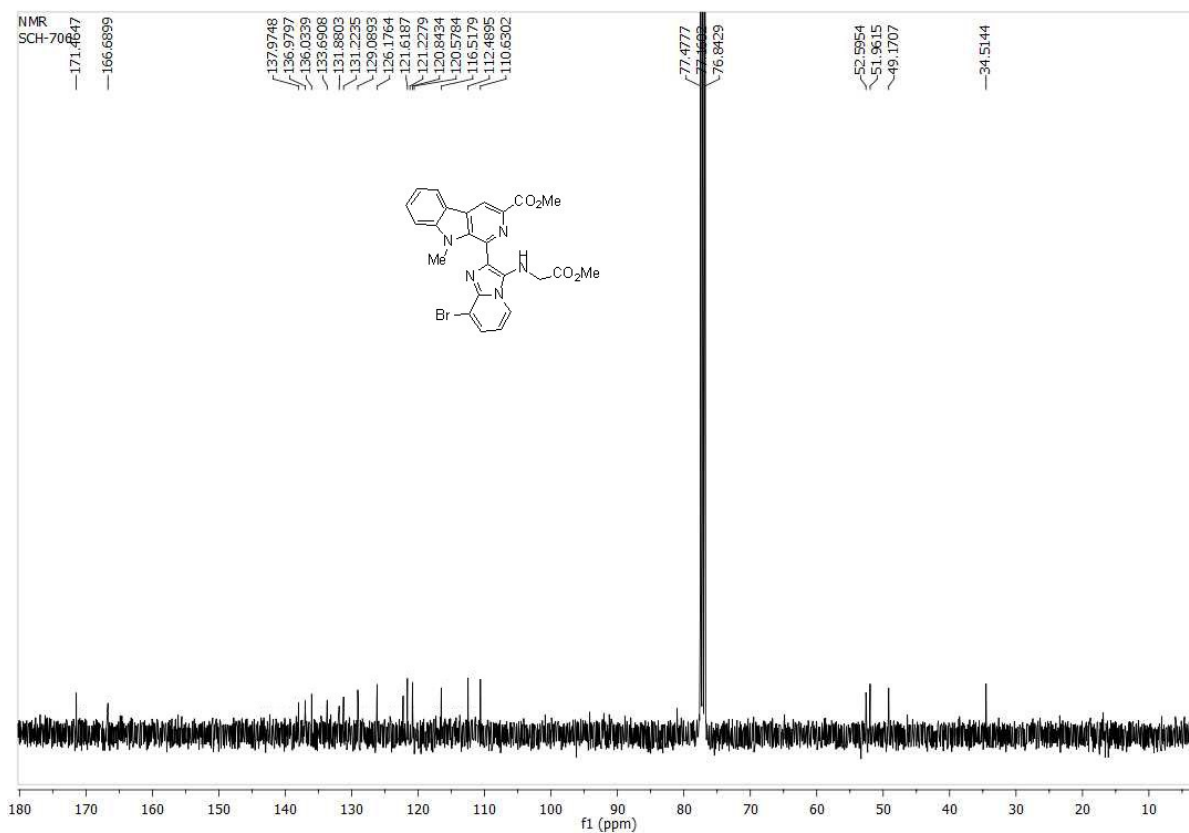


Fig. S 4. ¹³C-NMR of methyl 1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6aBX**).

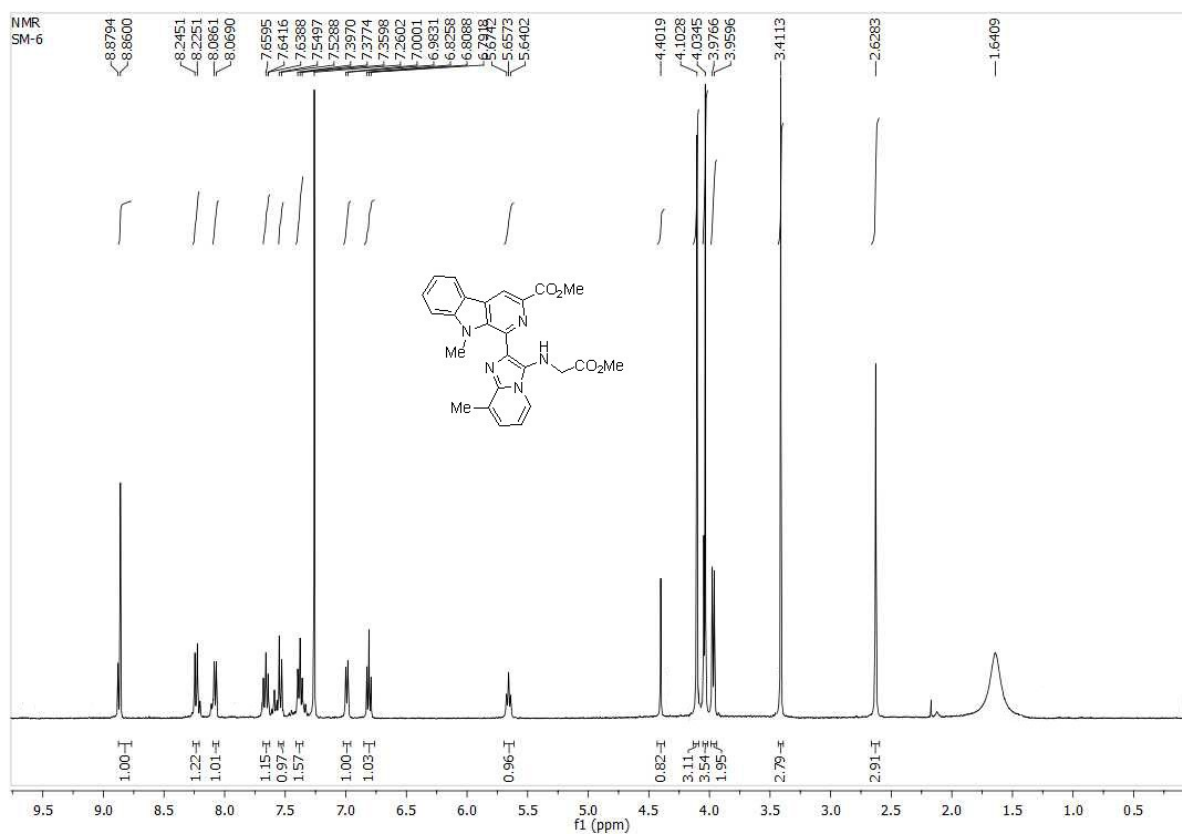


Fig. S 5. $^1\text{H-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6aCX**).

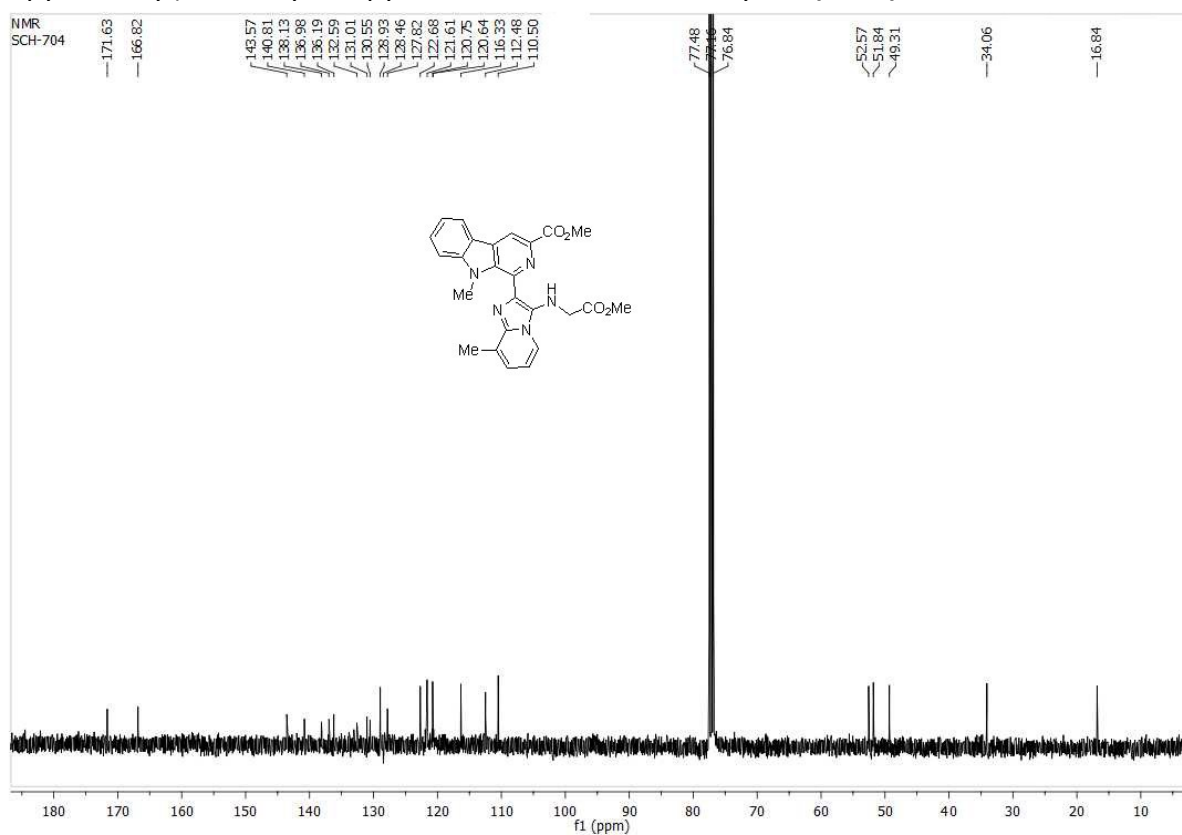


Fig. S 6. $^{13}\text{C-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6aCX**).

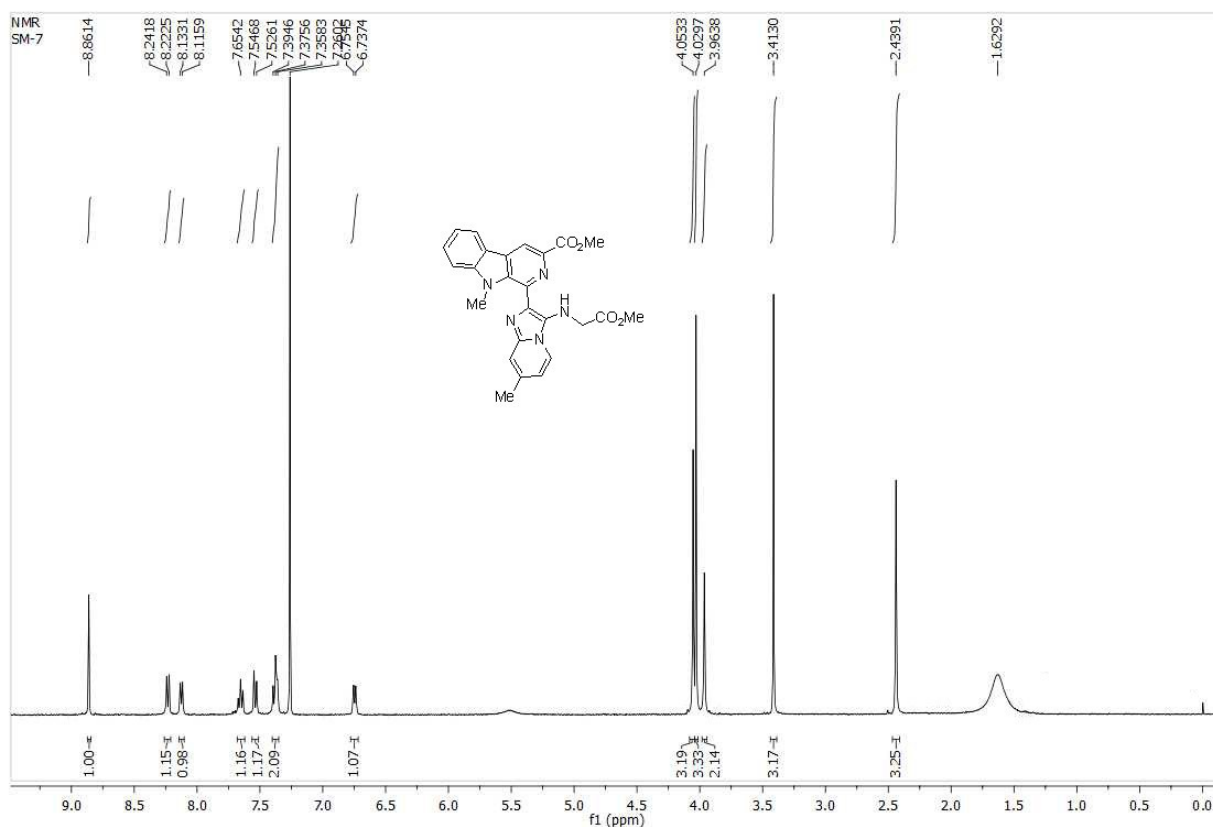


Fig. S 7. $^1\text{H-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (6aDX).

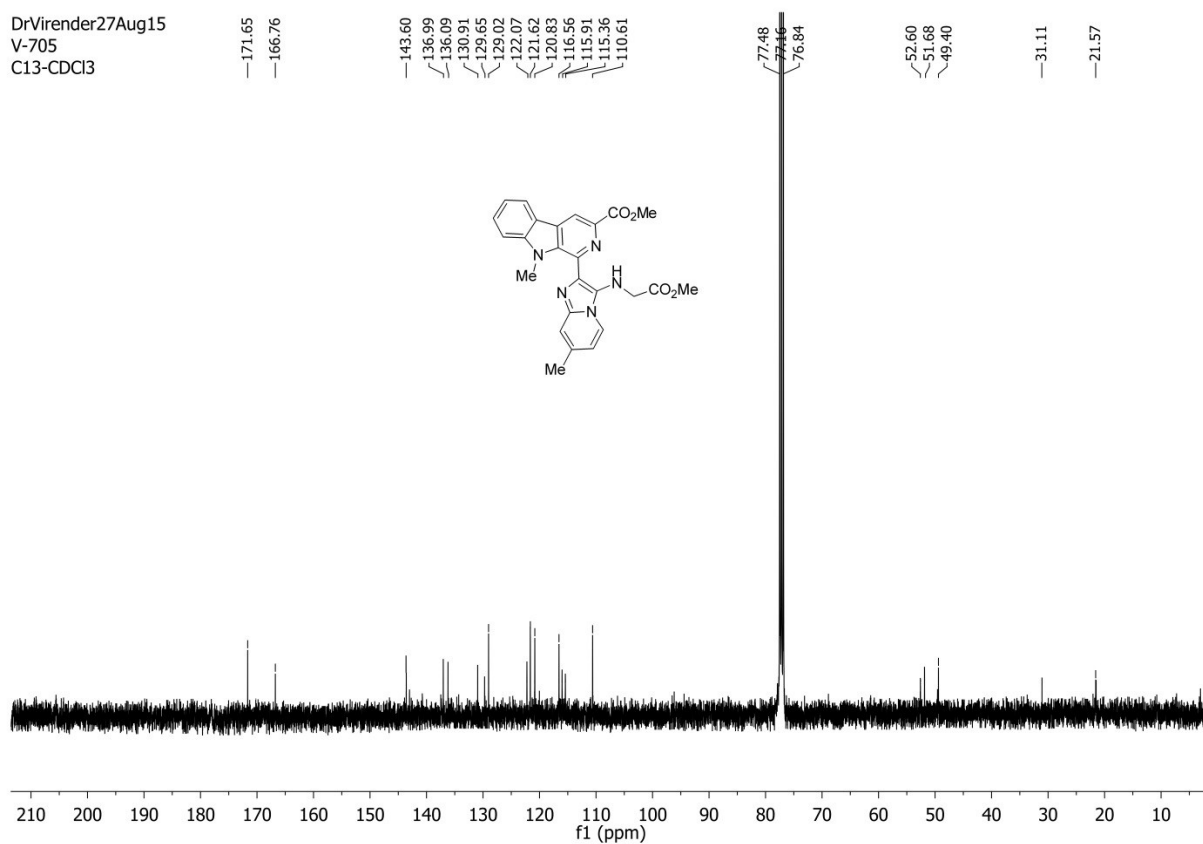


Fig. S 8. $^{13}\text{C-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (6aDX).

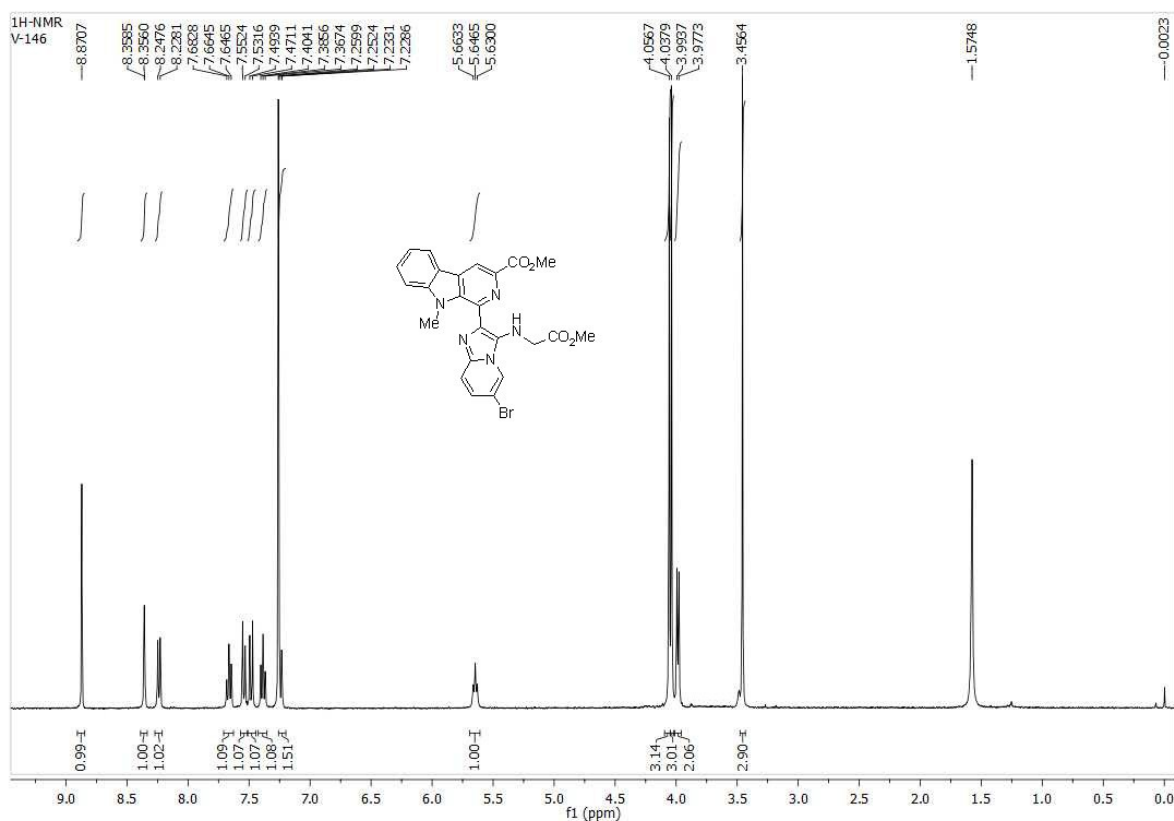


Fig. S 9. $^1\text{H-NMR}$ of methyl 1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aEX**).

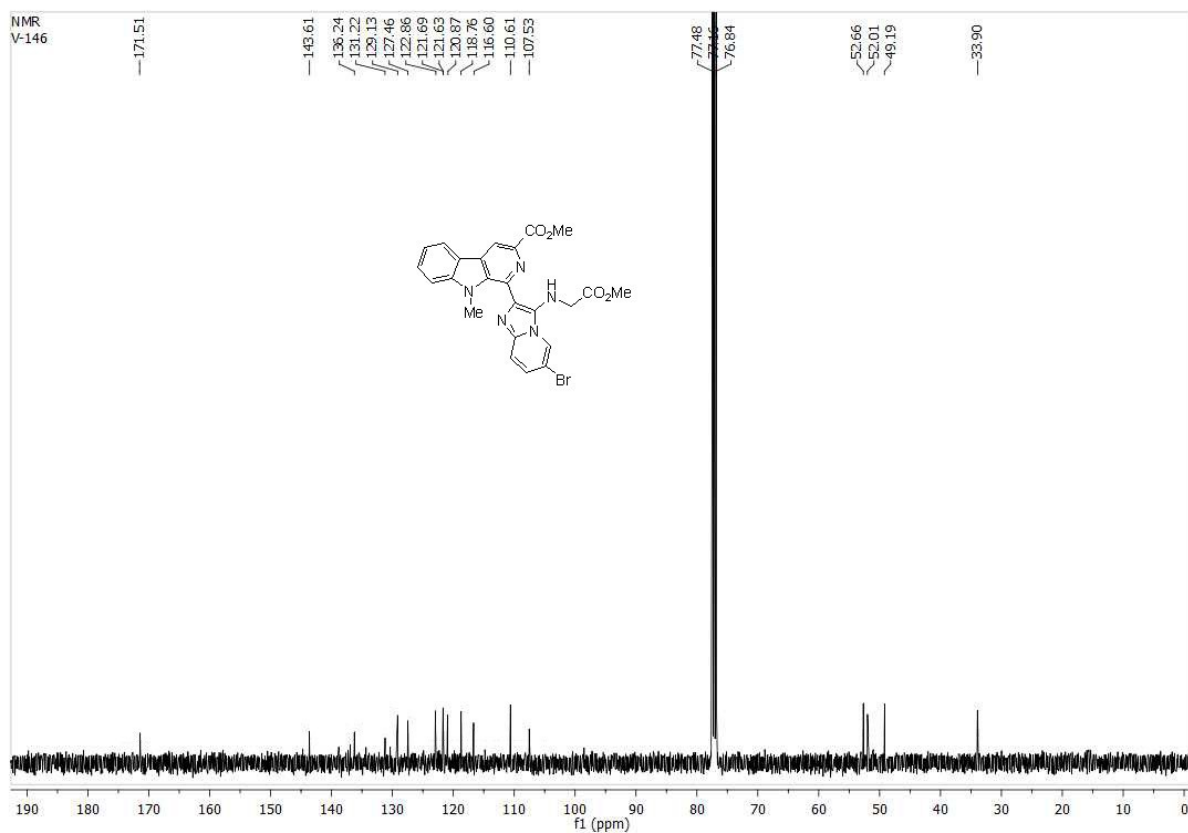


Fig. S 10. $^{13}\text{C-NMR}$ of methyl 1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aEX**).

