Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

$PhI(OAc)_2$-Mediated one-pot oxidative decarboxylation and aromatization of tetrahydro-*β*-carbolines: synthesis of norharmane, harmane, eudistomin U and eudistomin I

Ahmed Kamal*, Yellaiah Tangella, Kesari Lakshmi Manasa, Manda Sathish,
Vunnam Srinivasulu Jadala Chetna and Abdullah Alarifi

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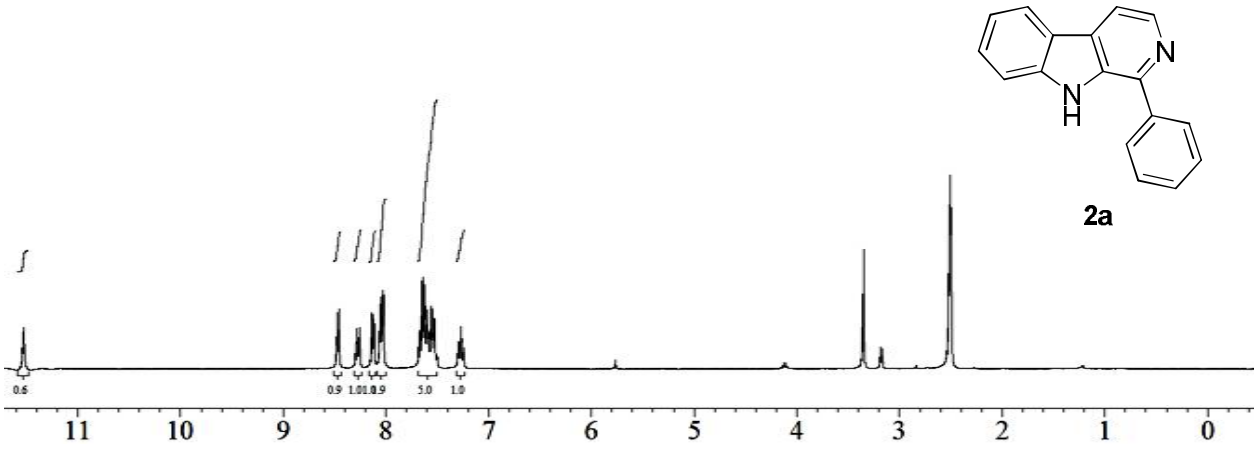
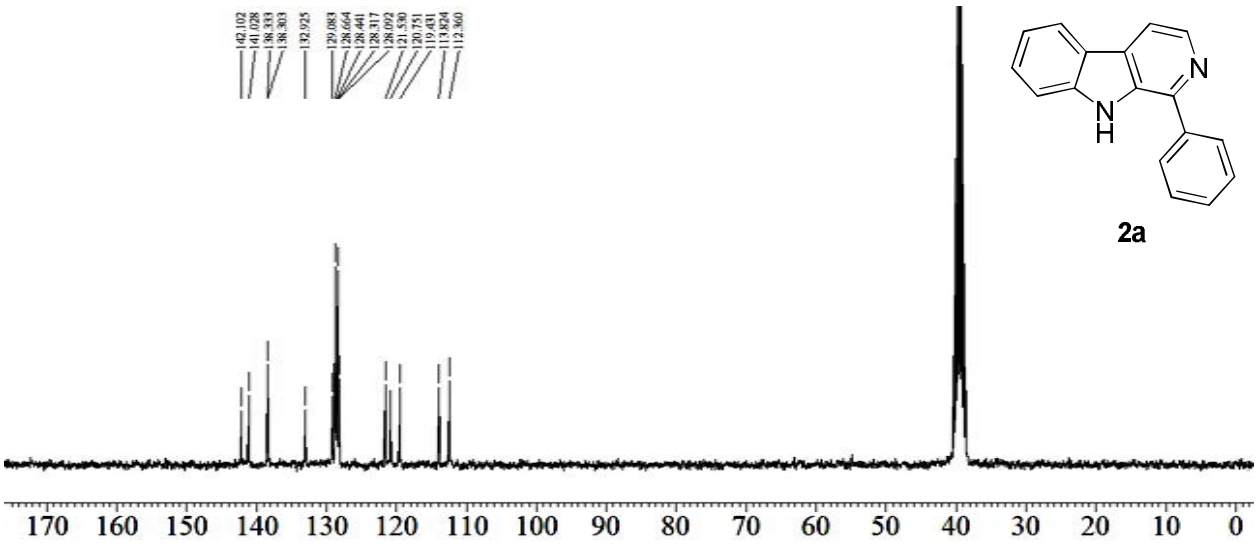
1-Phenyl-9*H*-pyrido[3,4-*b*]indole (2a)

^{1}H NMR (300 MHz, DMSO−d_6)

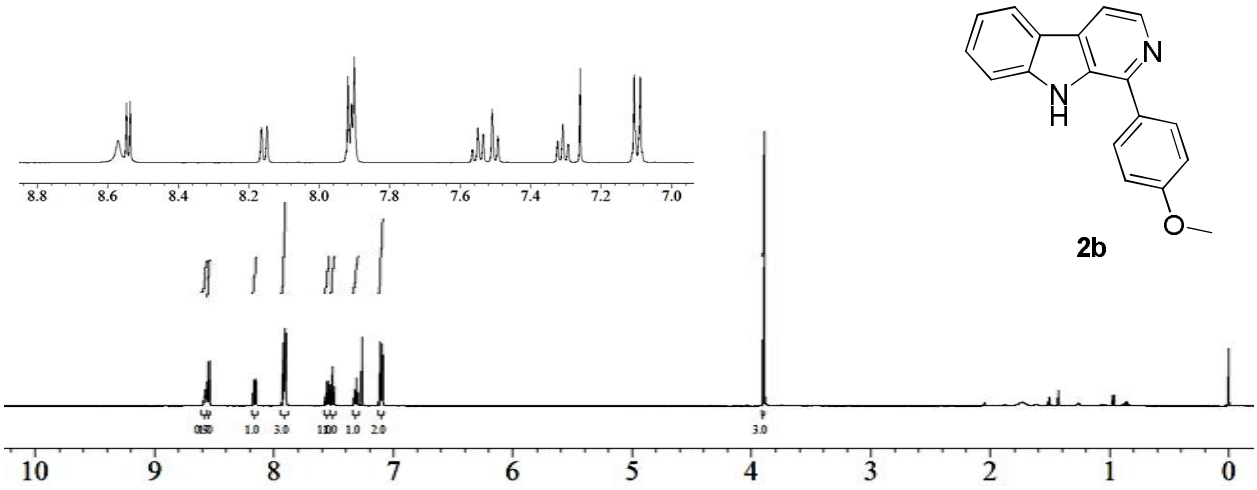


^{13}C NMR (75 MHz, DMSO−d_6)