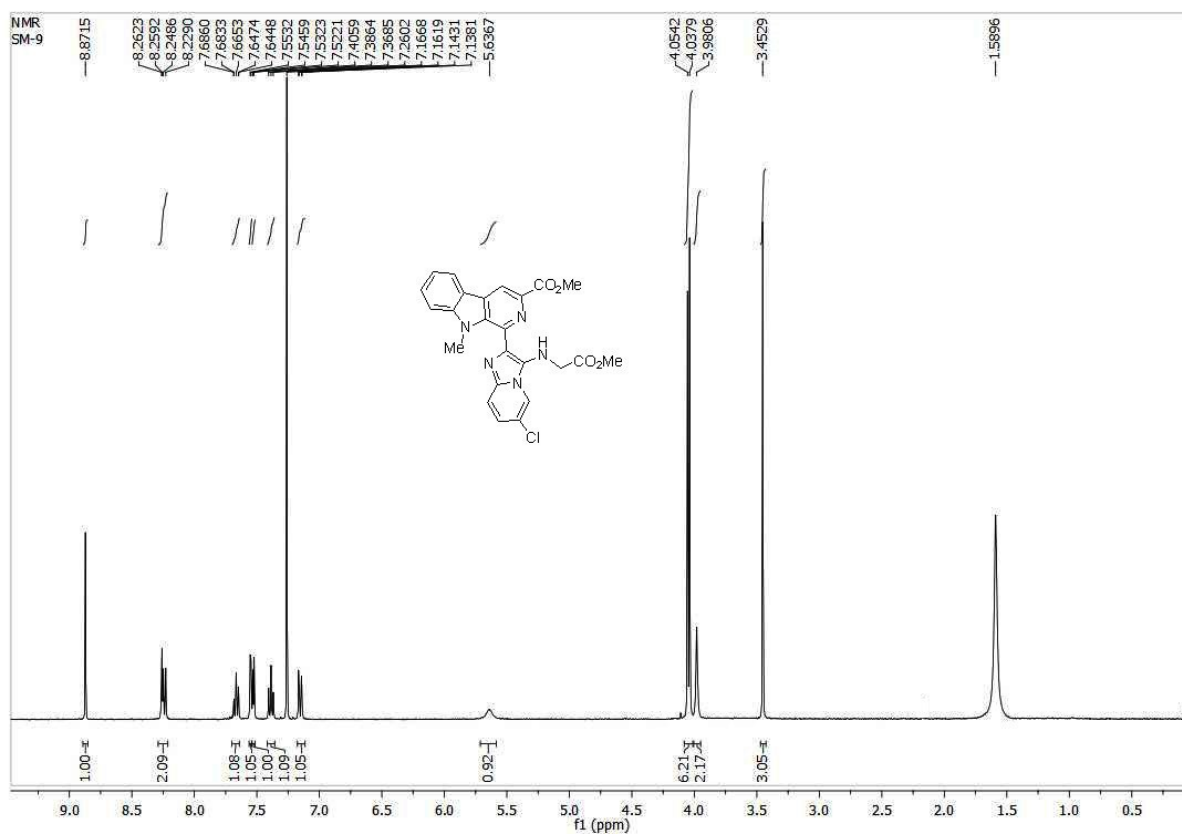


Fig. S 11. ¹H-NMR of methyl 1-(6-chloro-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aFX**).

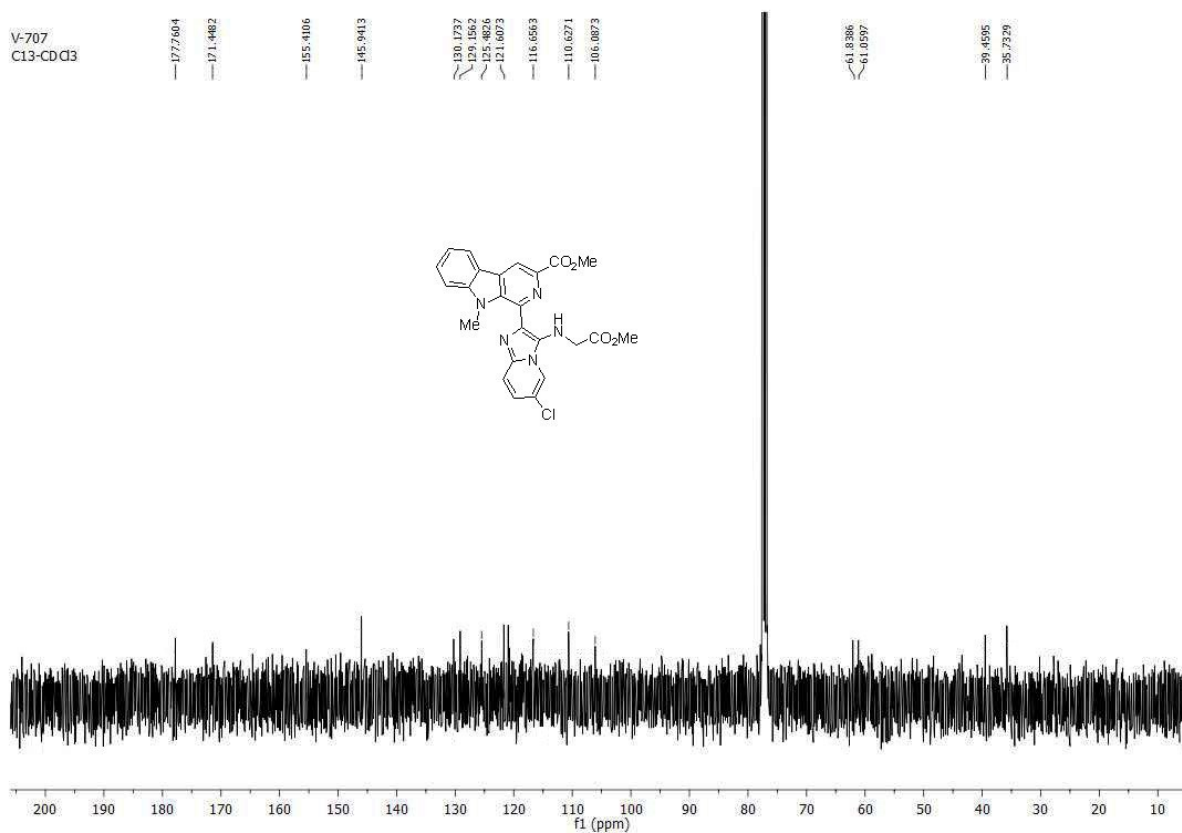


Fig. S 12. ¹³C-NMR of methyl 1-(6-chloro-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aFX**).

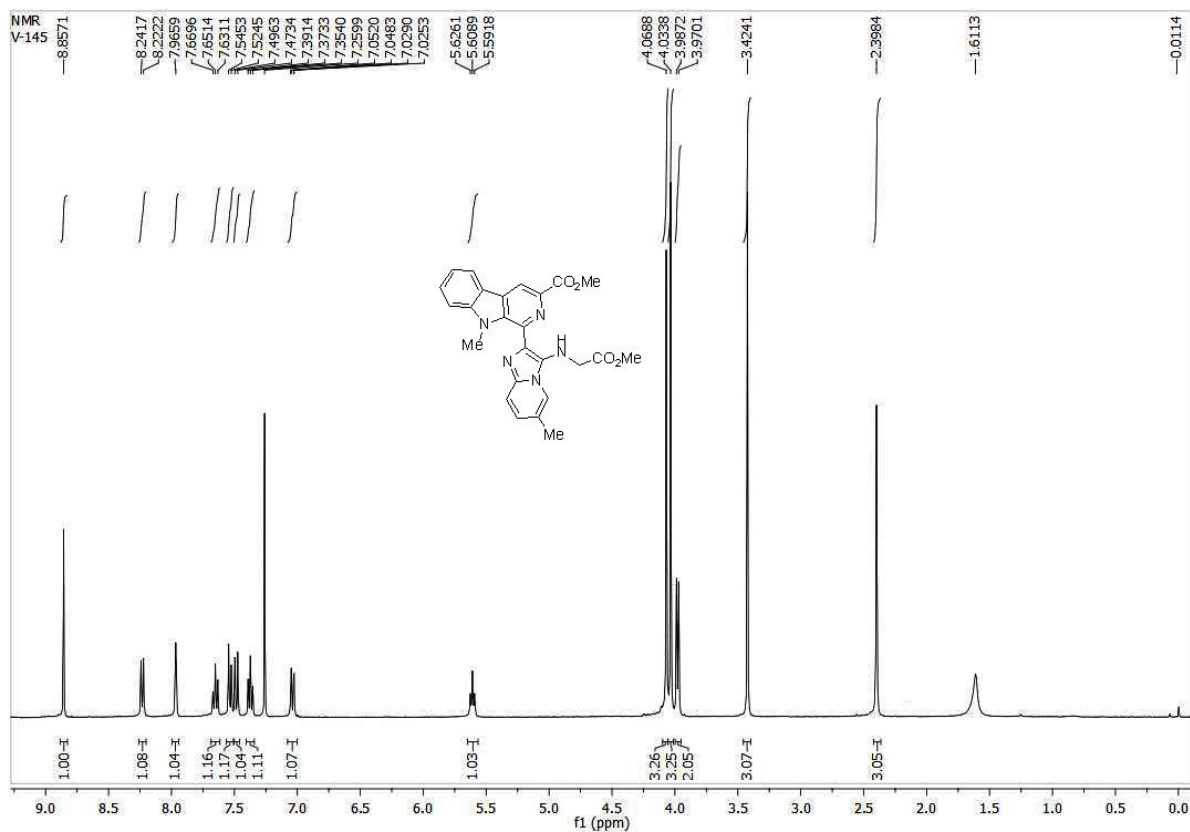


Fig. S 13. ¹H-NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aGX**).

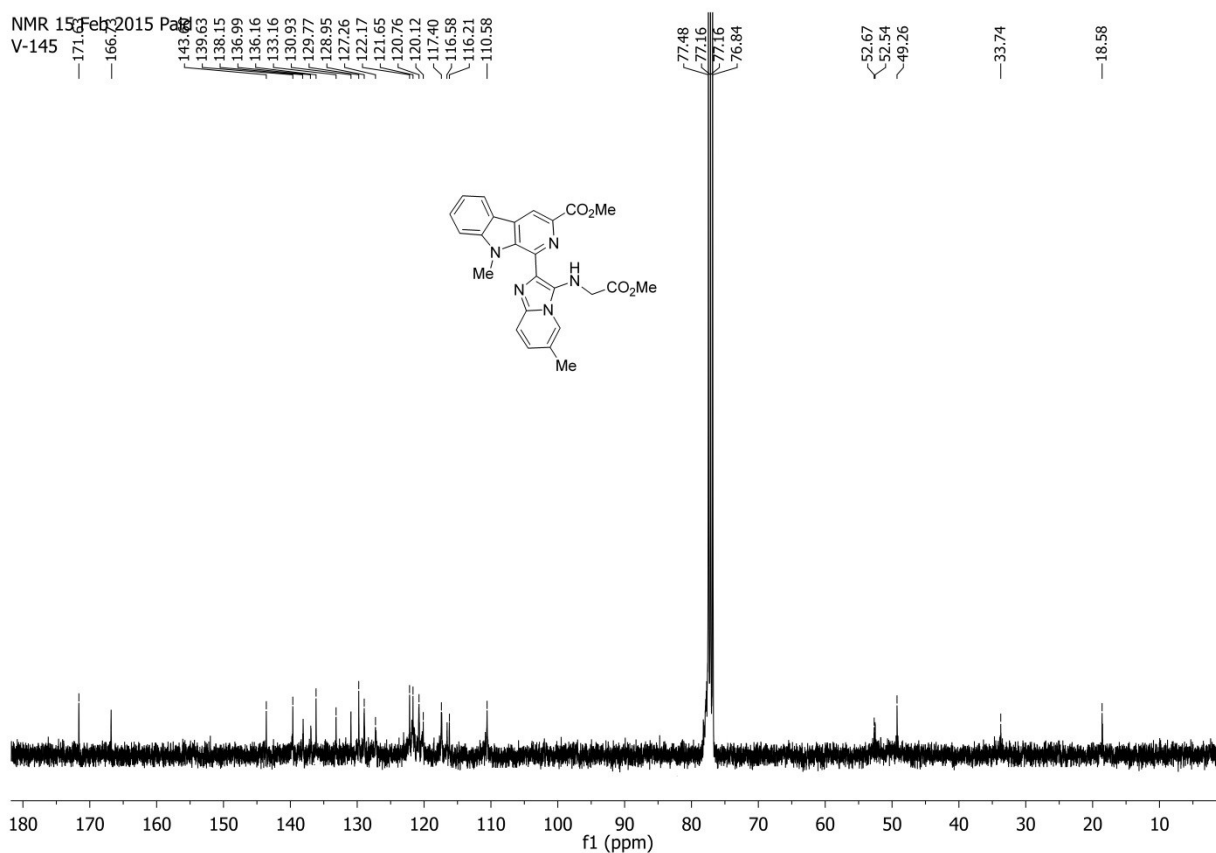


Fig. S 14. ¹³C-NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aGX**).

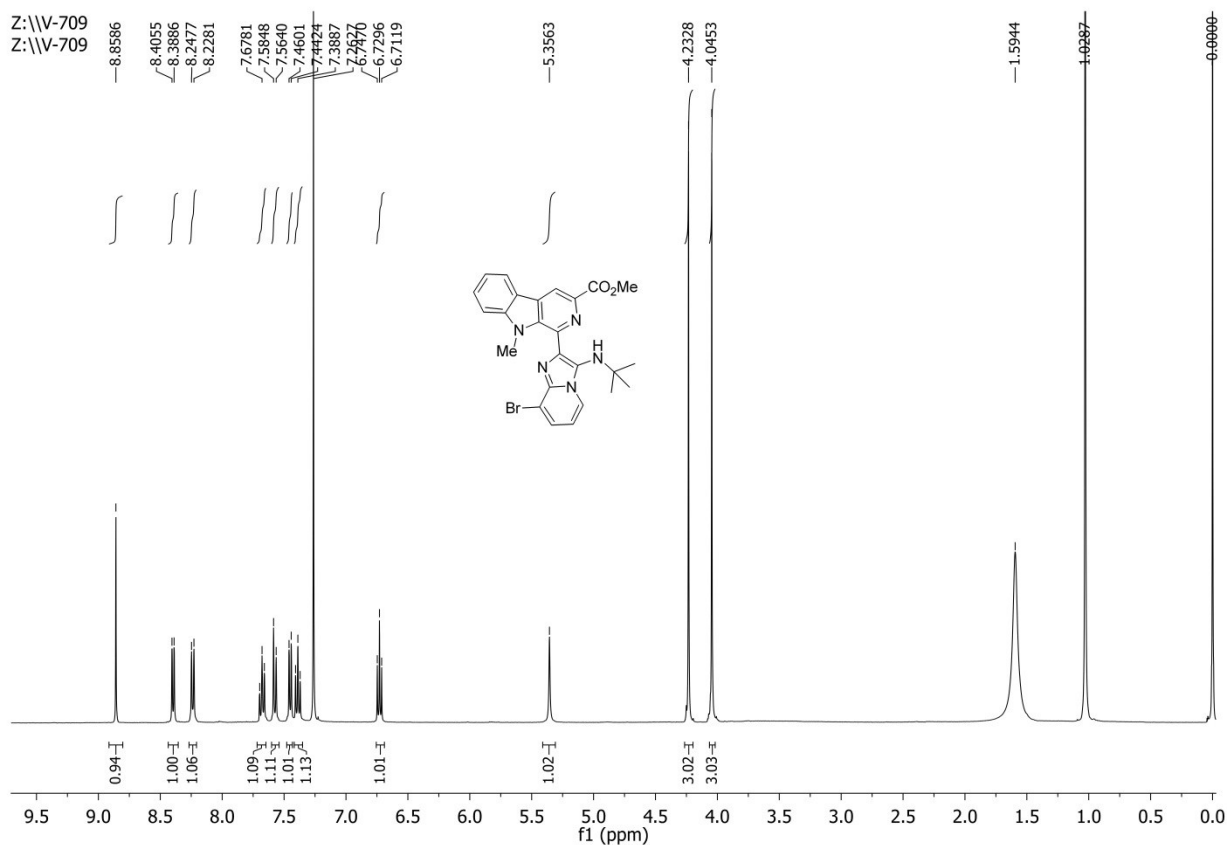
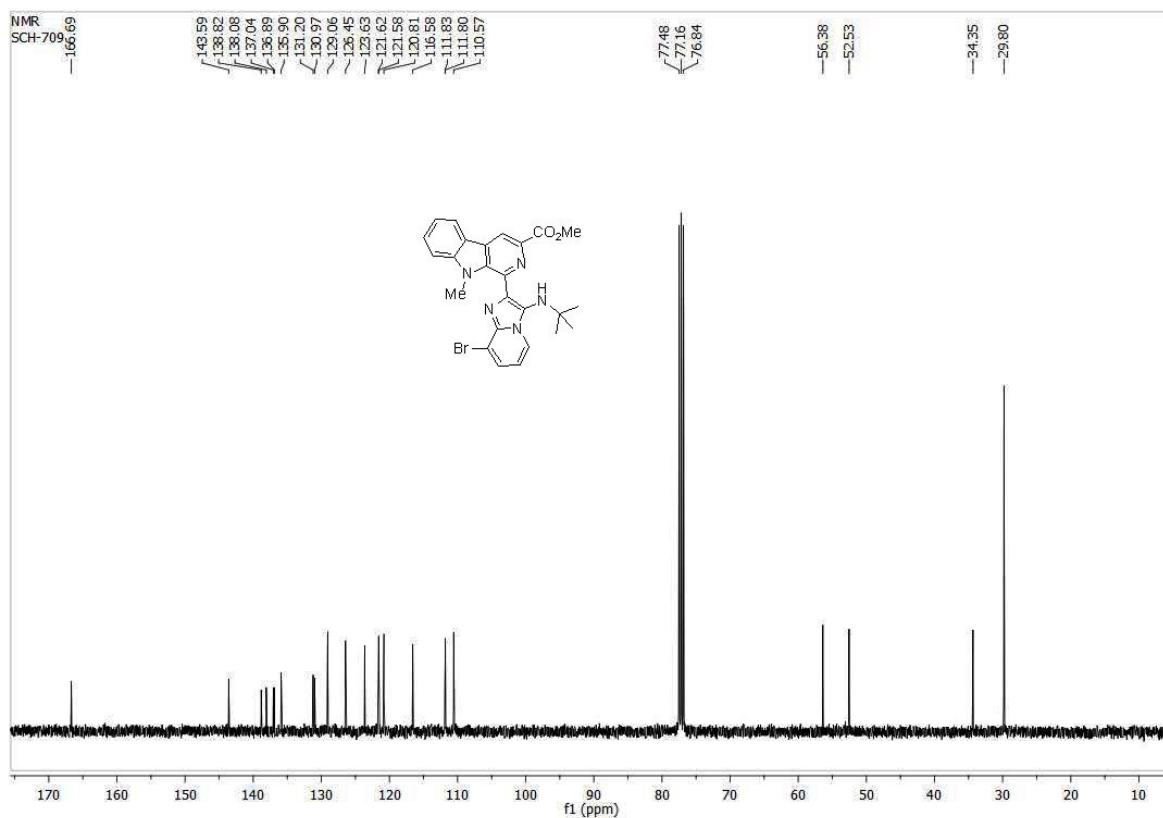


Fig. S 15. $^1\text{H-NMR}$ of methyl 1-(8-bromo-3-(*tert*-butylamino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aBZ**).



S 16. $^{13}\text{C-NMR}$ of methyl 1-(8-bromo-3-(*tert*-butylamino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aBZ**).

Fig

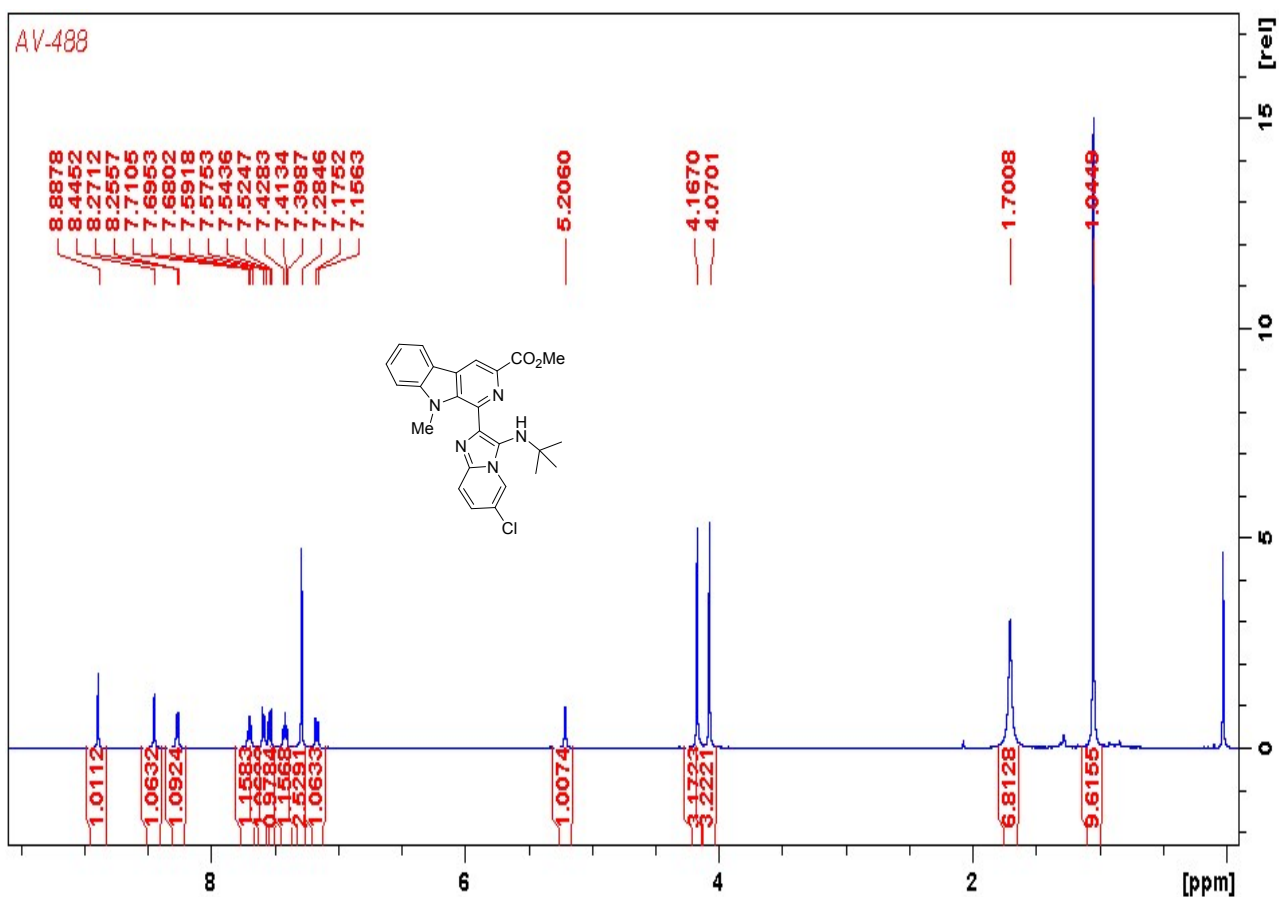


Fig. S 17. $^1\text{H-NMR}$ of methyl 1-(8-bromo-3-(*tert*-butylamino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aBY**).