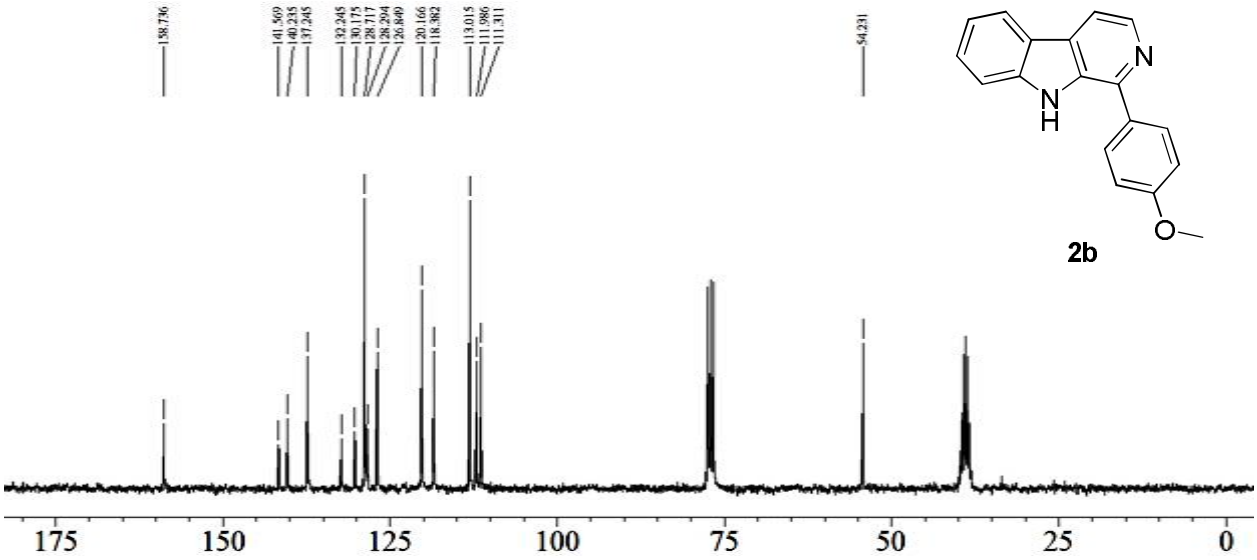


1-(4-Methoxyphenyl)-9*H*-pyrido[3,4-*b*]indole (2b)

^{1}H NMR (500 MHz, $CDCl_3$)

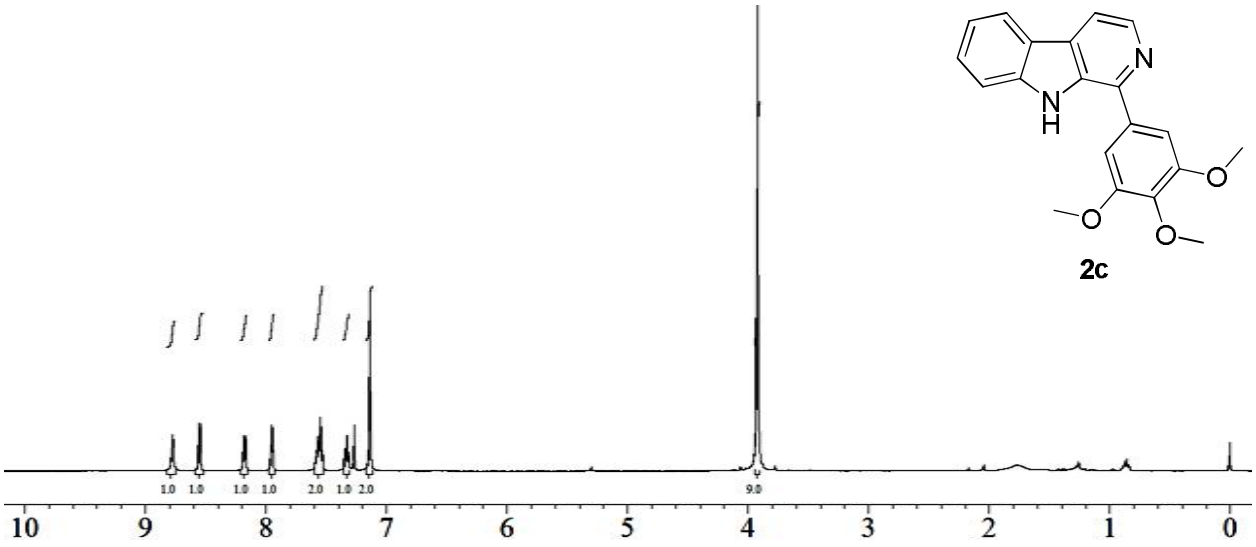


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

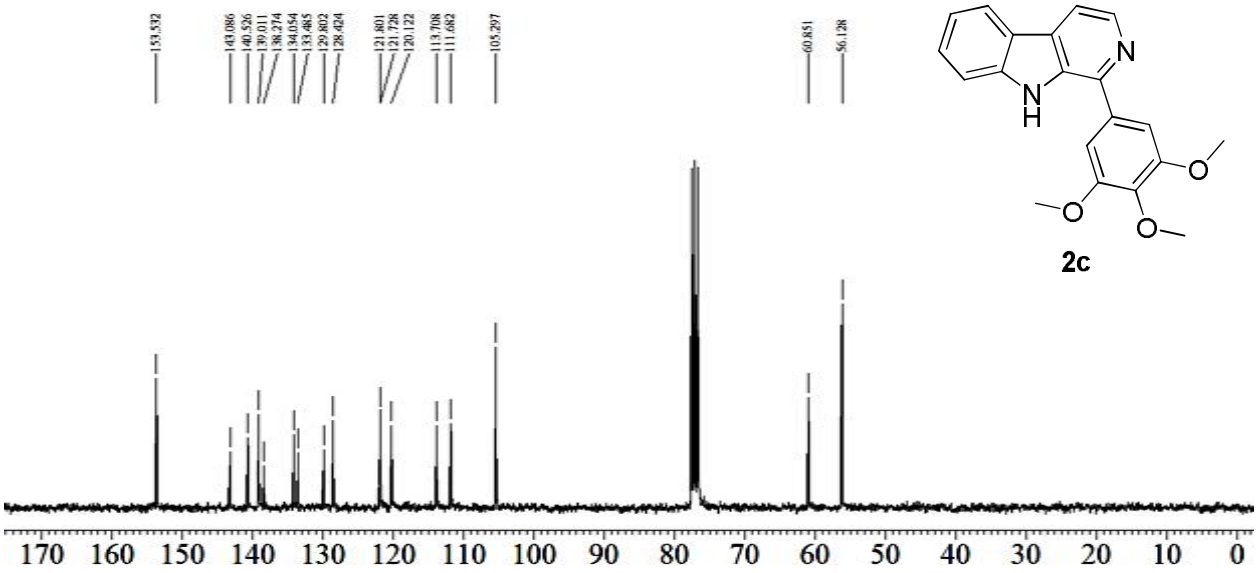


1-(3,4,5-Trimethoxyphenyl)-9*H*-pyrido[3,4-*b*]indole (2c)

^{1}H NMR (500 MHz, $CDCl_3$)

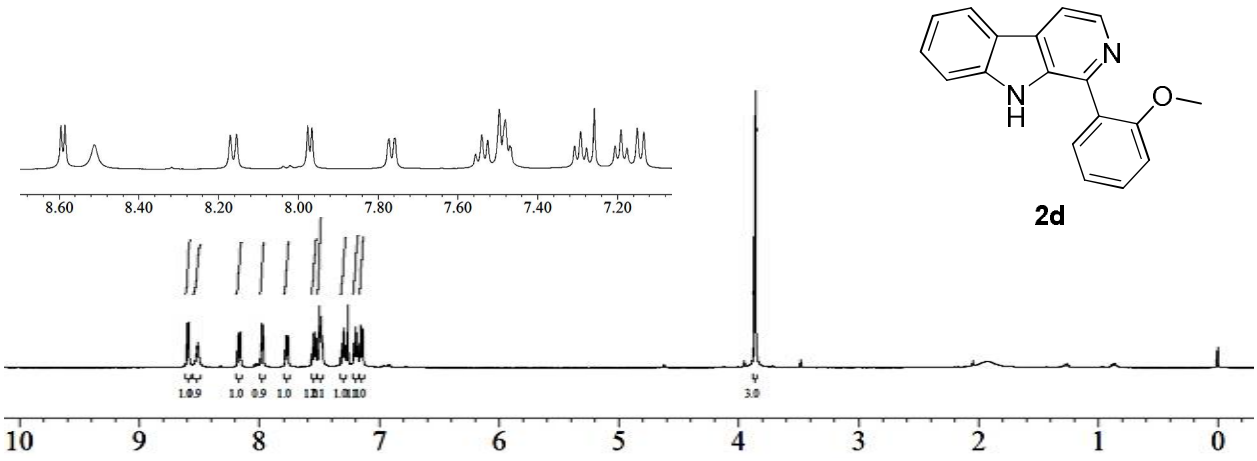


^{13}C NMR (75 MHz, $CDCl_3$)

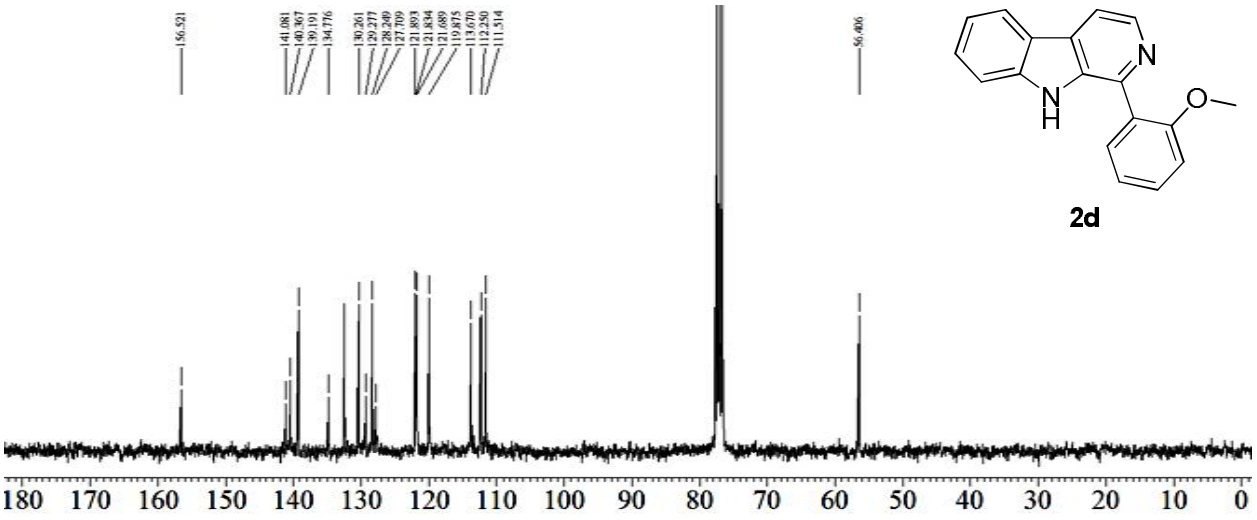


1-(2-Methoxyphenyl)-9*H*-pyrido[3,4-*b*]indole (2d)

^{1}H NMR (500 MHz, $CDCl_3$)