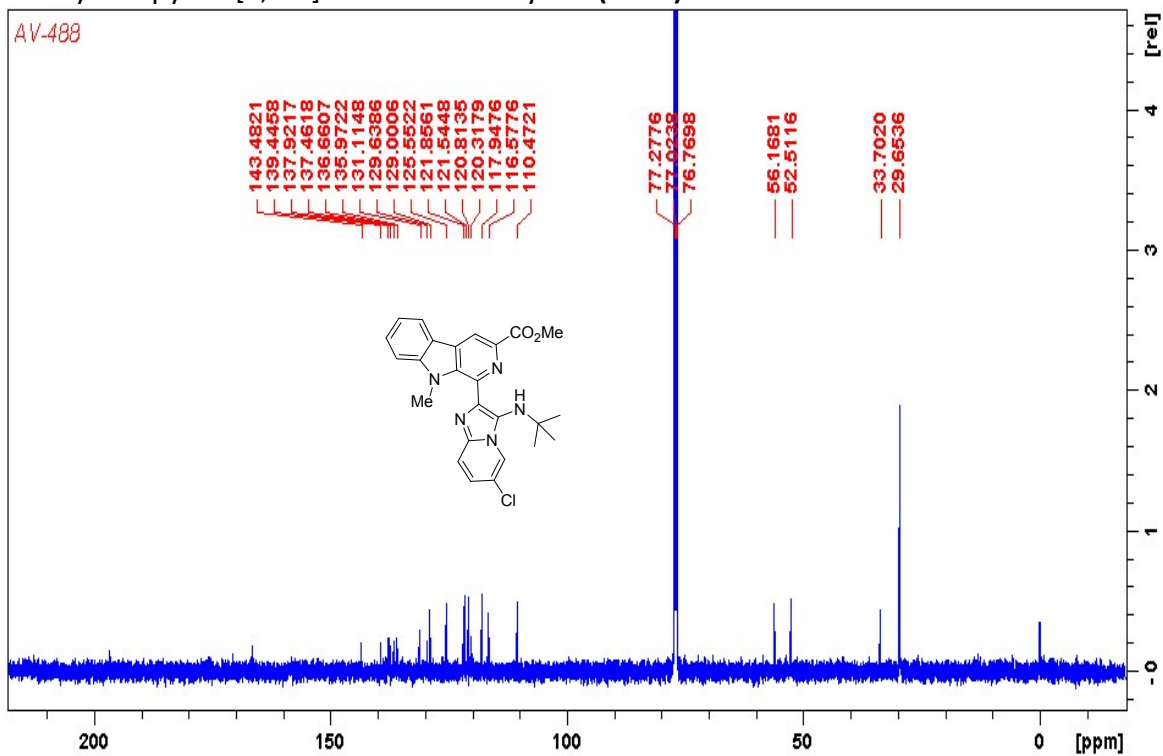


Fig. S 18. $^{13}\text{C-NMR}$ of methyl 1-(8-bromo-3-(*tert*-butylamino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6aBY**).

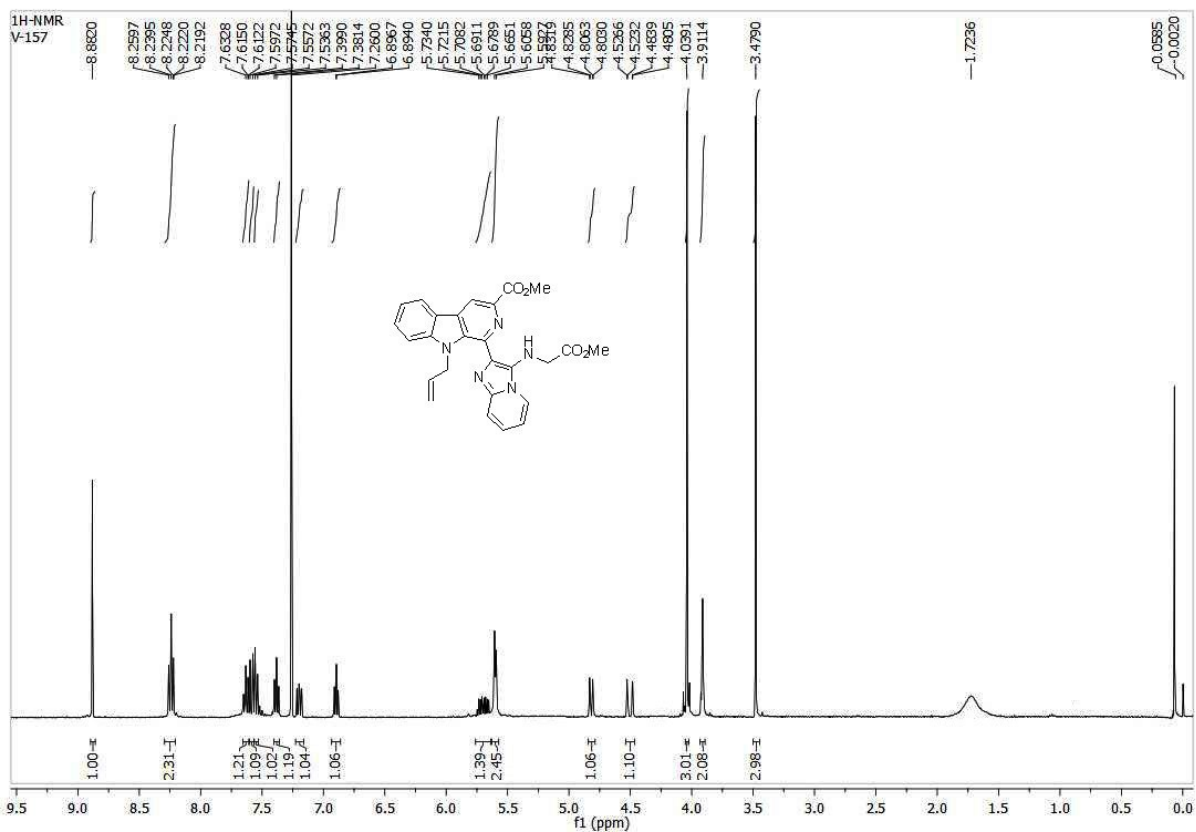


Fig. S 19. $^1\text{H-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6bAX**).

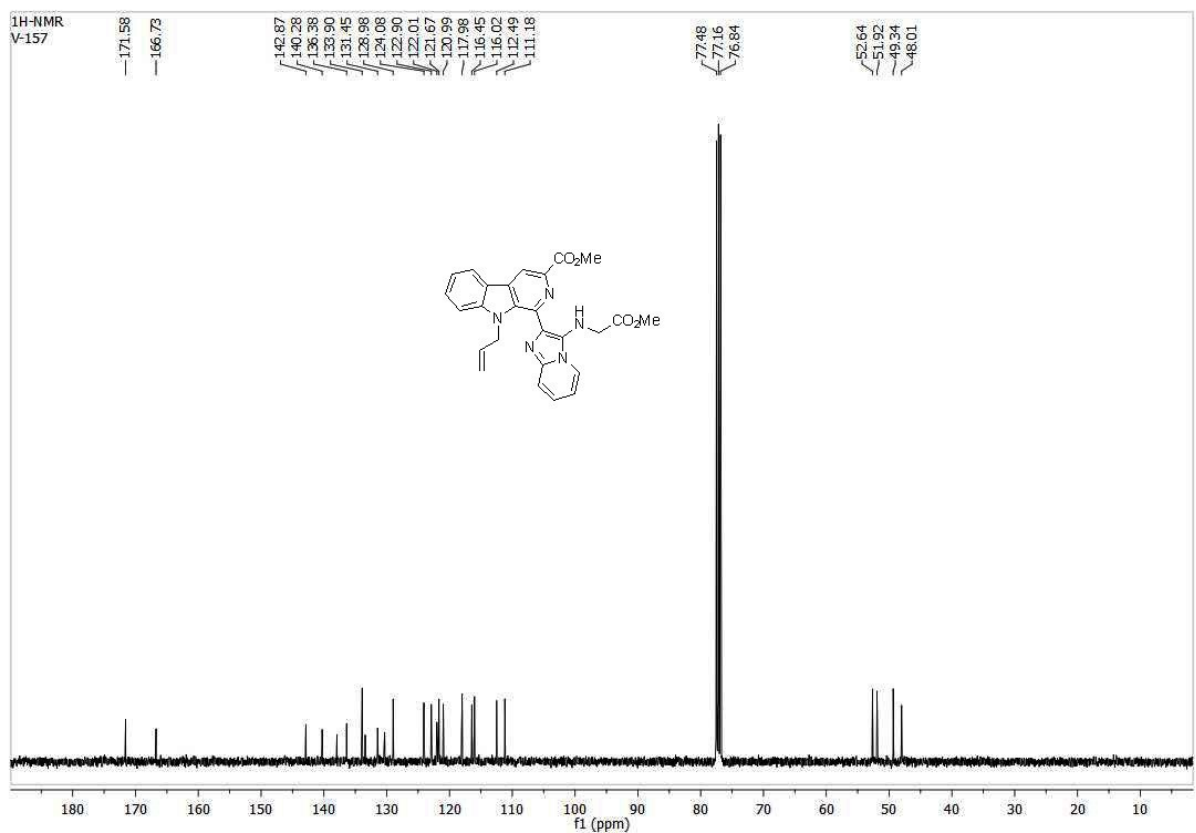


Fig. S 20. $^{13}\text{C-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9H-pyrido[3,4-*b*]indole-3-carboxylate (**6bAX**).

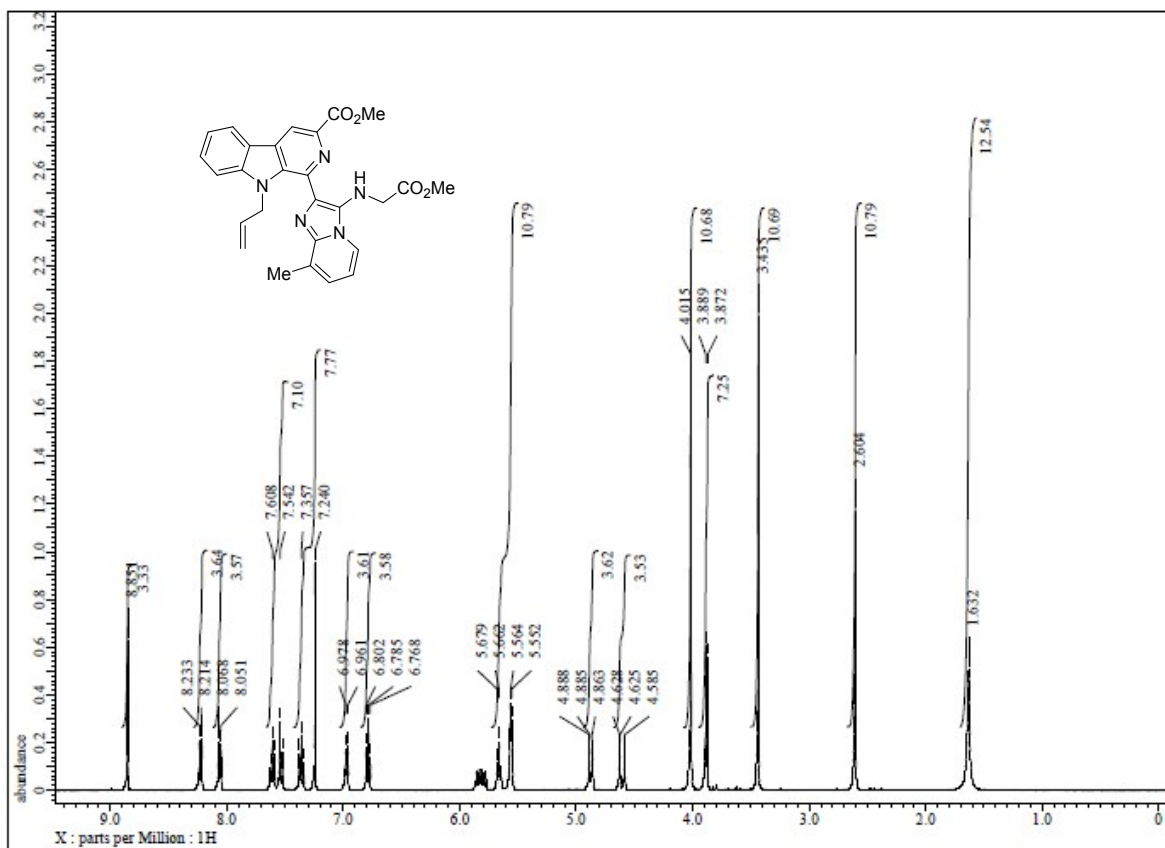


Fig. S 21. $^1\text{H-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bCX**).

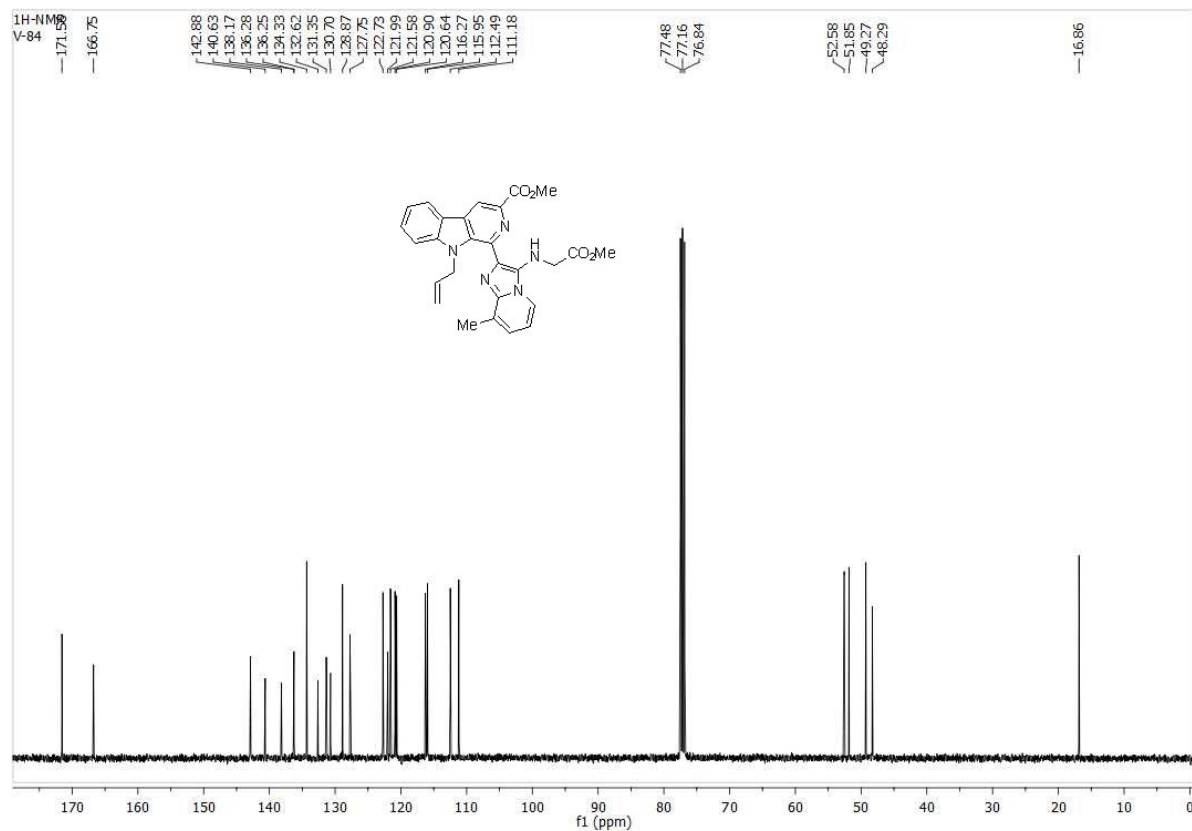


Fig. S 22. $^{13}\text{C-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bCX**).

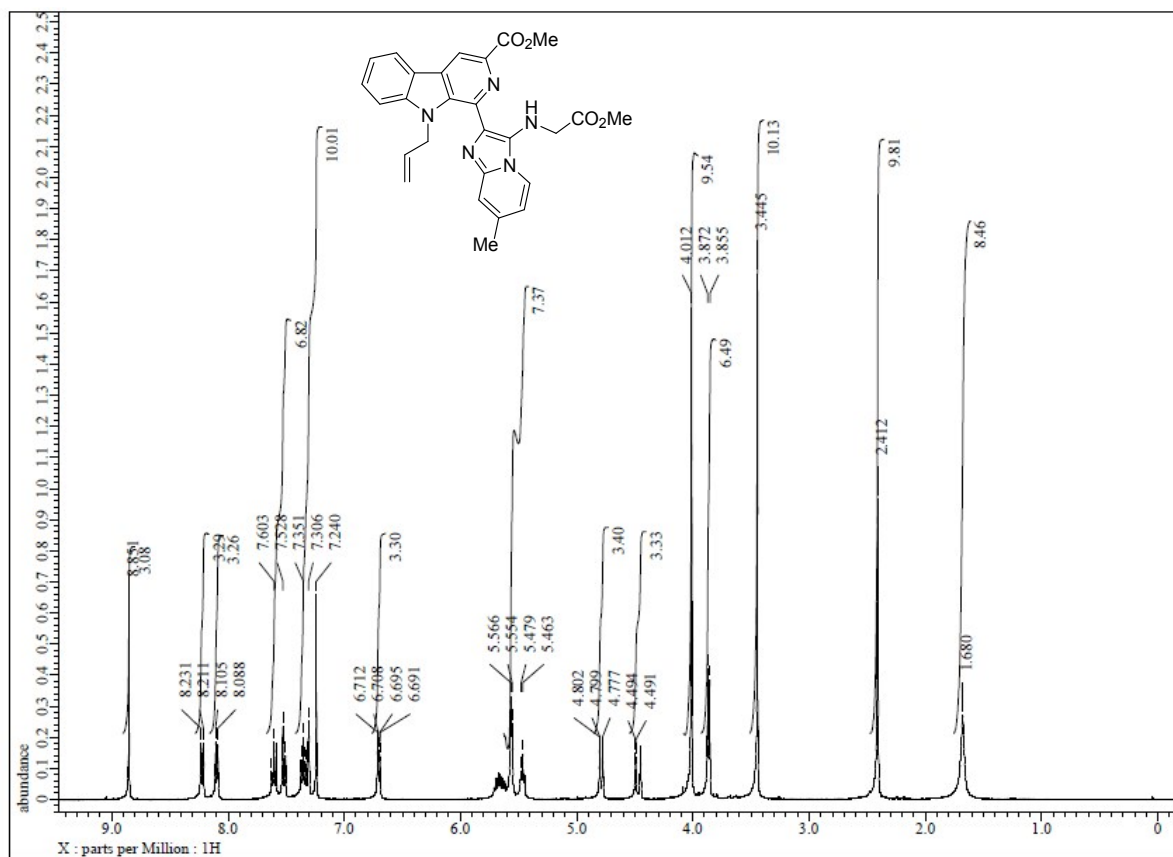


Fig. S 23. $^1\text{H-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bDX**).

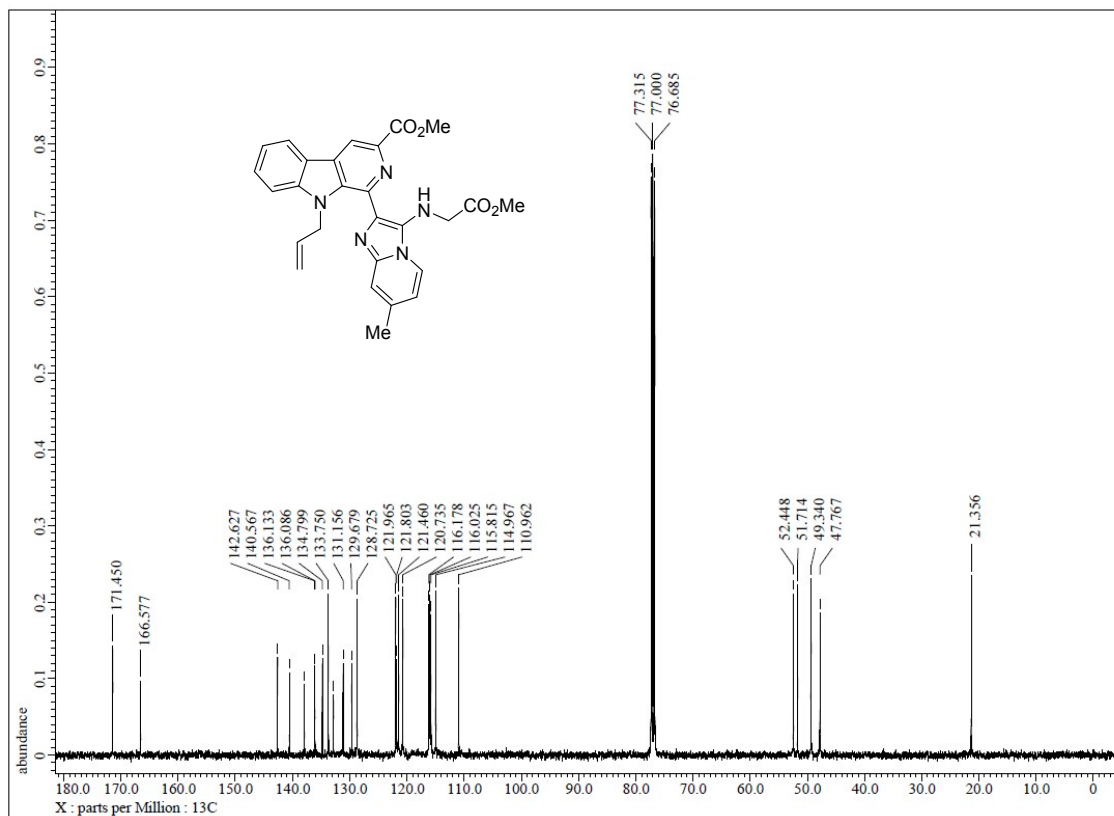


Fig. S 24. $^{13}\text{C-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bDX**).

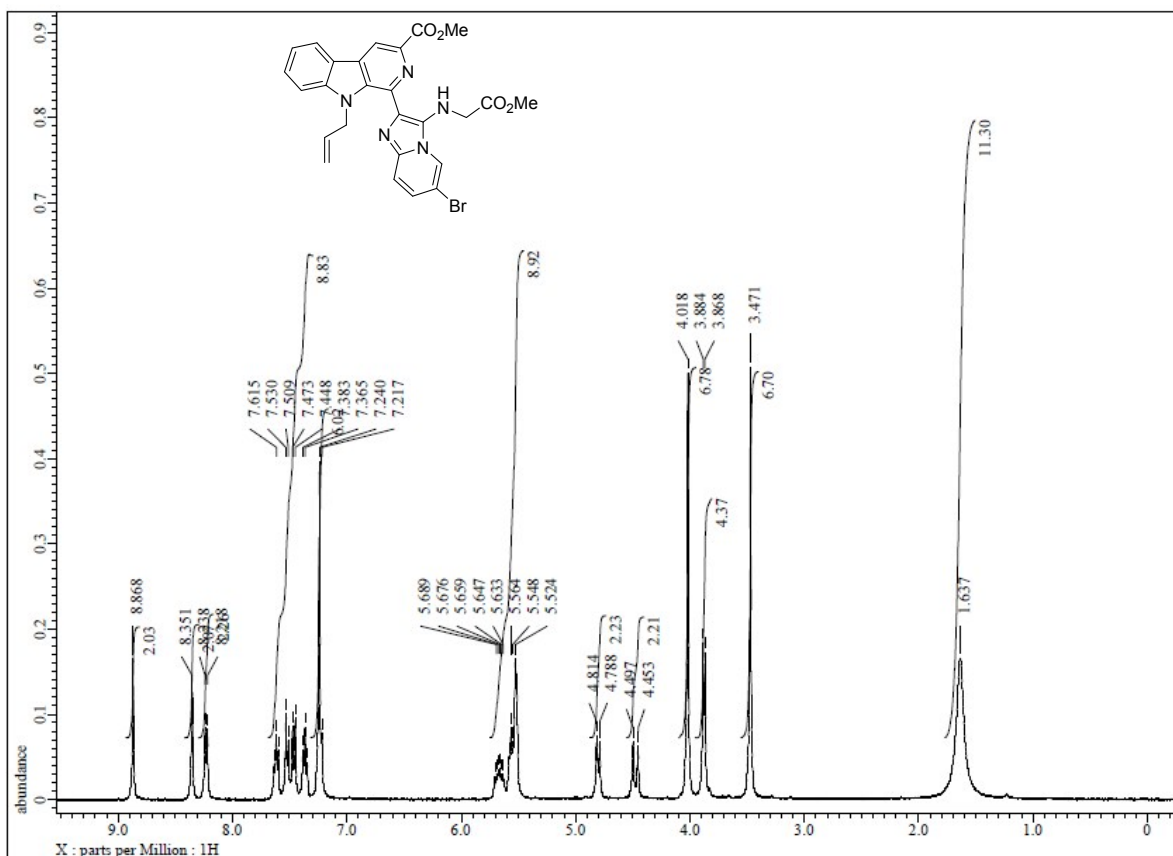


Fig. S 25. ¹H-NMR of methyl 9-allyl-1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-a]pyridin-2-yl)-9H-pyrido[3,4-b]indole-3-carboxylate (**6bEX**).

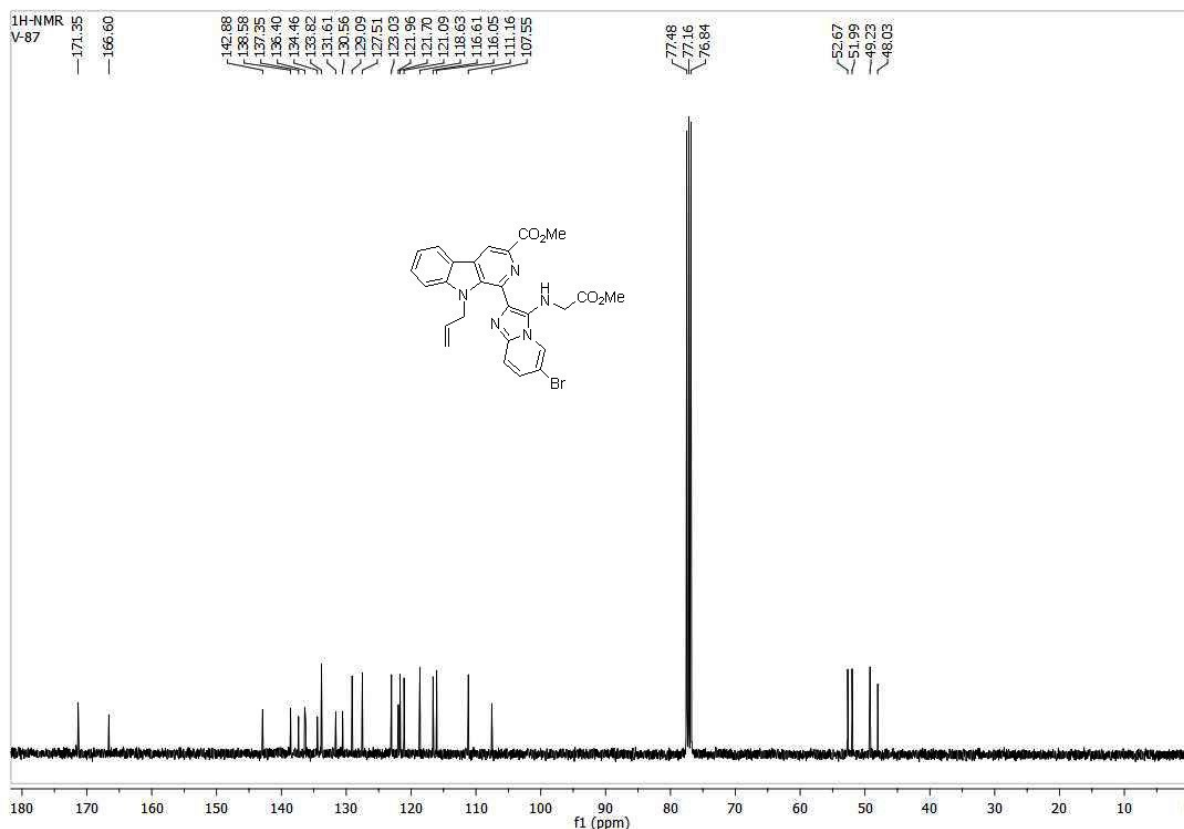


Fig. S 26. ¹³C-NMR of methyl 9-allyl-1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-a]pyridin-2-yl)-9H-pyrido[3,4-b]indole-3-carboxylate (**6bEX**).

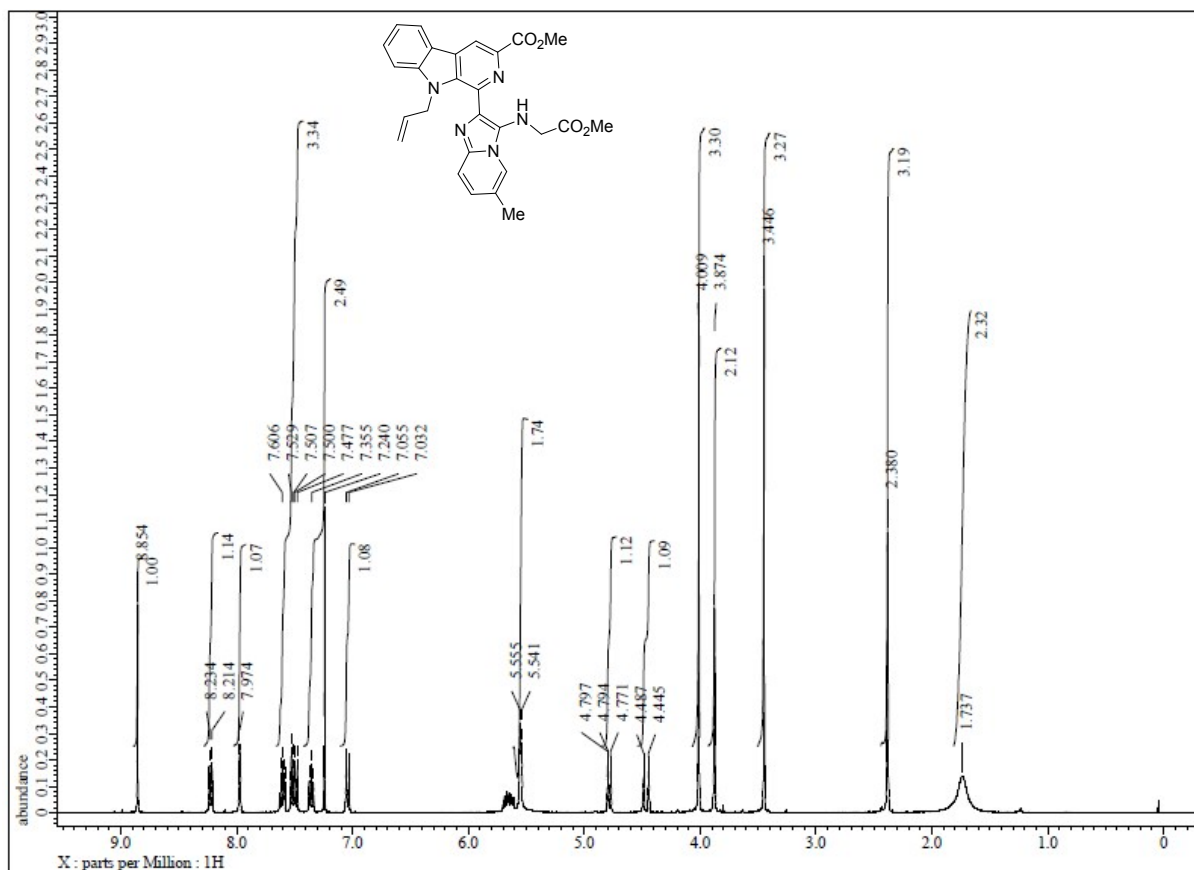


Fig. S 27. ¹H-NMR of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bGX**).

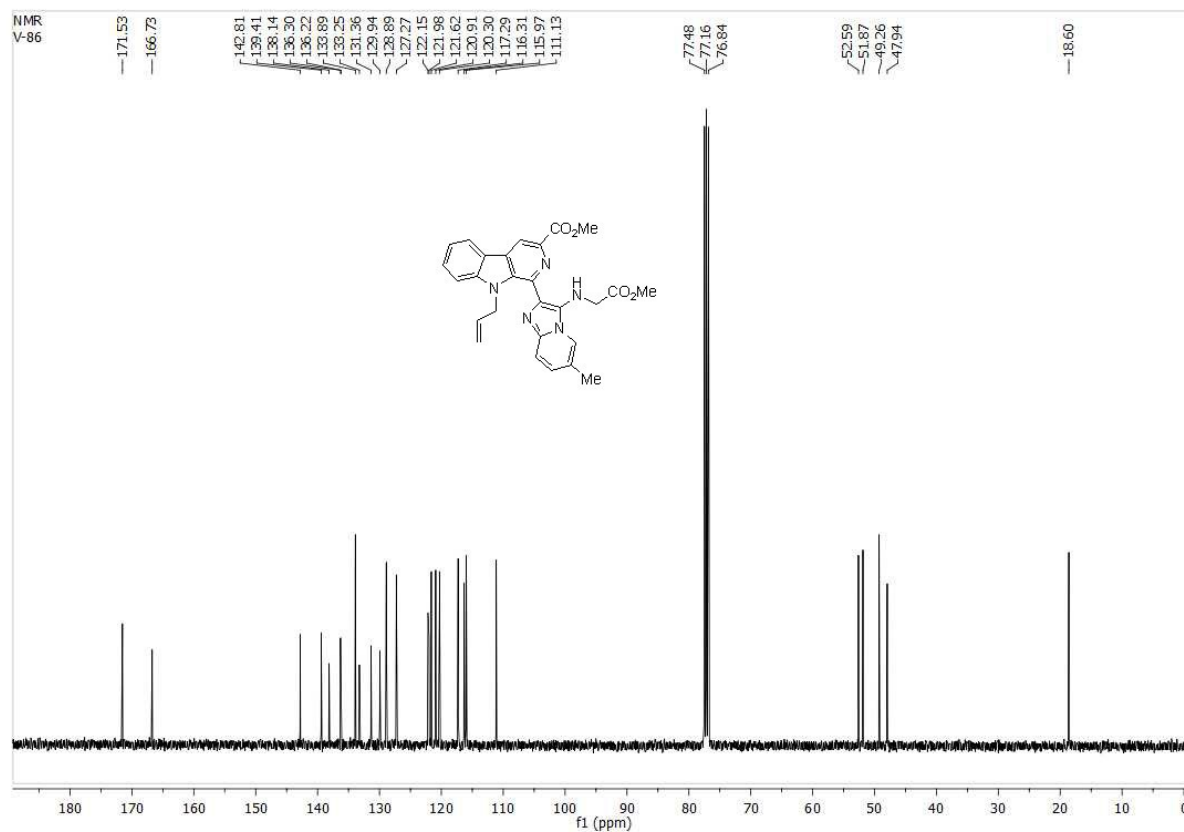


Fig. S 28. ^{13}C -NMR of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6bGX**).

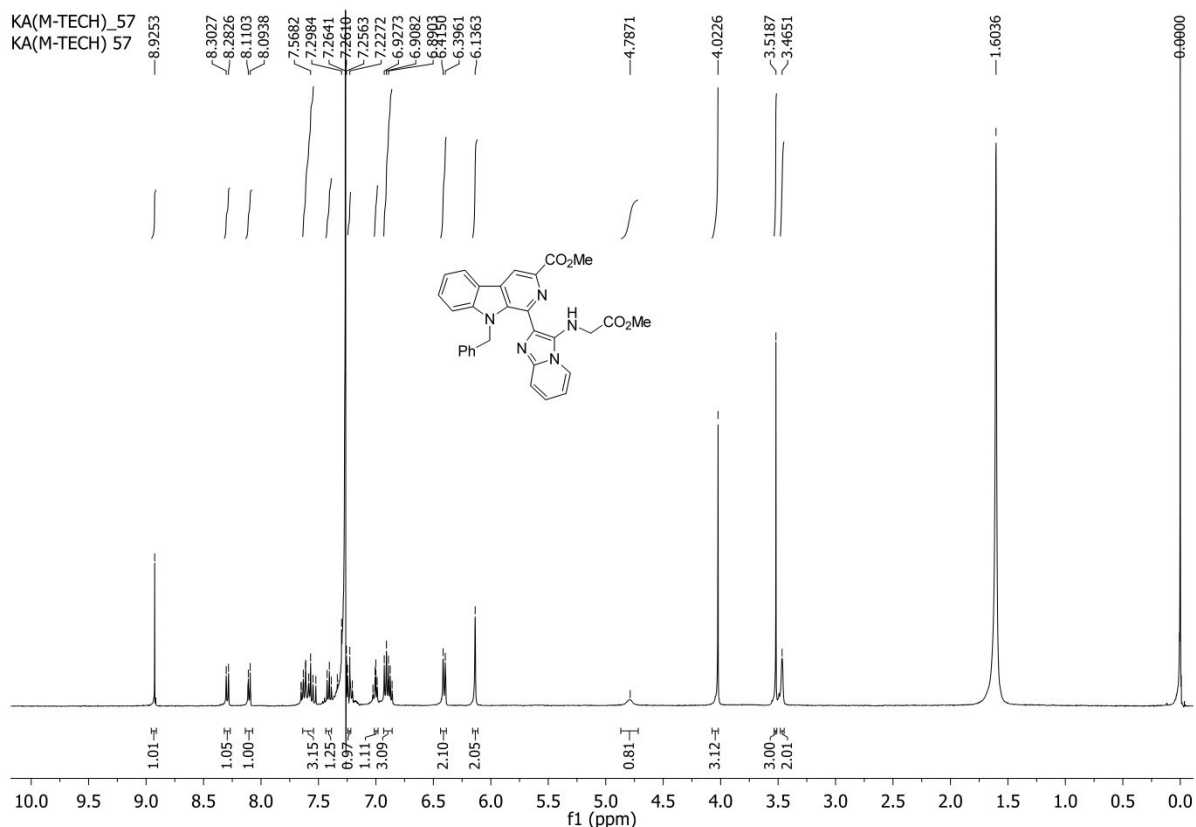


Fig. S 29. ^1H -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cAX**).

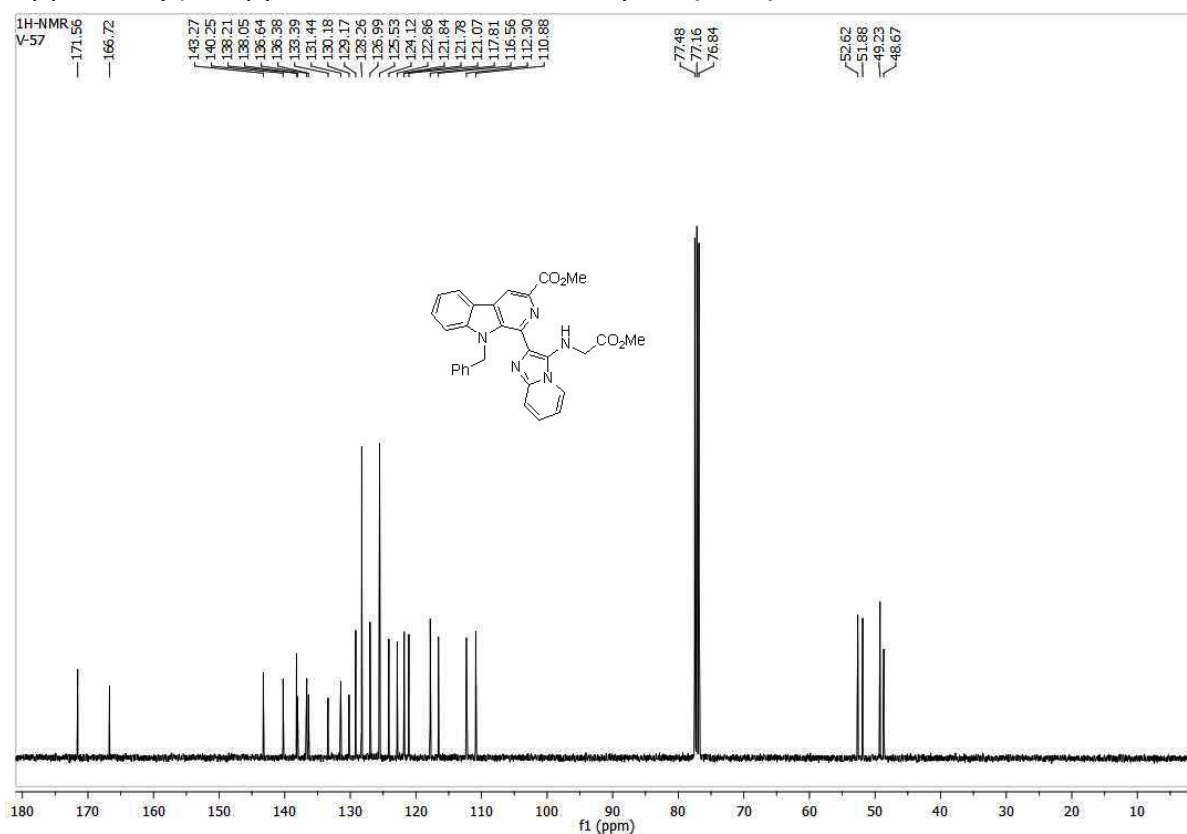


Fig. S 30. ^{13}C -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cAX**).