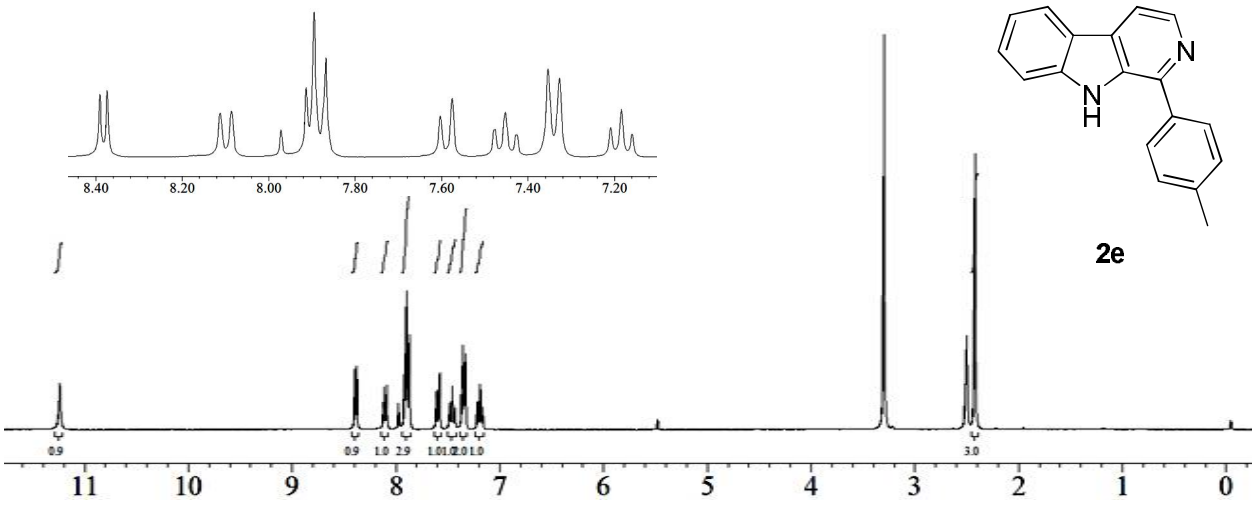


^{13}C NMR (75 MHz, $CDCl_3$)

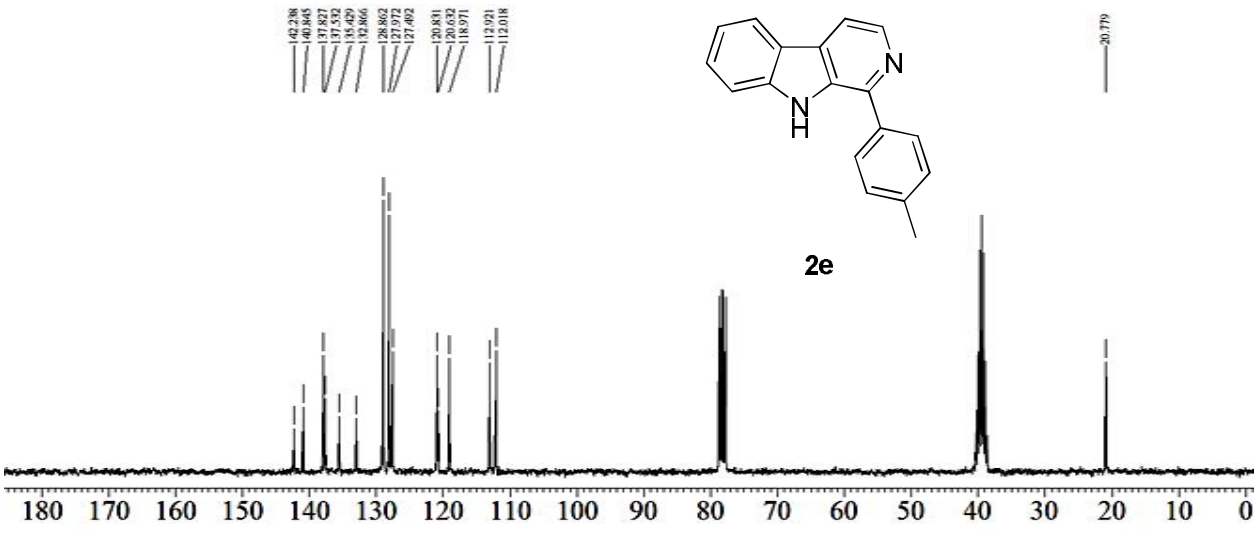


1-(*p*-Tolyl)-9*H*-pyrido[3,4-*b*]indole (2e)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

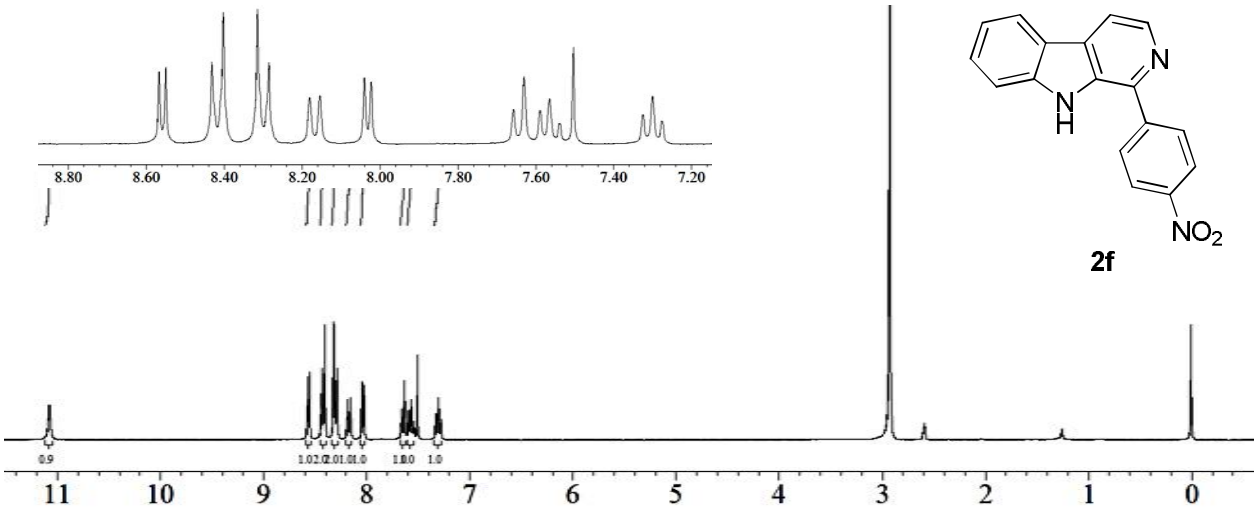


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

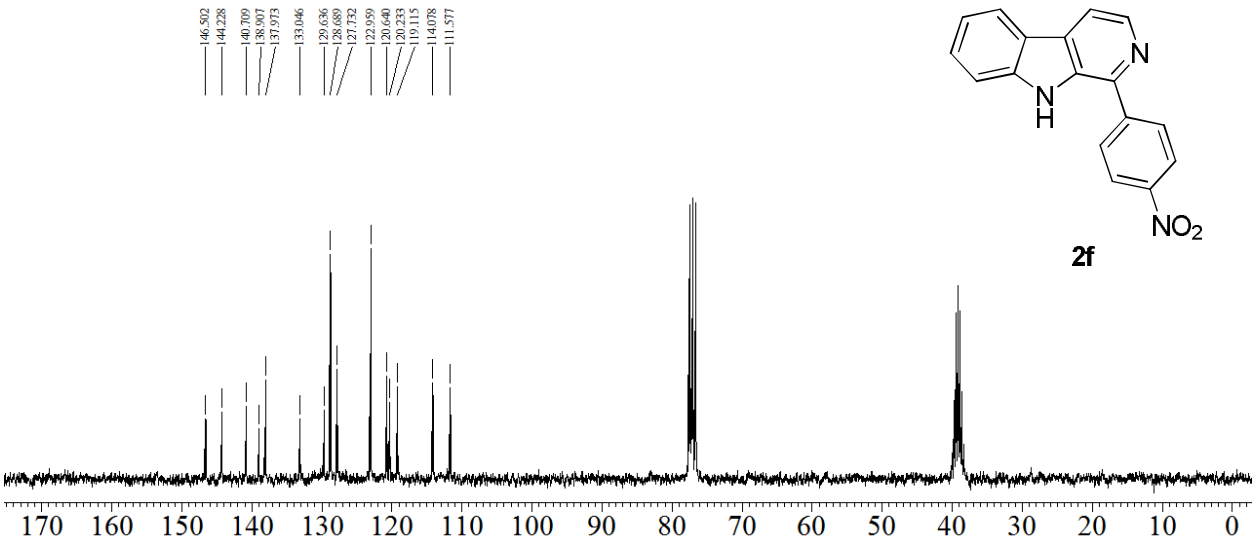


1-(4-Nitrophenyl)-9*H*-pyrido[3,4-*b*]indole (2f)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

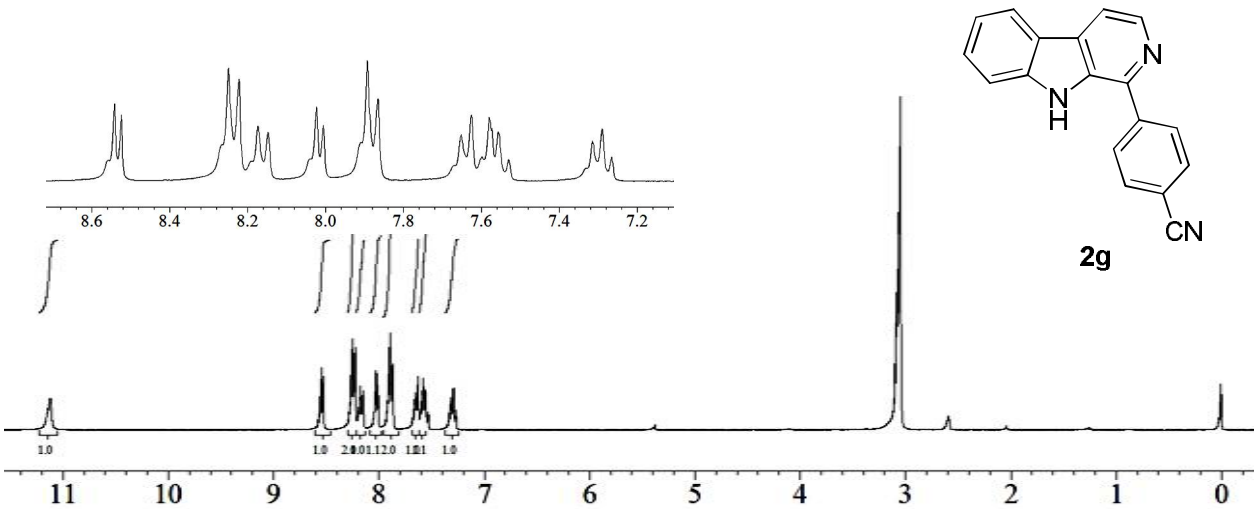


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO–d_6)