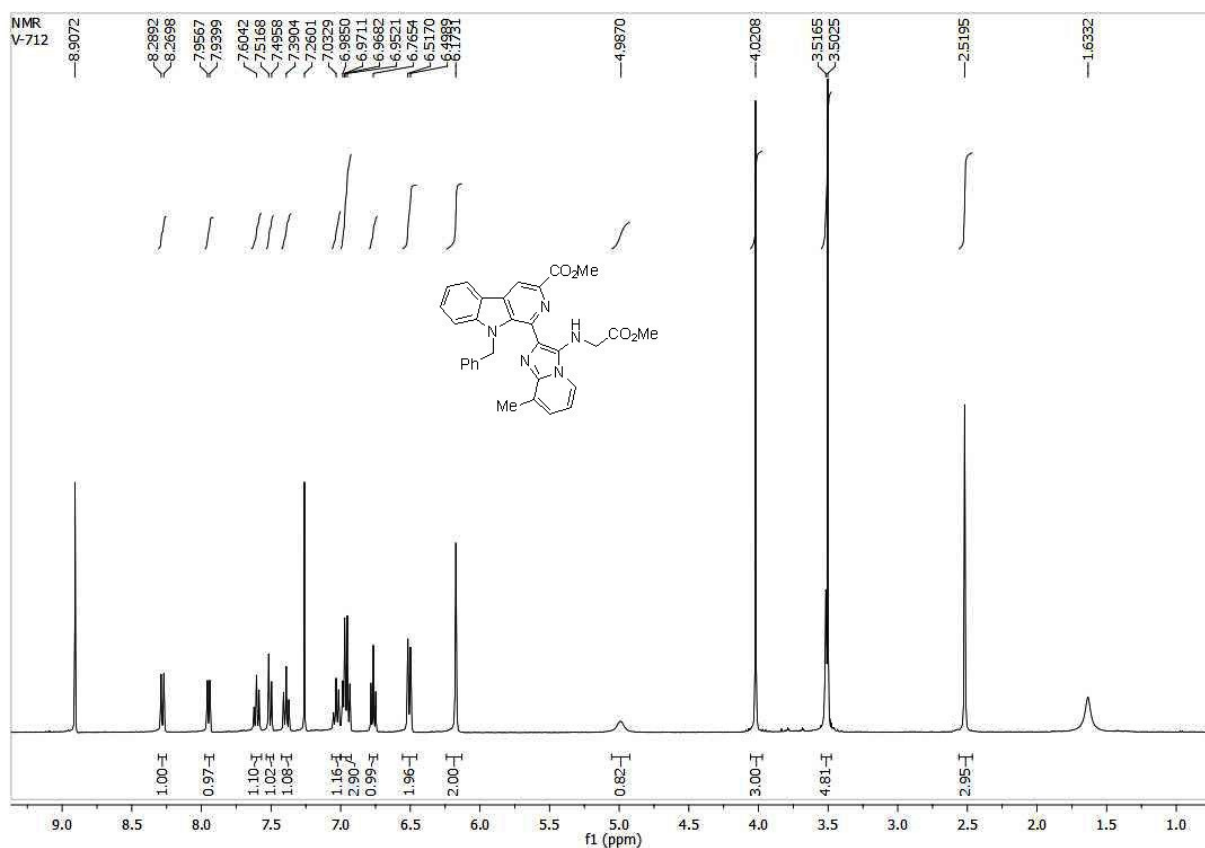


Fig. S 31. ^1H -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cCX**).

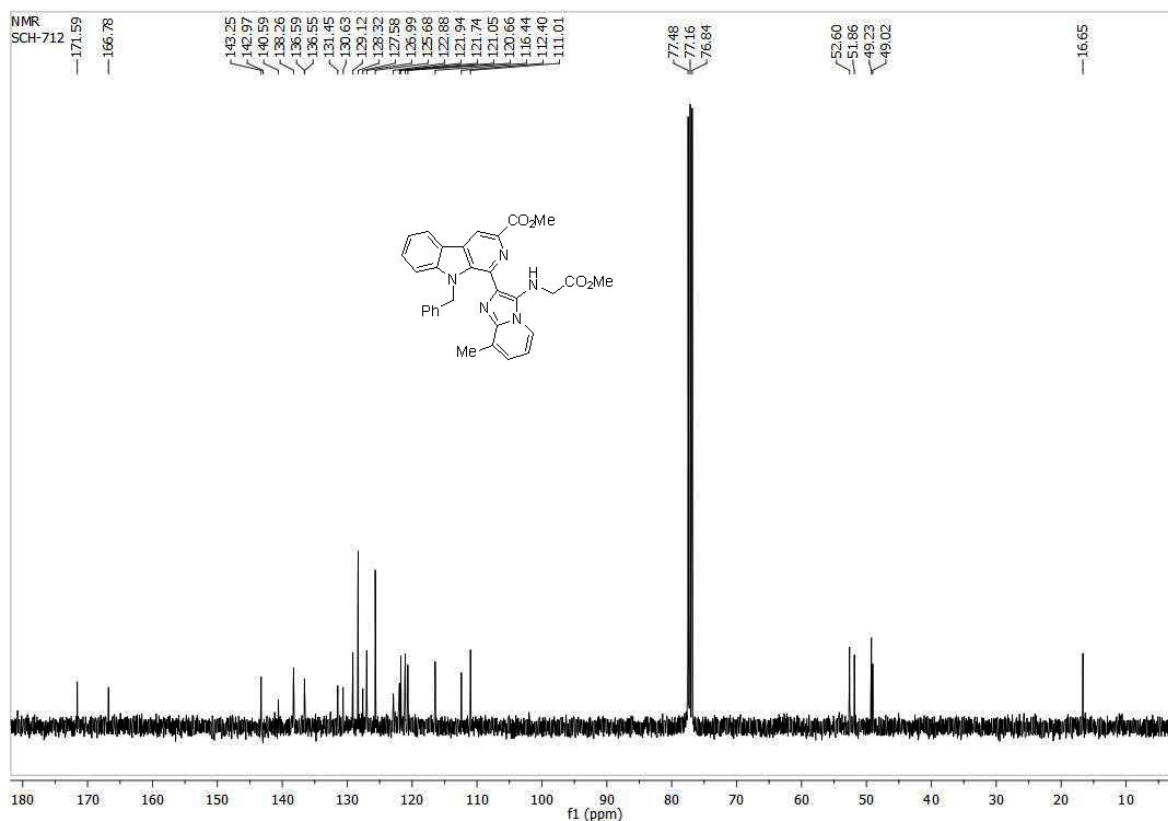


Fig. S 32. ^{13}C -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cCX**).

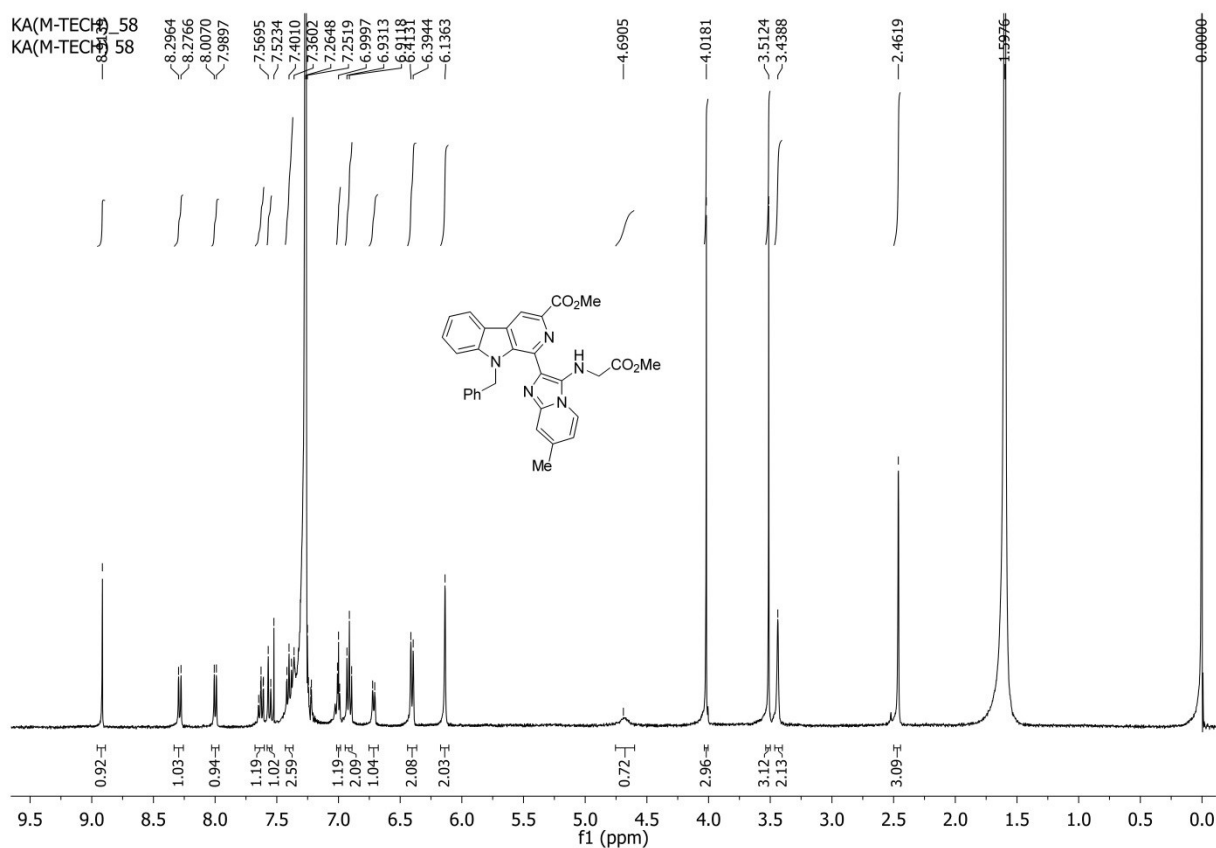


Fig. S 33. ^1H -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cDX**).

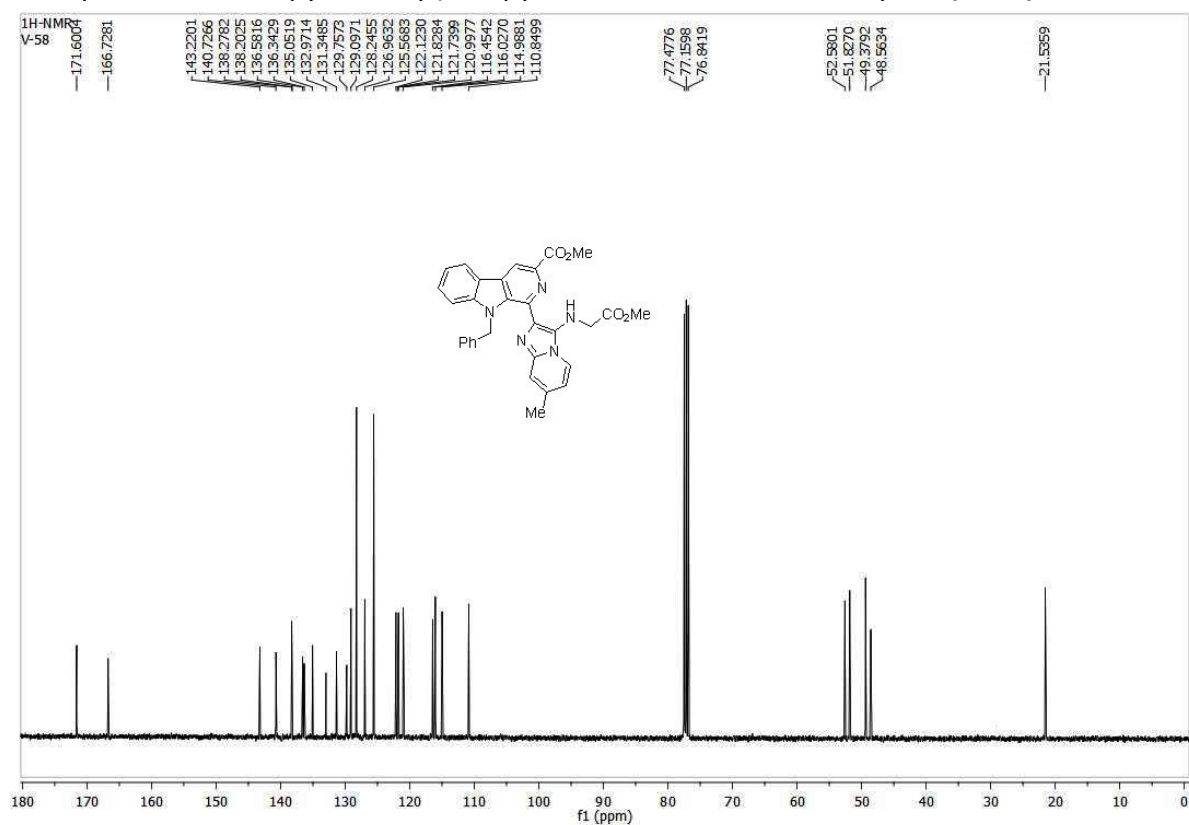


Fig. S 34. ^{13}C -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cDX**).

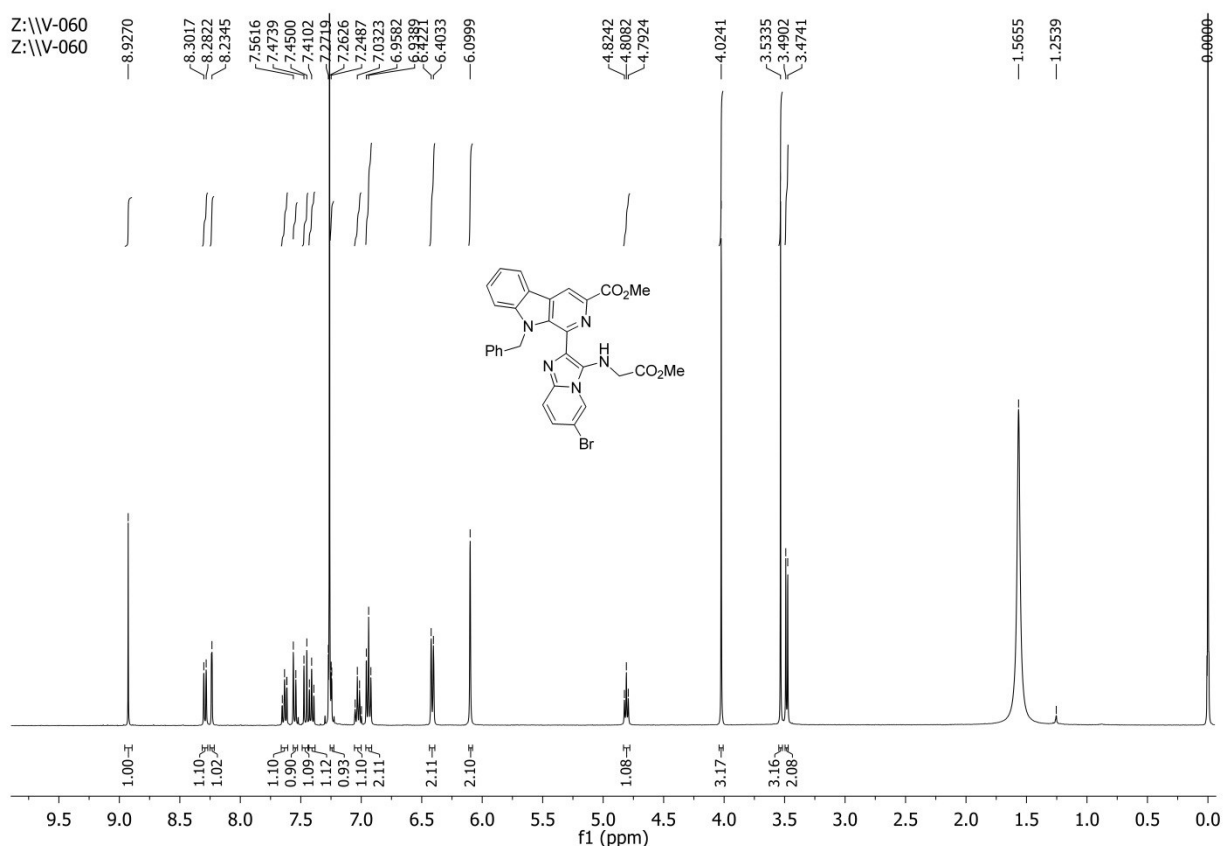


Fig. S 35. ^1H -NMR of methyl 9-benzyl-1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cEX**).

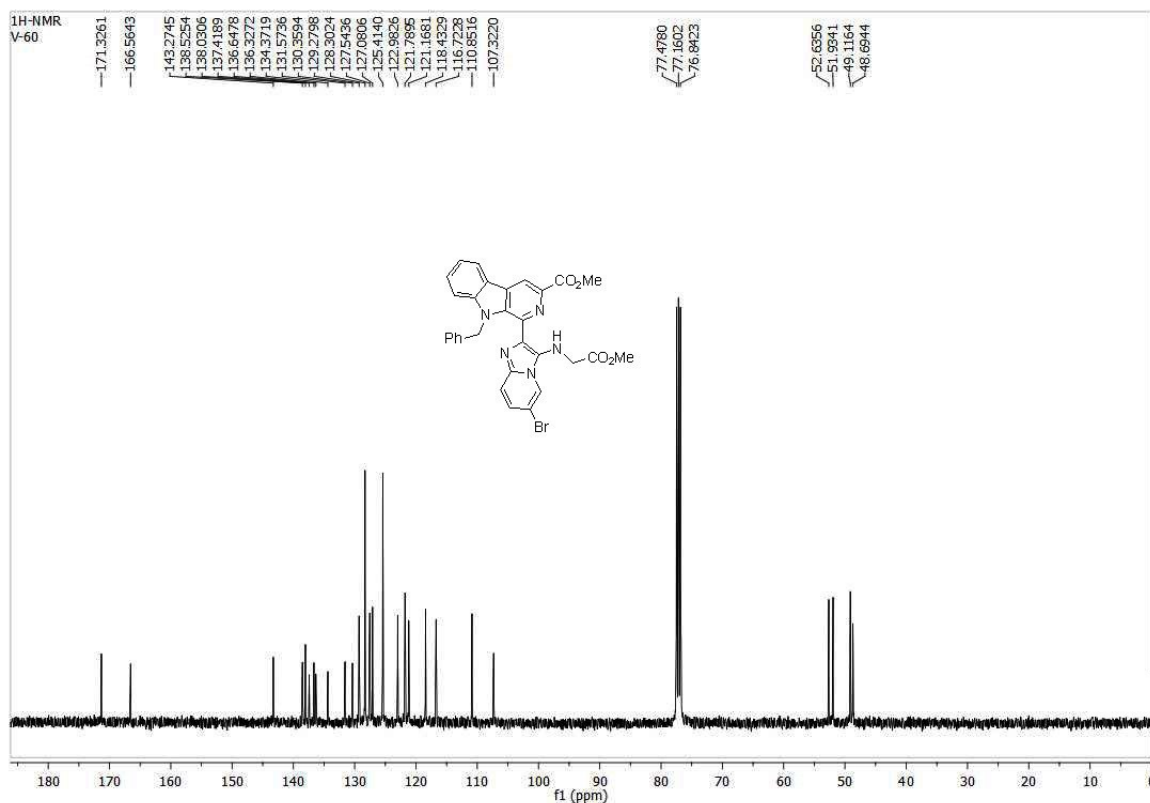


Fig. S 36. ^{13}C -NMR of methyl 9-benzyl-1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cEX**).

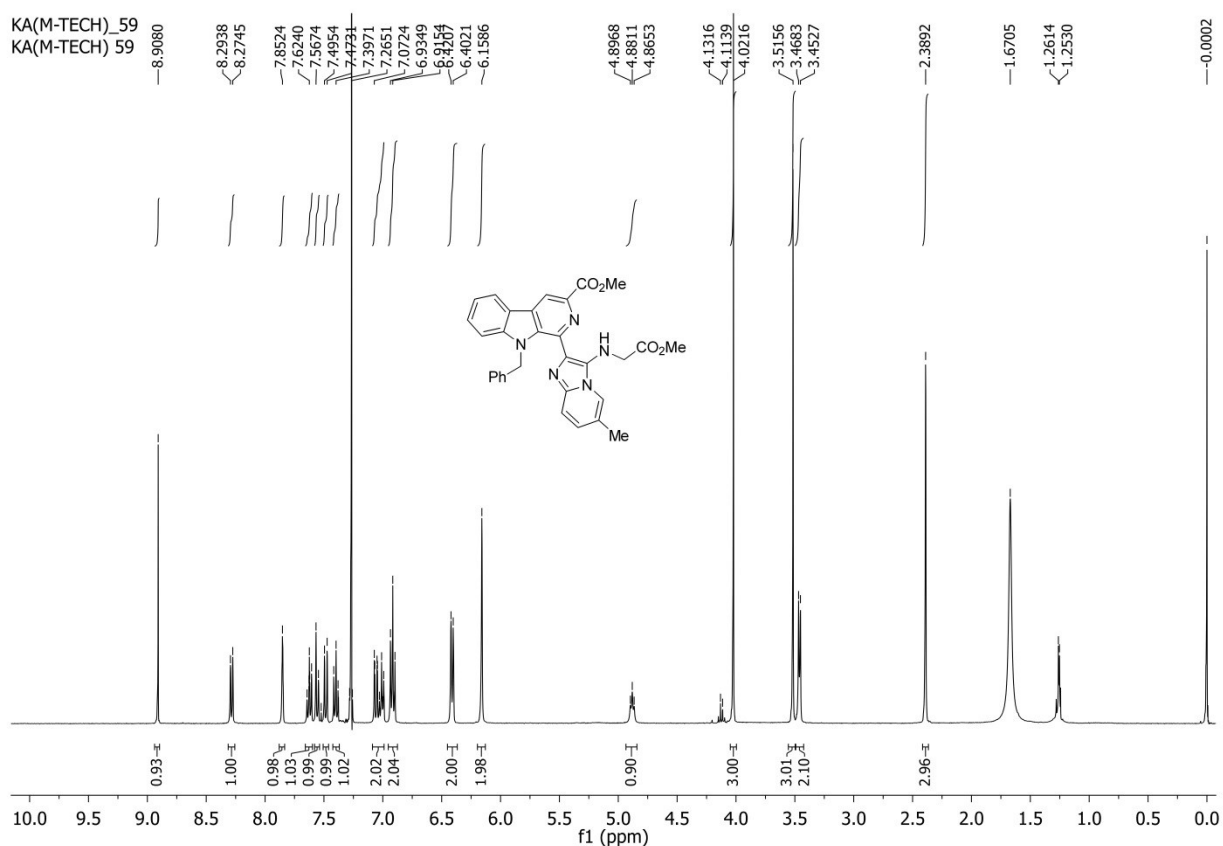


Fig. S 37. ^1H -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cGX**).