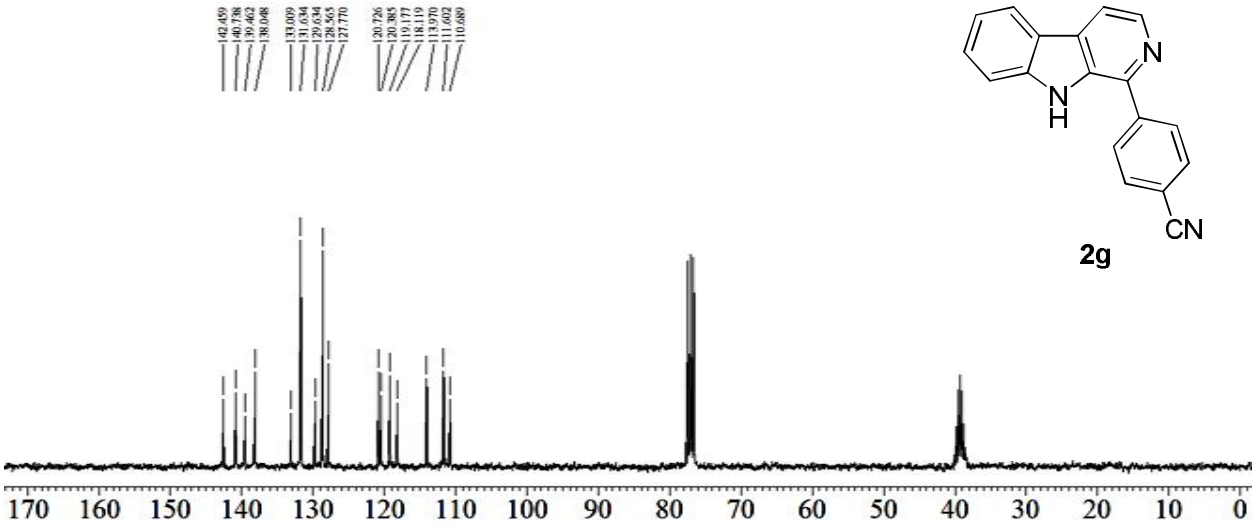


4-(9*H*-Pyrido[3,4-*b*]indol-1-yl)benzonitrile (2g)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

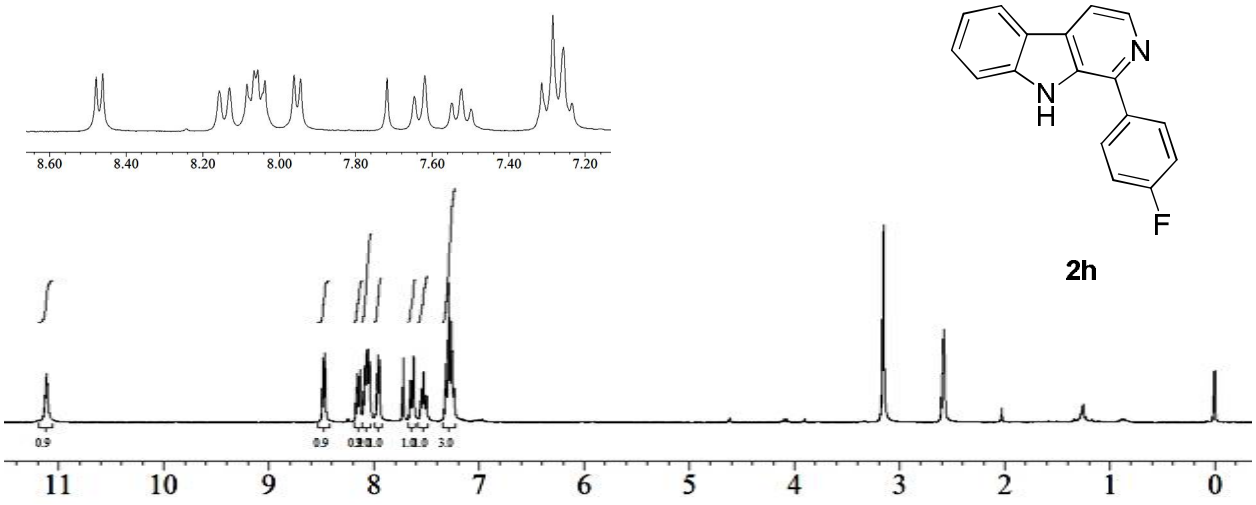


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO–d_6)

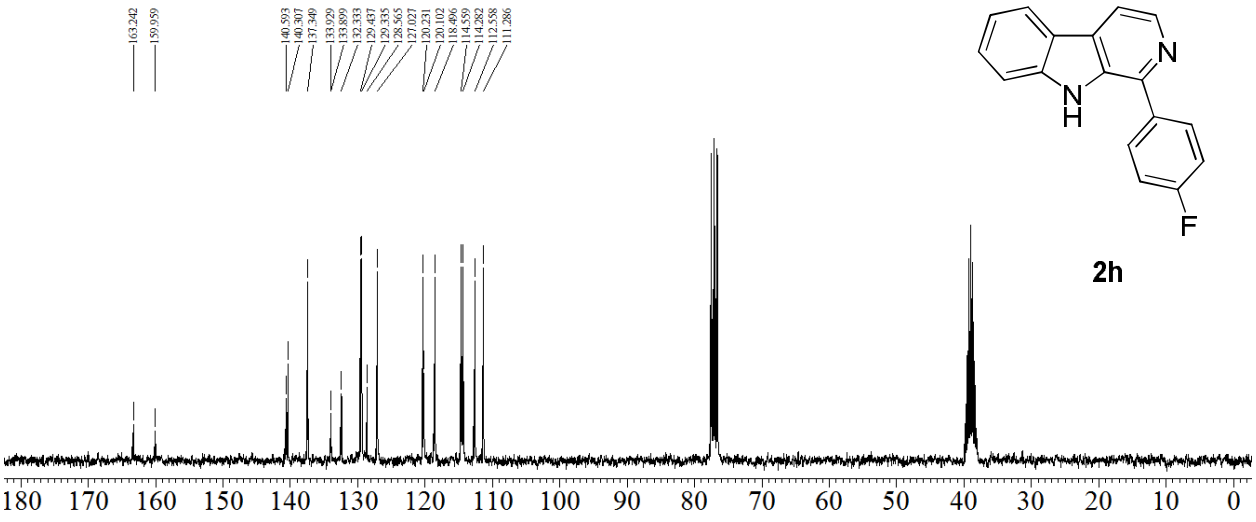


1-(4-Fluorophenyl)-9*H*-pyrido[3,4-*b*]indole (2h)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