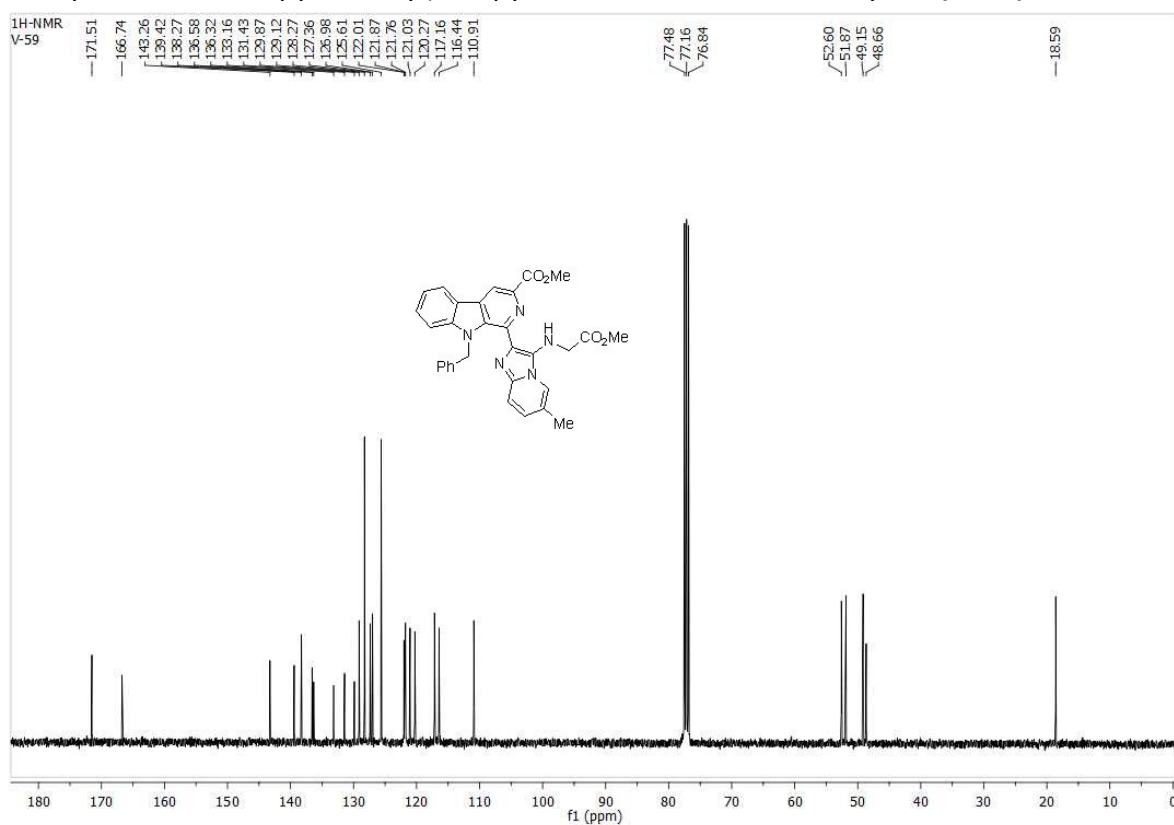


Fig. S 38. ^{13}C -NMR of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)-6-methylimidazo[1,2-*a*]pyridin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6cGX**).

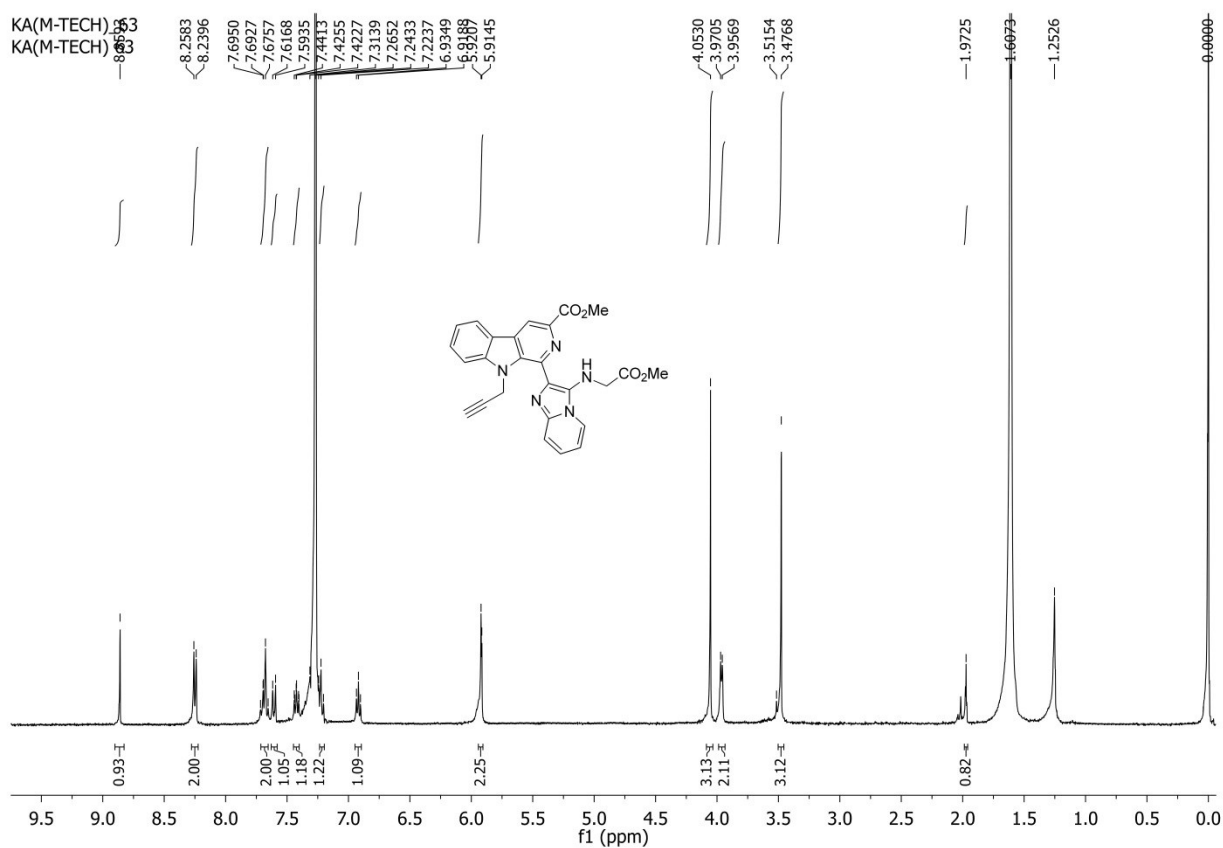


Fig. S 39. ^1H -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dAX**).

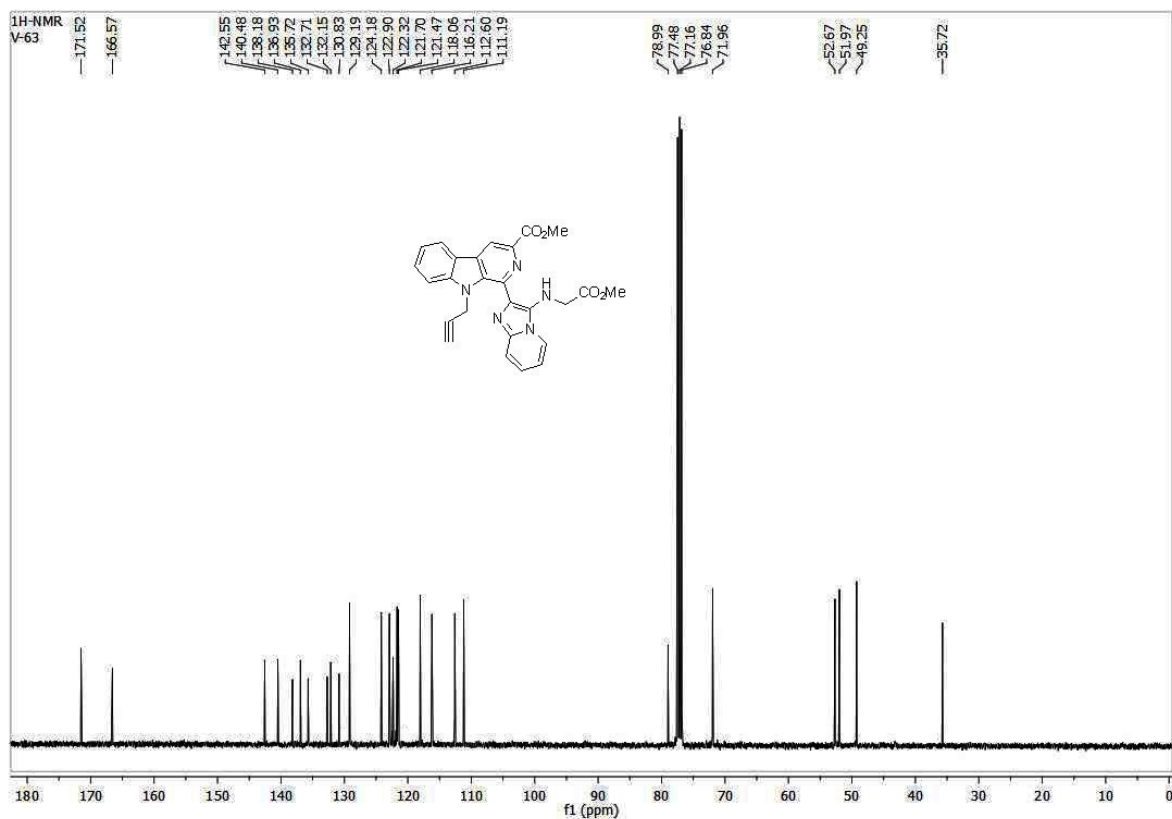


Fig. S 40. ^{13}C -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dAX**).

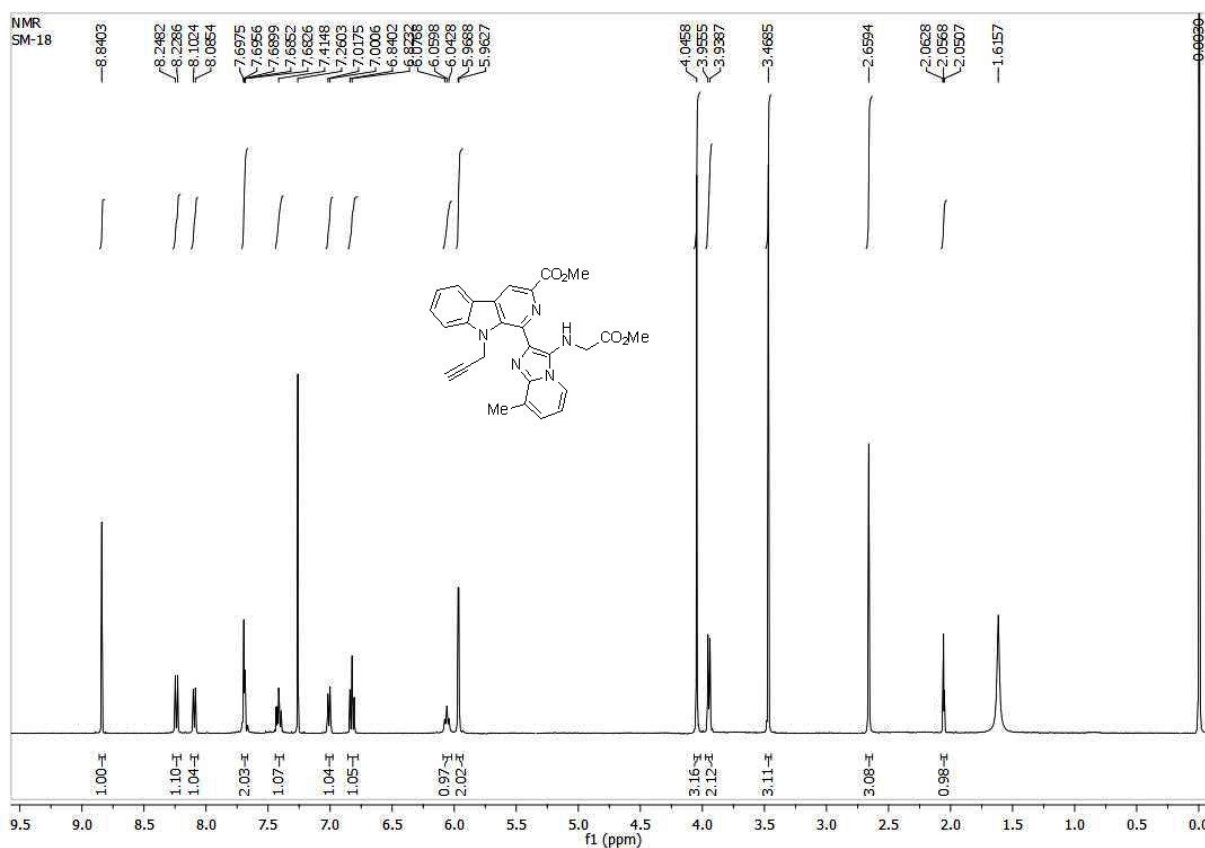


Fig. S 41. ^1H -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dCX**).

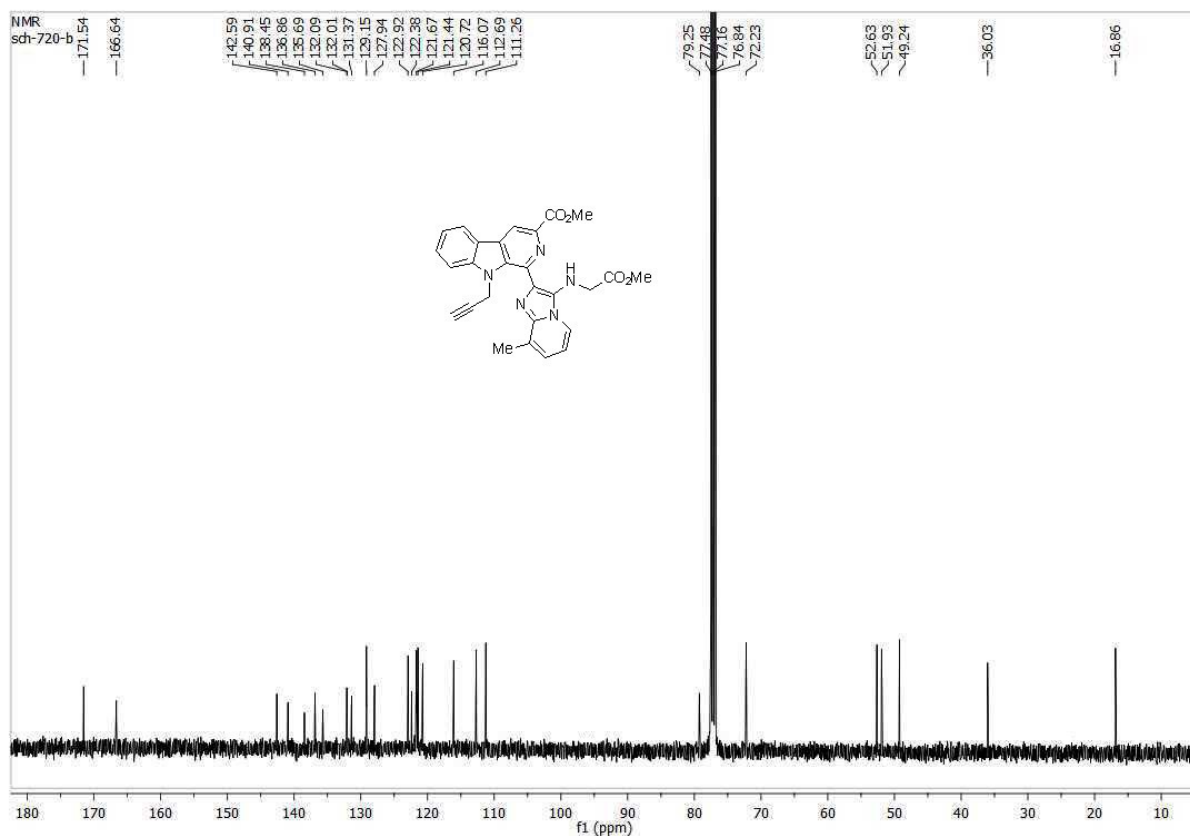


Fig. S 42. ^{13}C -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-8-methylimidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dCX**).

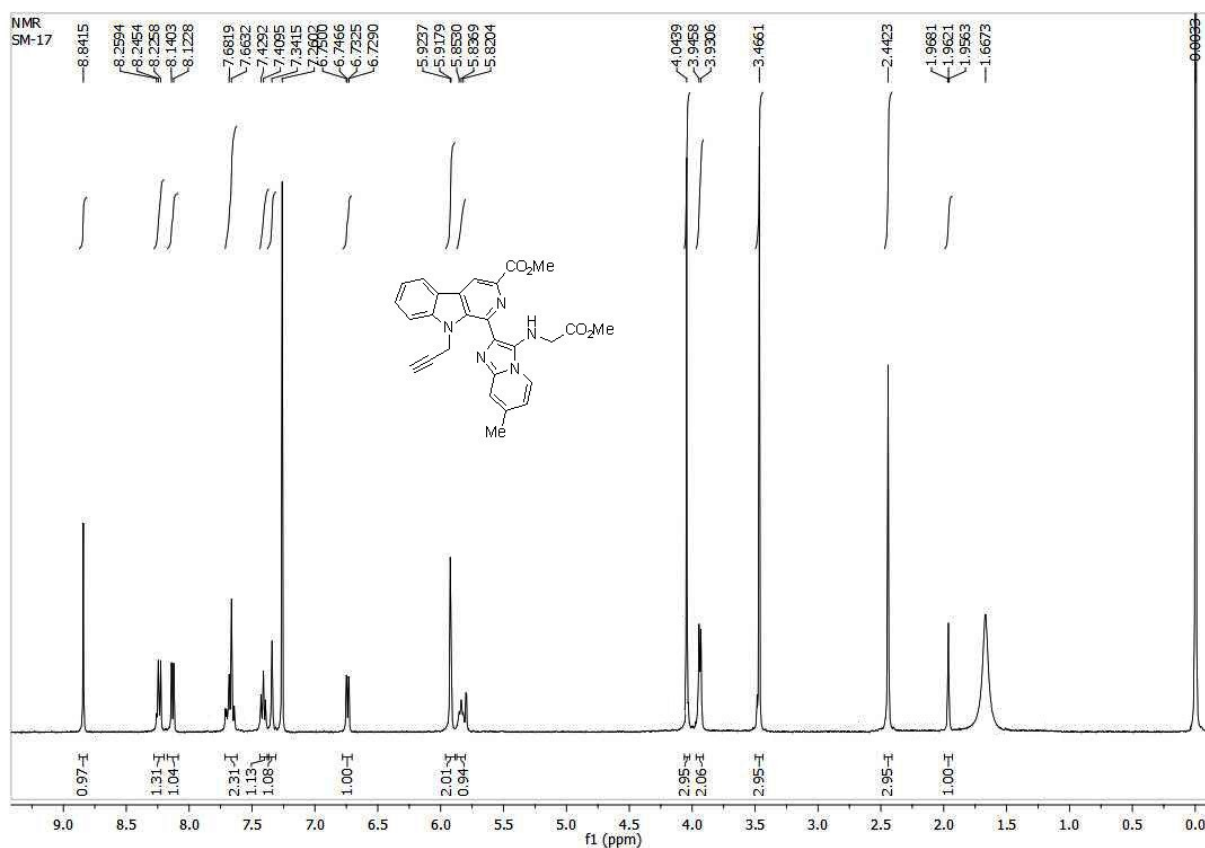


Fig. S 43. ^1H -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dDX**).

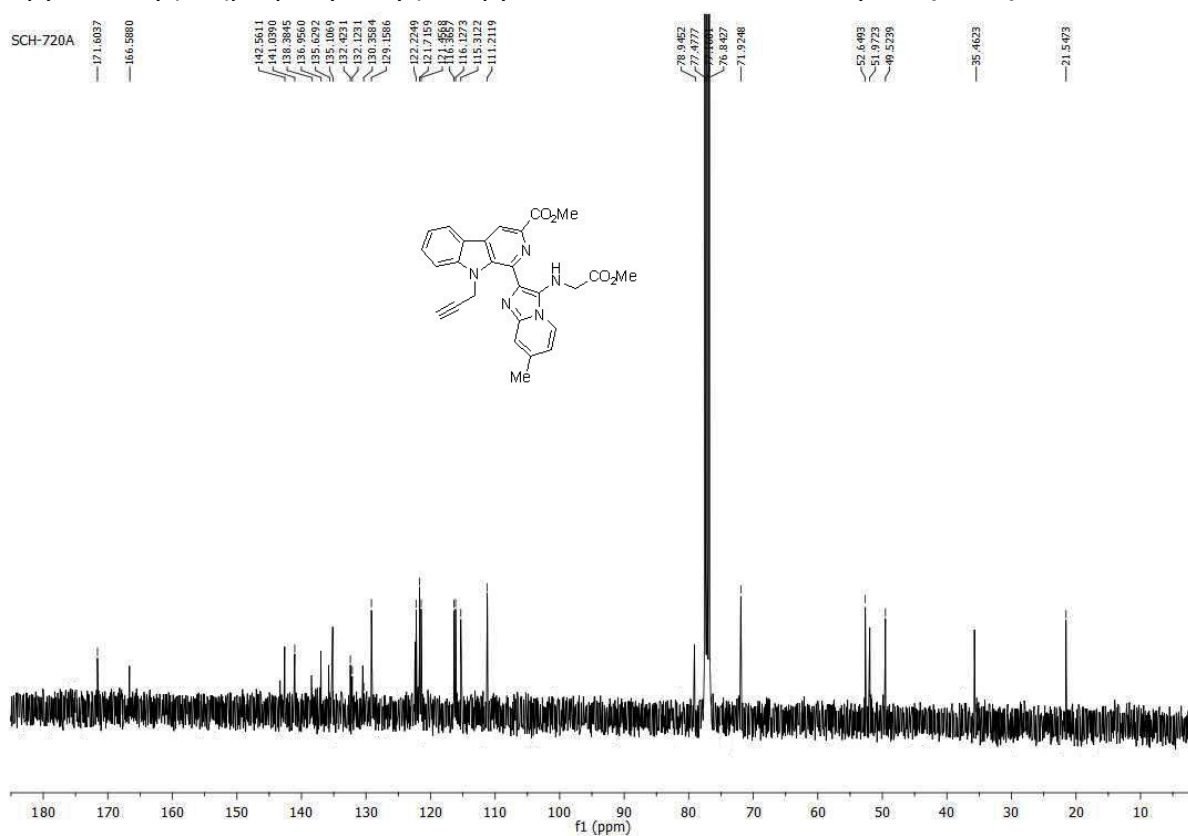


Fig. S 44. ^{13}C -NMR of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)-7-methylimidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dDX**).

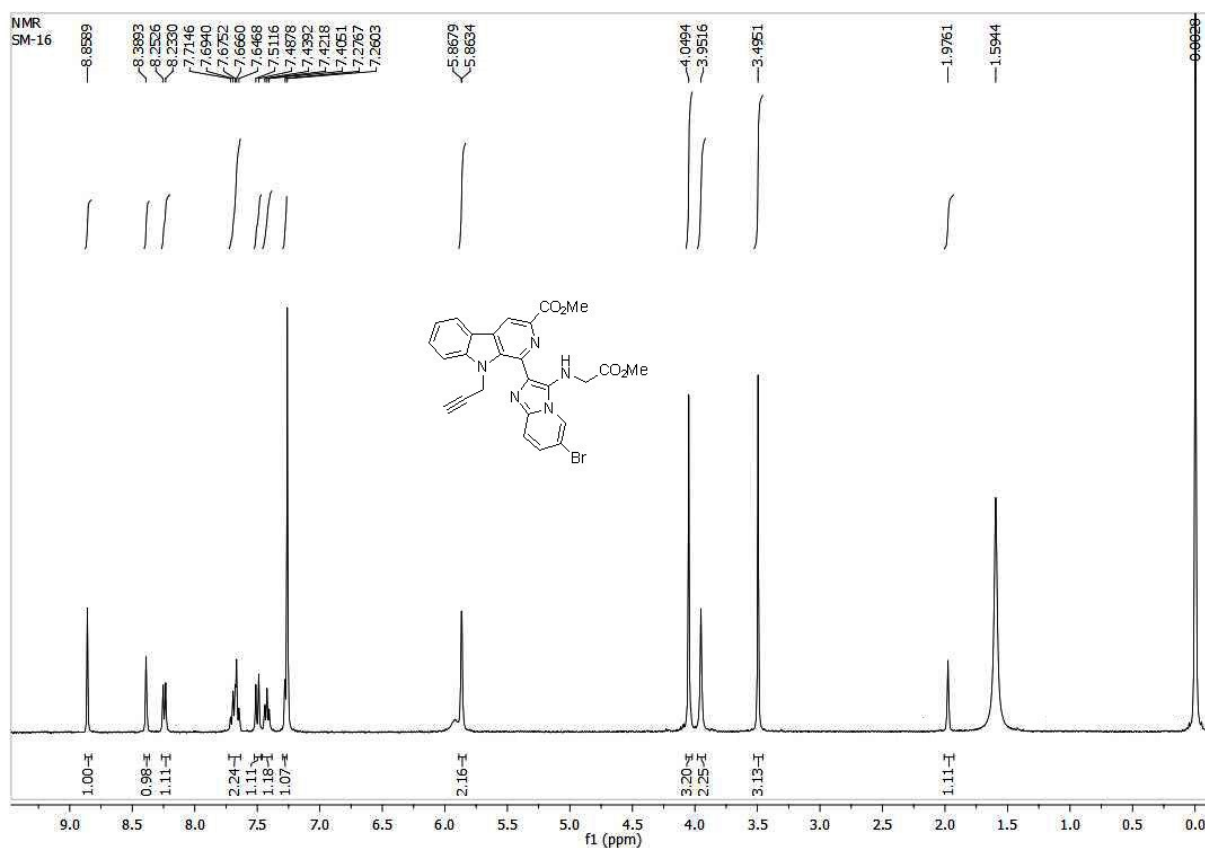


Fig. S 45. ^1H -NMR of methyl 1-(6-bromo-3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-(prop-2-yn-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**6dEX**).

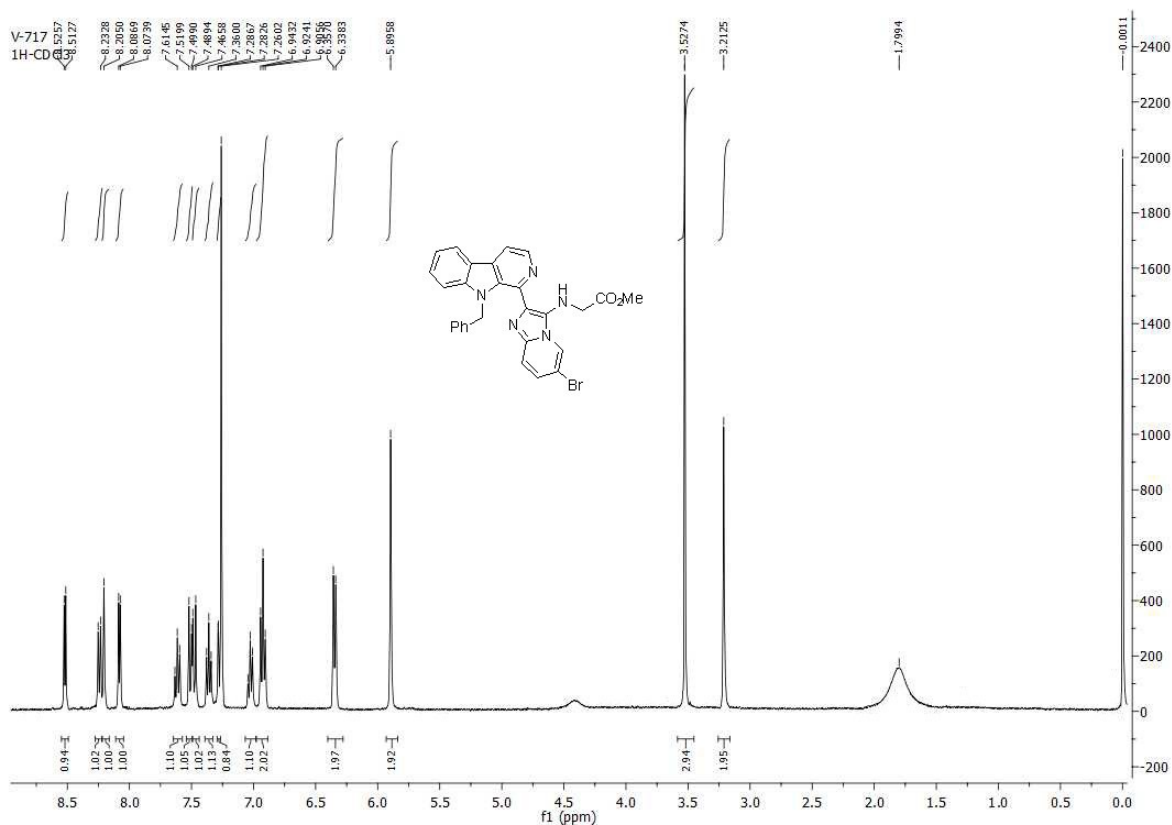


Fig. S 46. ¹H-NMR of methyl 2-((2-(9-benzyl-9H-pyrido[3,4-b]indol-1-yl)-6-bromoimidazo[1,2-a]pyridin-3-yl)amino)acetate (**6eAX**).

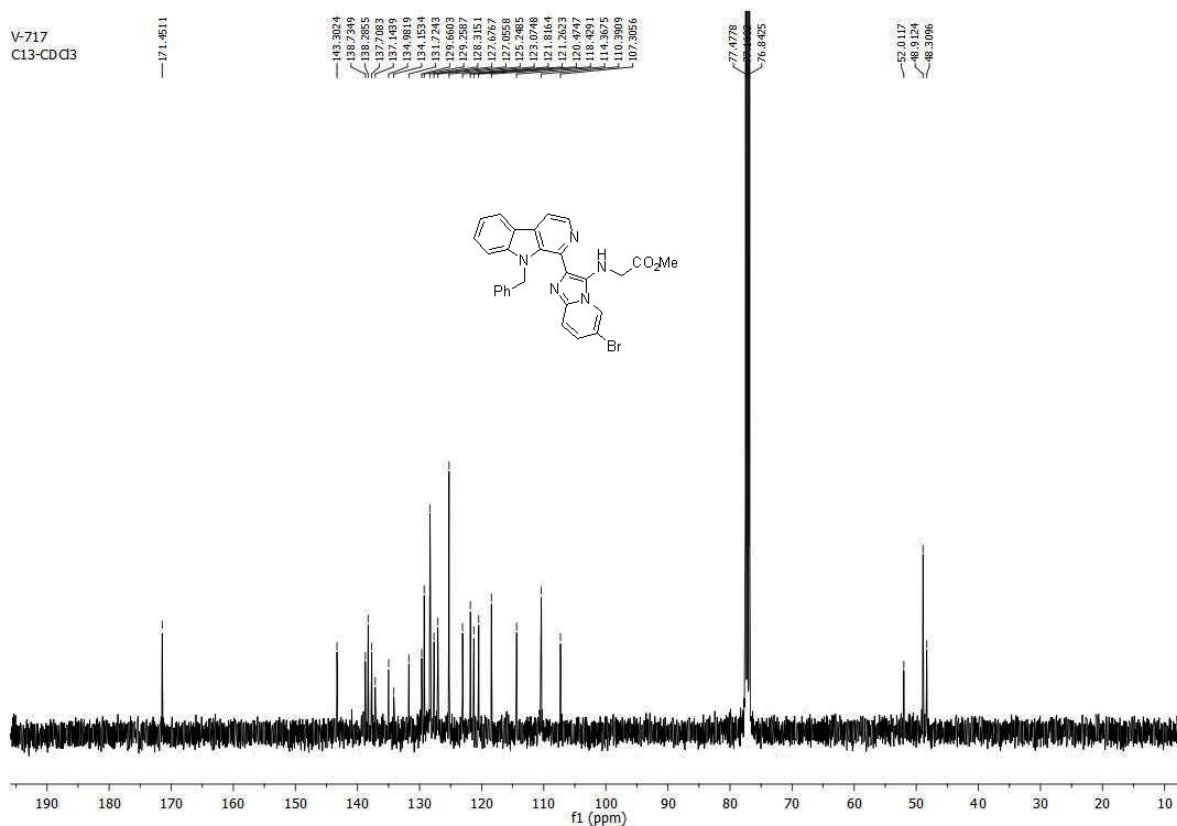


Fig. S 47. ¹³C-NMR of methyl 2-((2-(9-benzyl-9H-pyrido[3,4-b]indol-1-yl)-6-bromoimidazo[1,2-a]pyridin-3-yl)amino)acetate (**6eAX**).

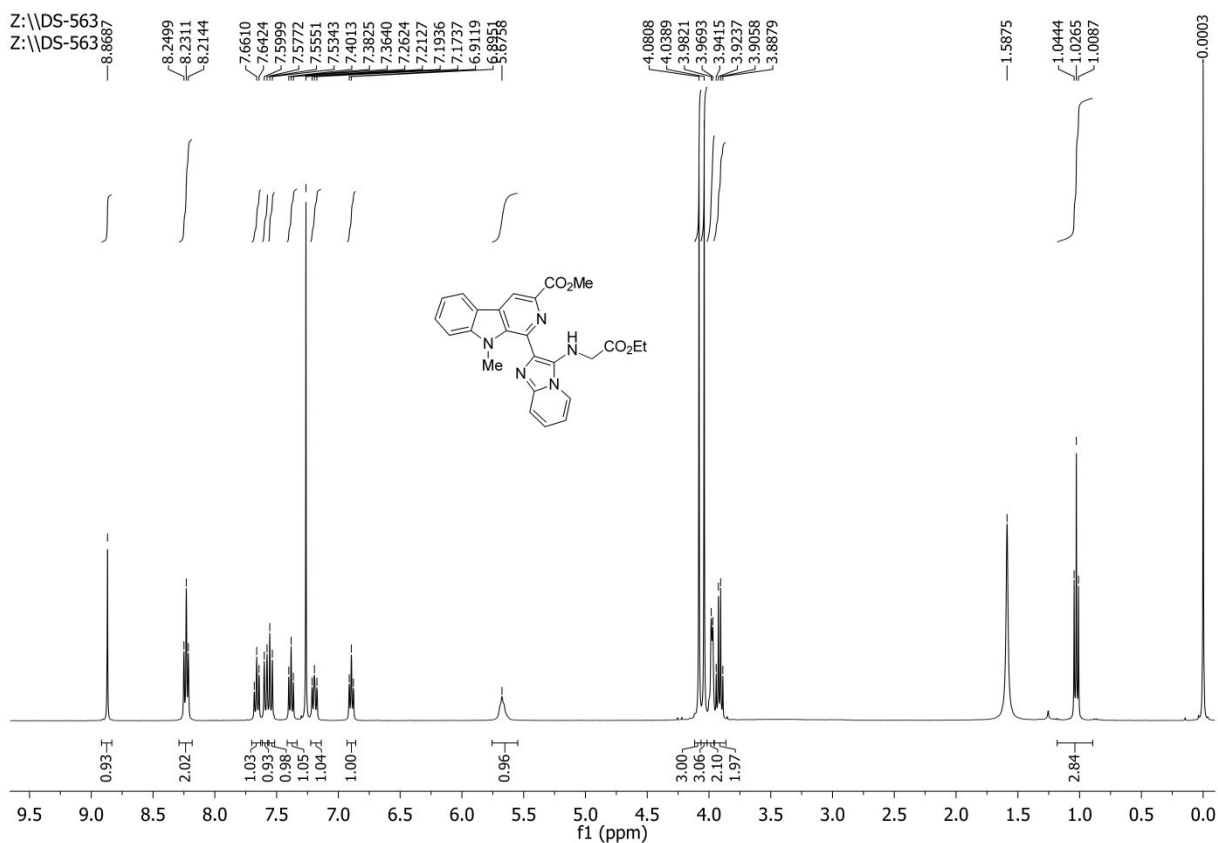


Fig. S 48. $^1\text{H-NMR}$ of methyl 1-(3-((2-ethoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate. (**6aAY**).

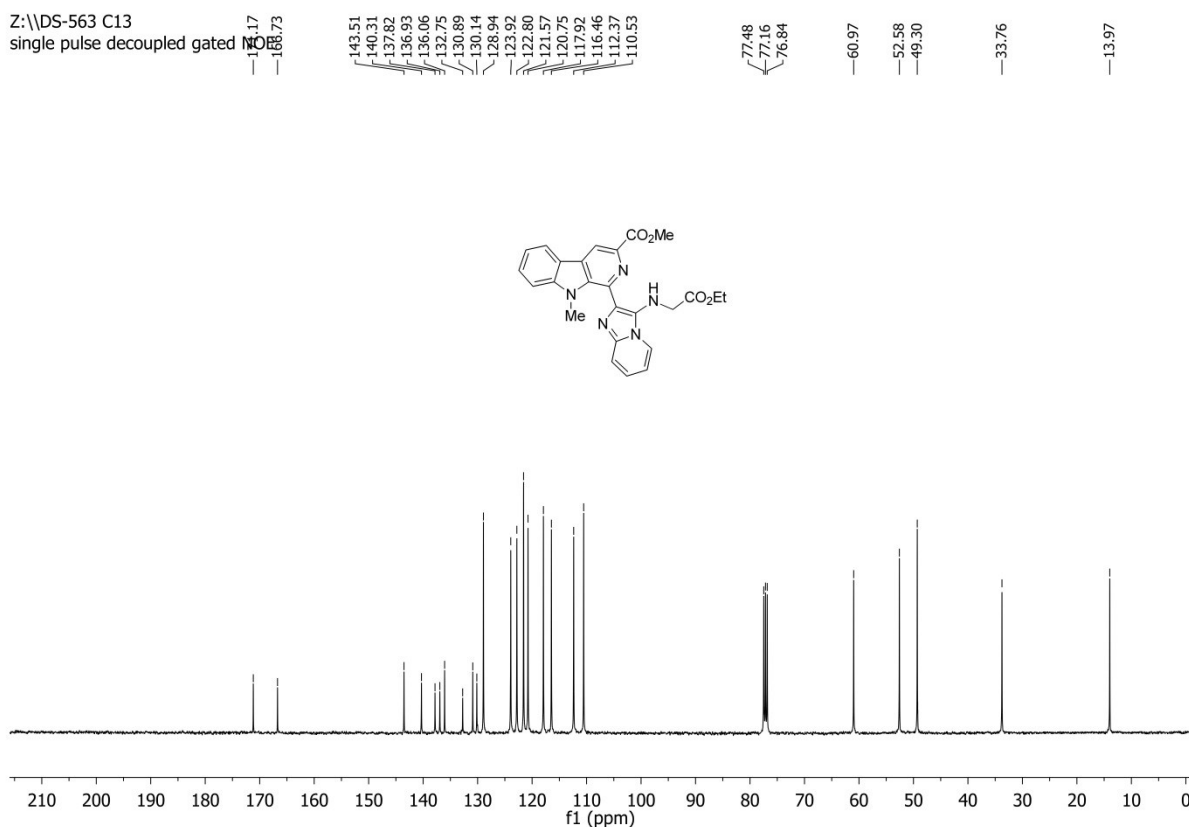


Fig. S 49. $^{13}\text{C-NMR}$ of methyl 1-(3-((2-ethoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyridin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate. (**6aAY**).

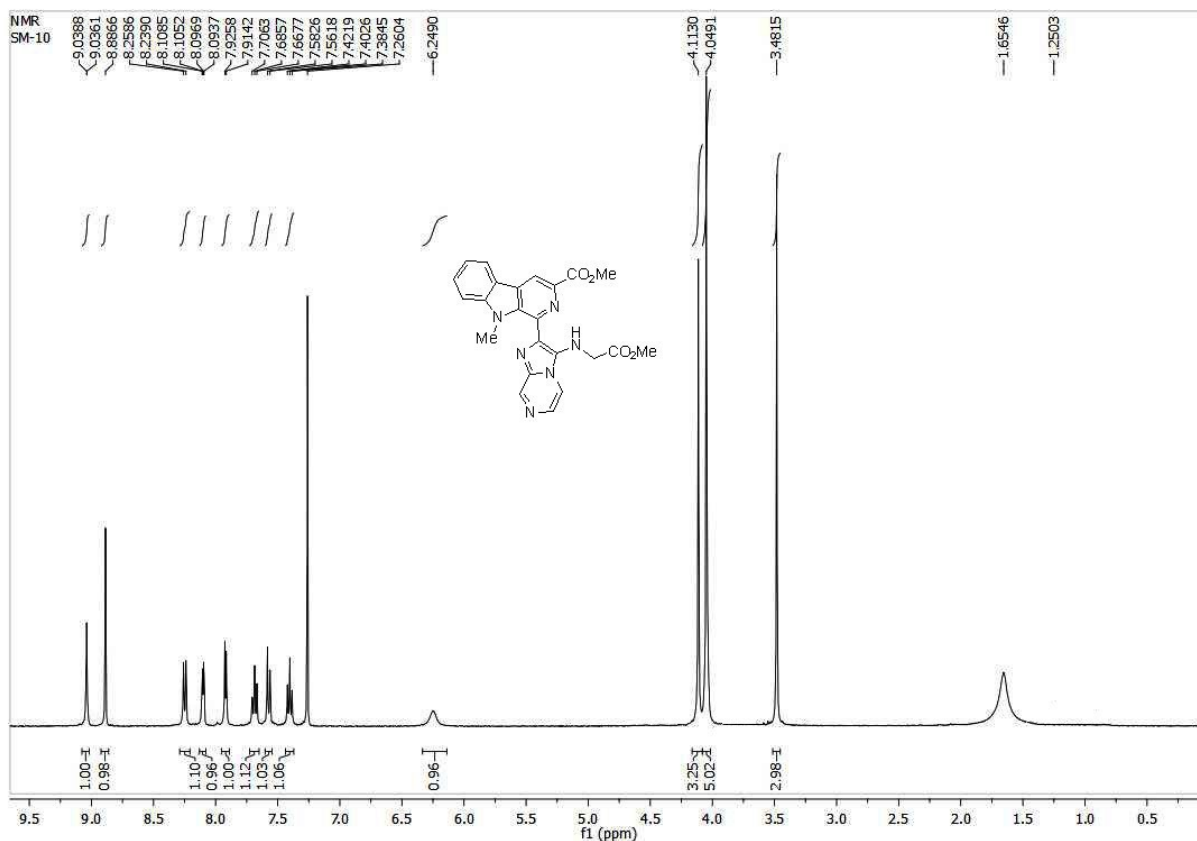


Fig. S 50. $^1\text{H-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7aHX 708**).