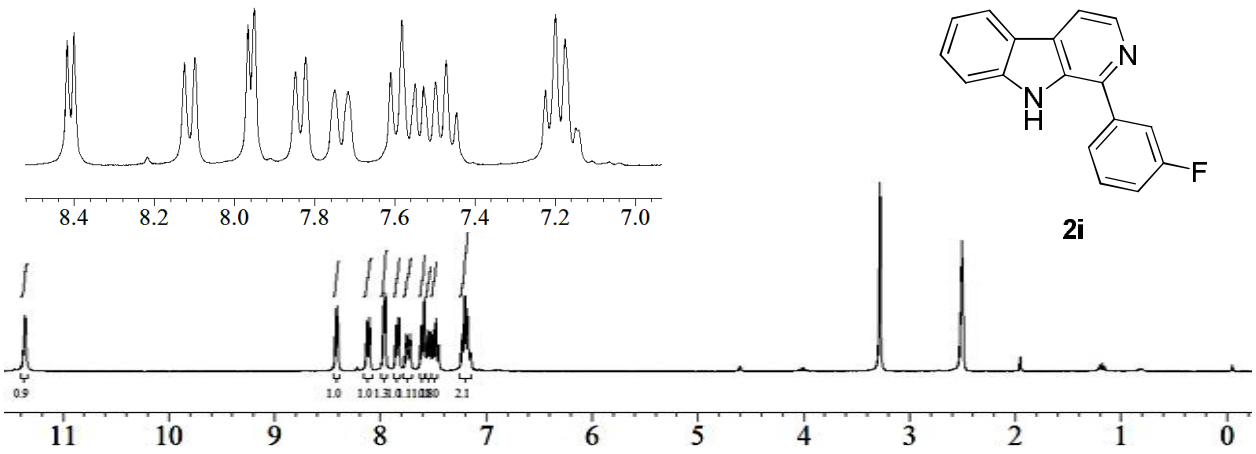


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

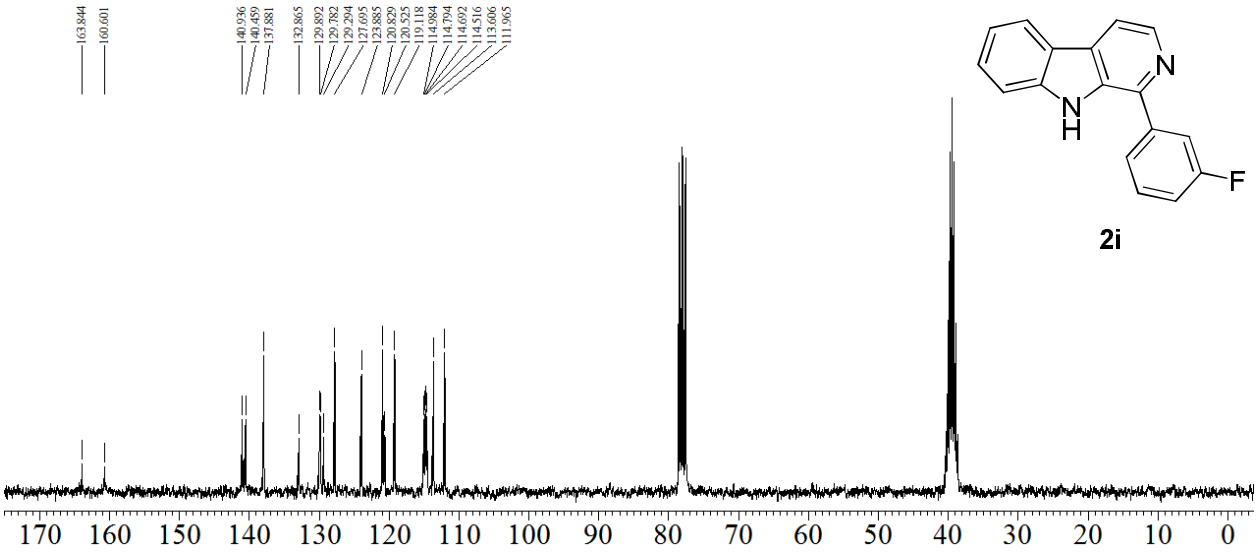


1-(3-Fluorophenyl)-9*H*-pyrido[3,4-*b*]indole (2i)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