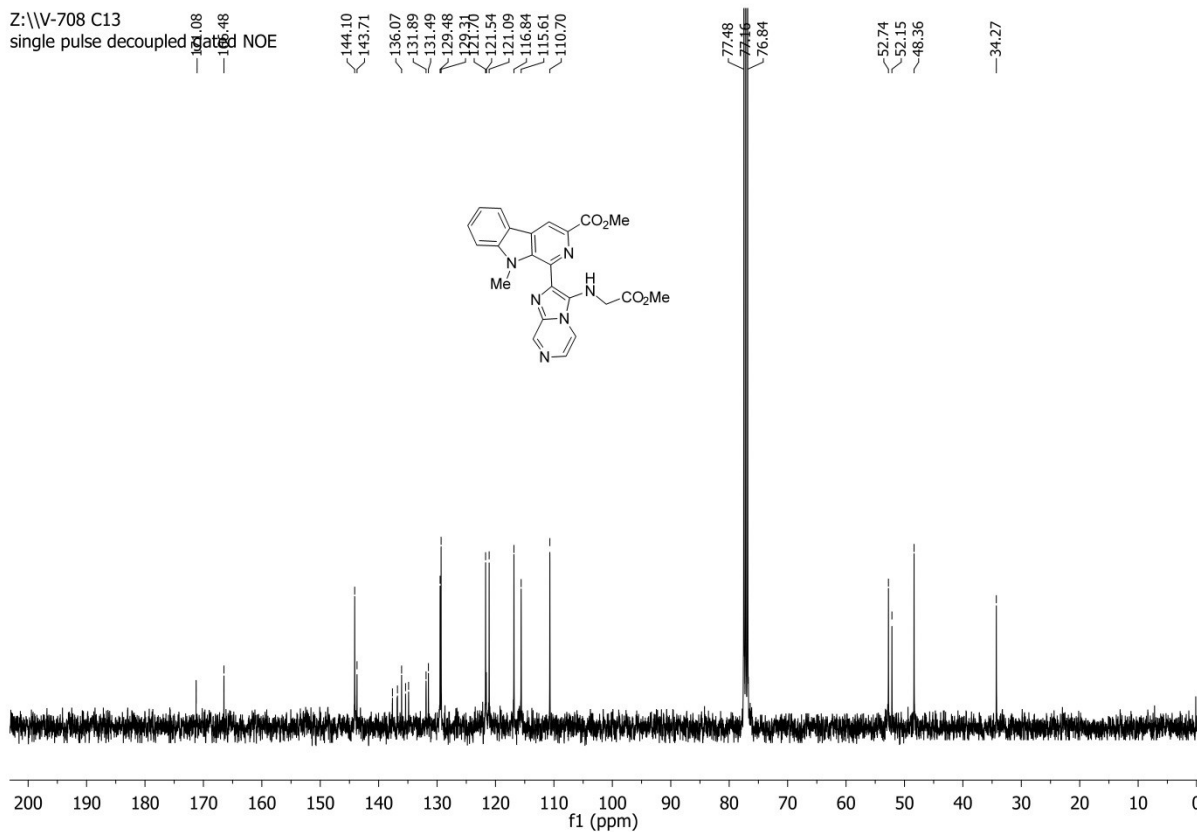


Fig. S 51. $^{13}\text{C-NMR}$ of methyl 1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7aHX**).

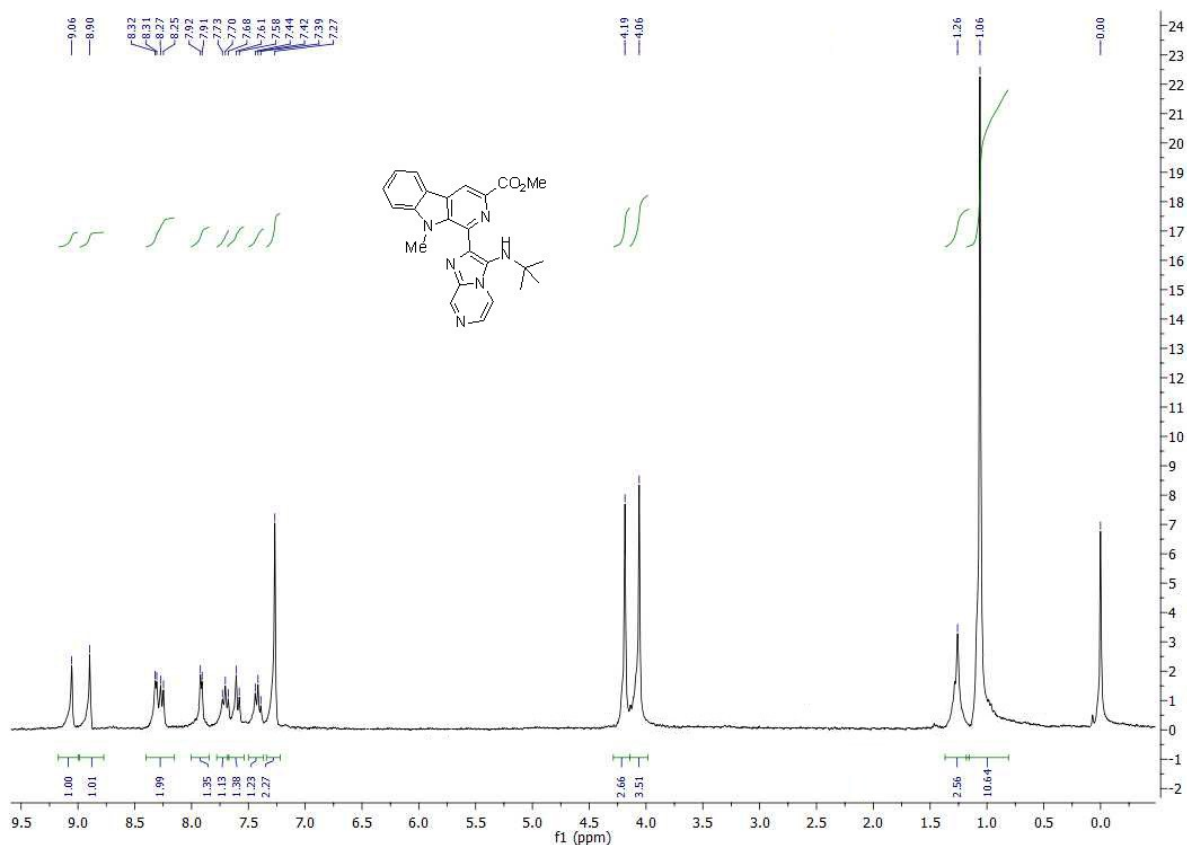


Fig. S 52. ¹H-NMR of methyl 1-(3-(*tert*-butylamino)imidazo[1,2-*a*]pyrazin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**7aHZ**).

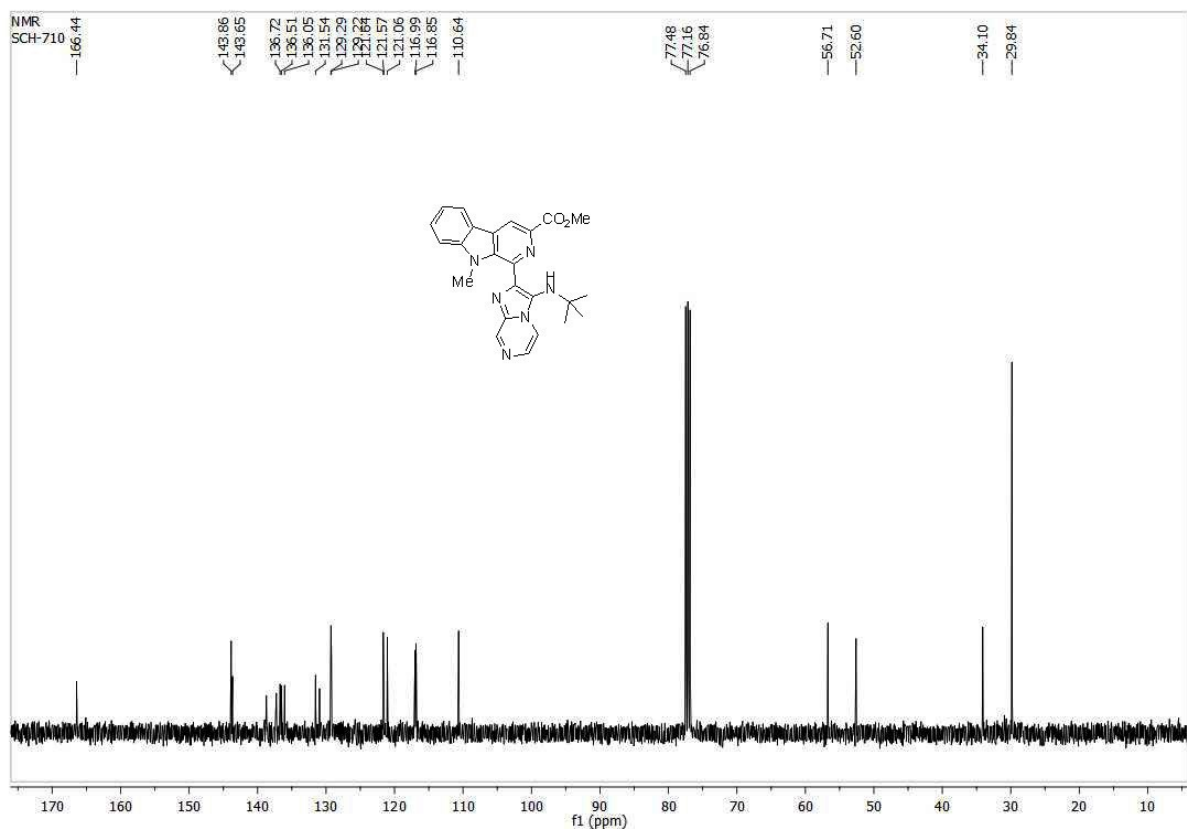


Fig. S 53. ¹³C-NMR of methyl 1-(3-(*tert*-butylamino)imidazo[1,2-*a*]pyrazin-2-yl)-9-methyl-9H-pyrido[3,4-*b*]indole-3-carboxylate (**7aHZ**).

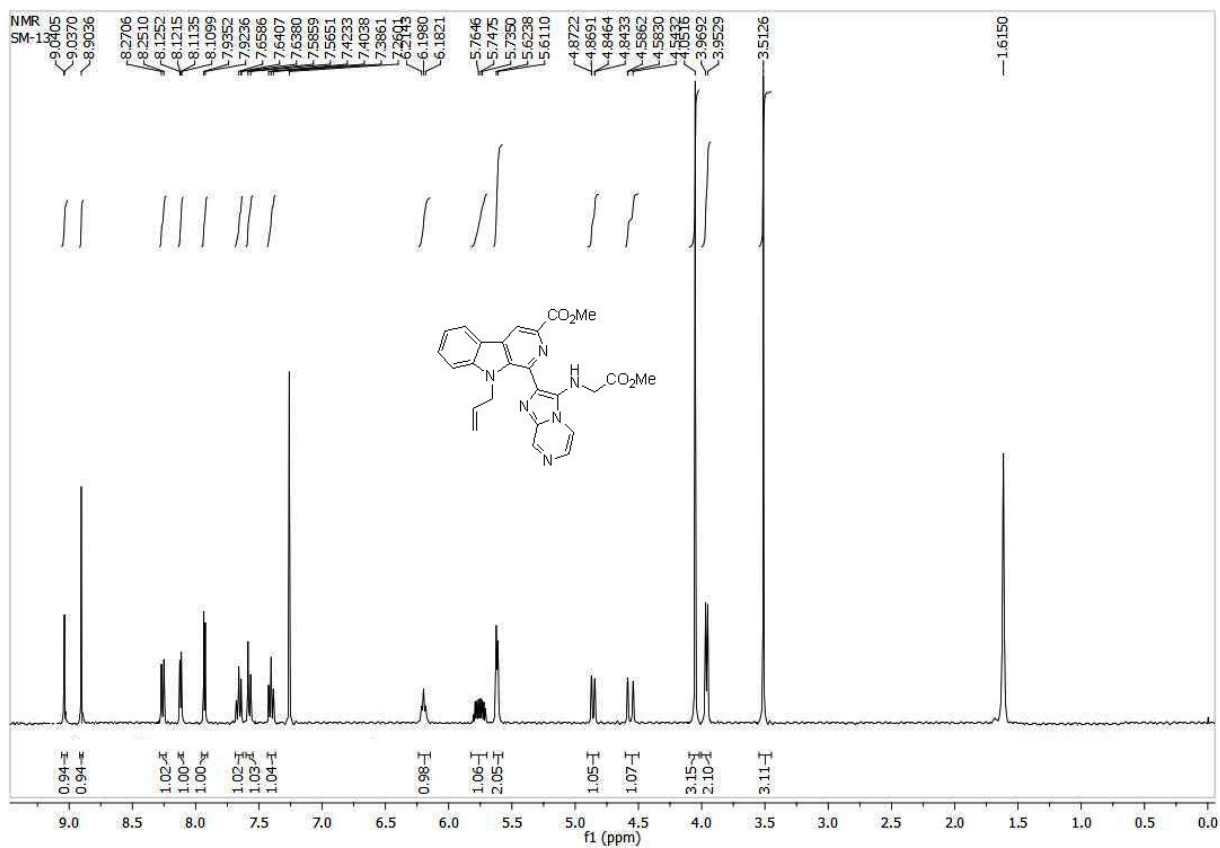


Fig. S 54. $^1\text{H-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7bHX**).

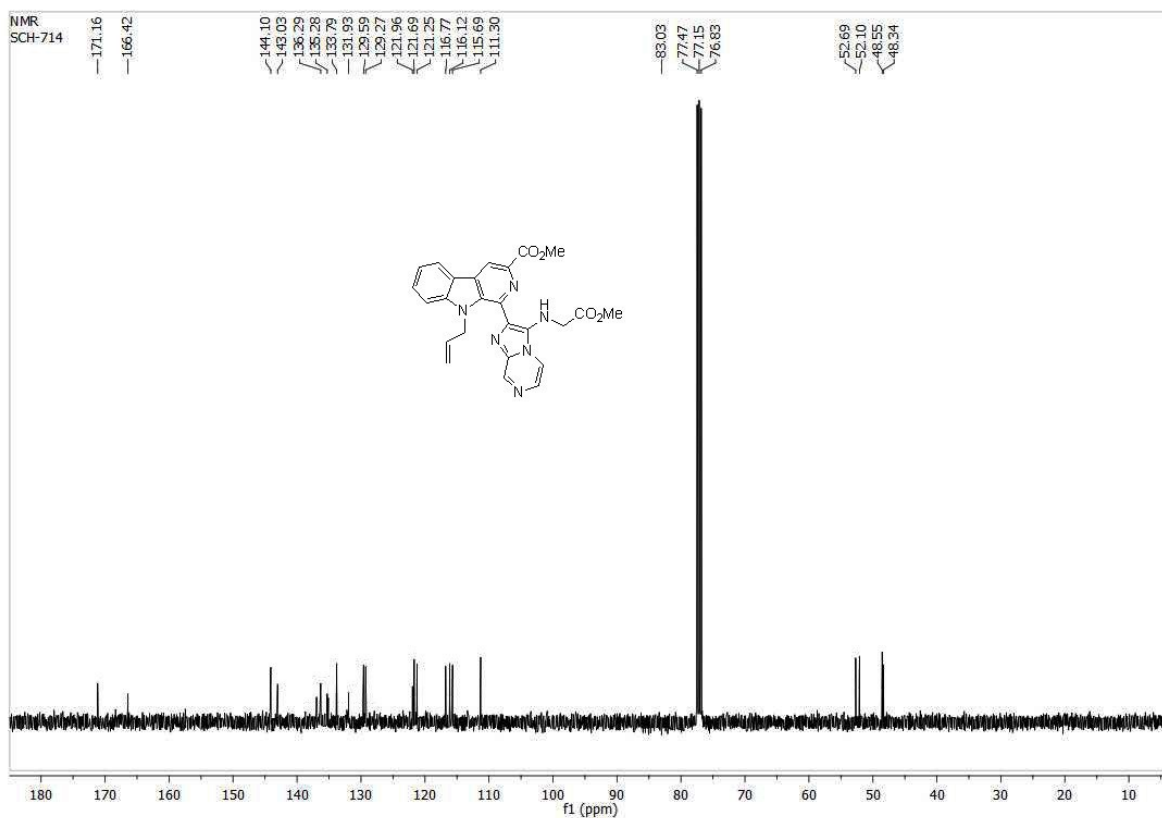


Fig. S 55. $^{13}\text{C-NMR}$ of methyl 9-allyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7bHX**).

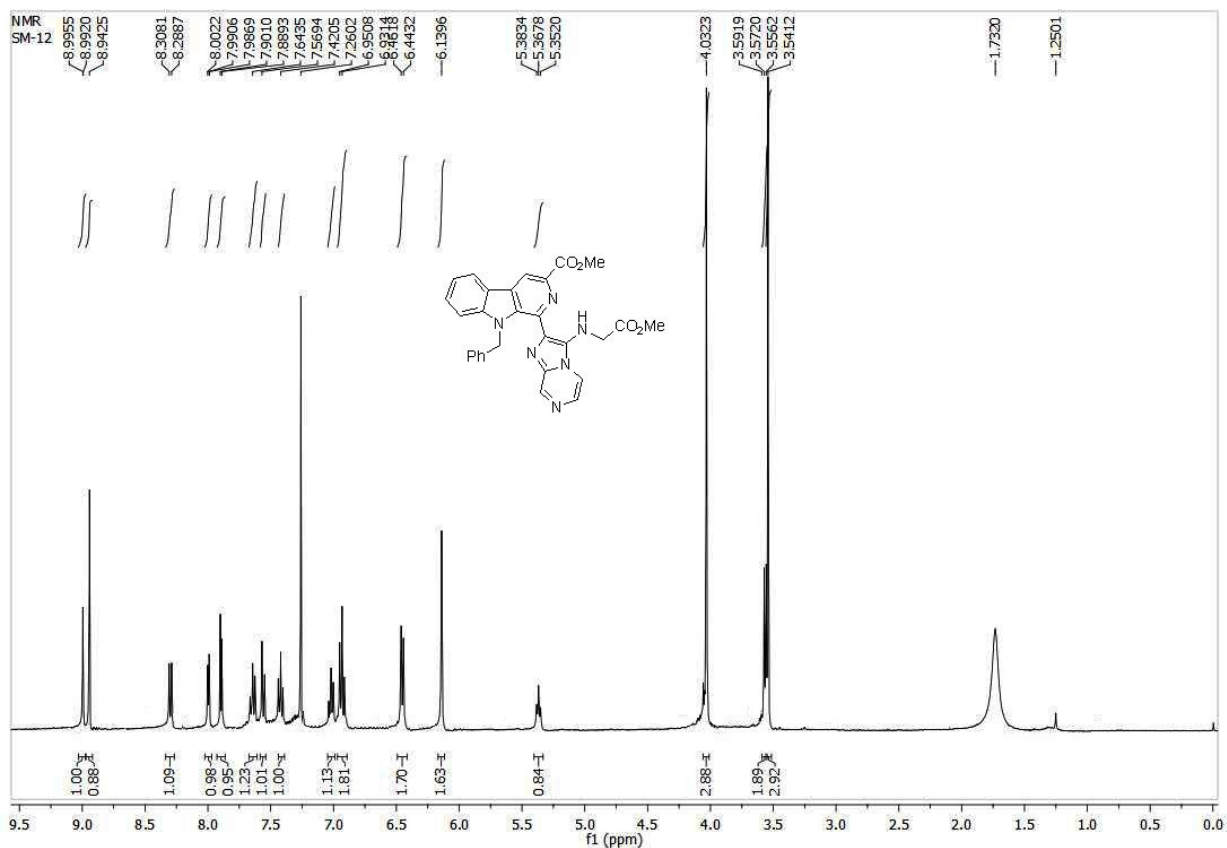


Fig. S 56. $^1\text{H-NMR}$ of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7CHX**).

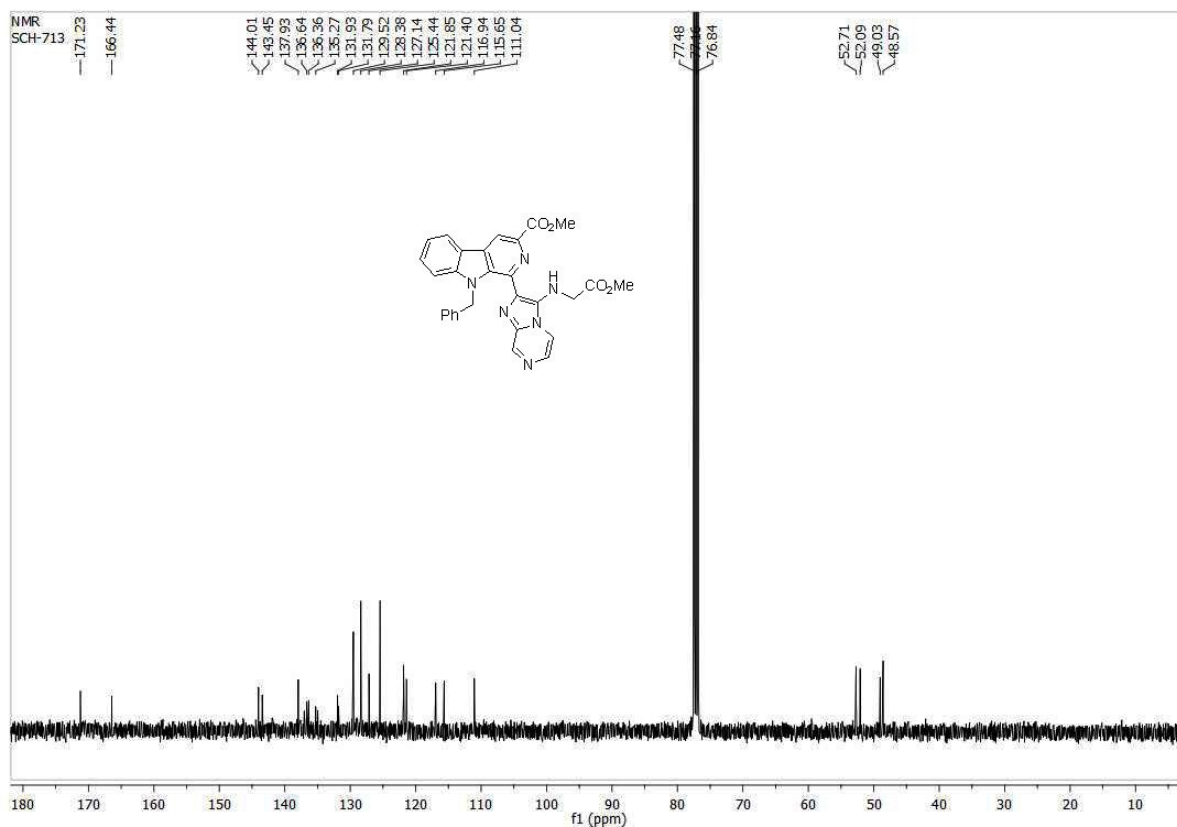


Fig. S 57. $^{13}\text{C-NMR}$ of methyl 9-benzyl-1-(3-((2-methoxy-2-oxoethyl)amino)imidazo[1,2-*a*]pyrazin-2-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (**7CHX**).