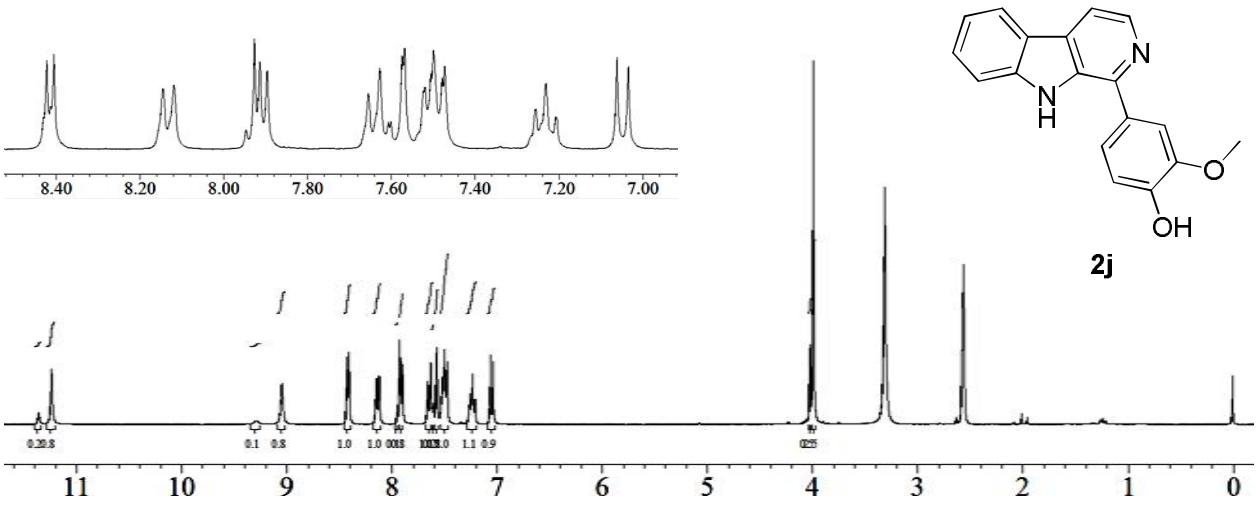


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

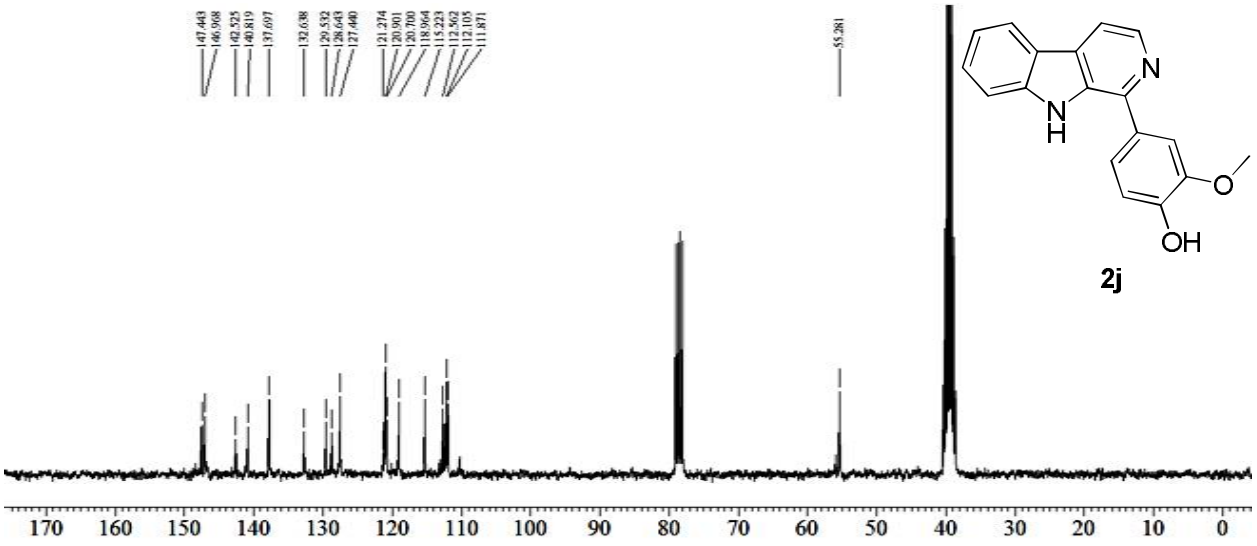


2-Methoxy-4-(9*H*-pyrido[3,4-*b*]indol-1-yl)phenol (2j)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

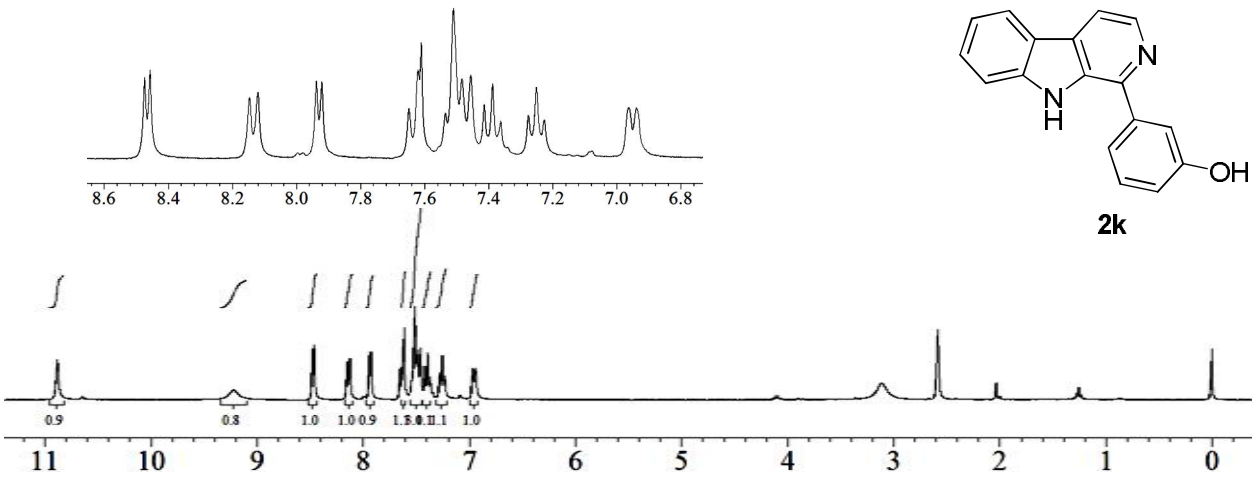


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

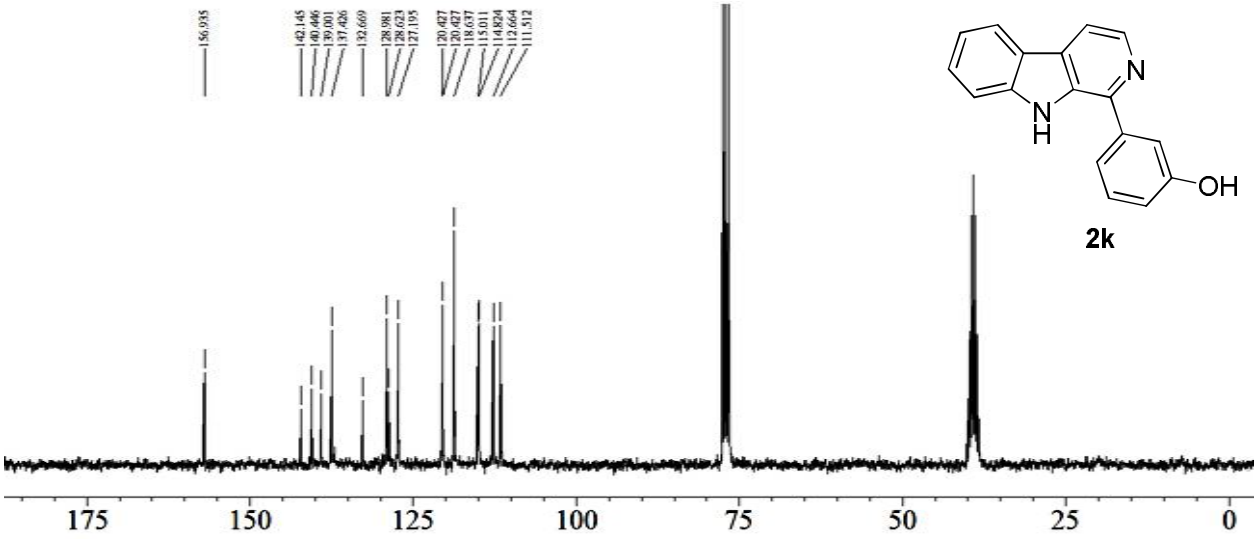


3-(9*H*-Pyrido[3,4-*b*]indol-1-yl)phenol (2k)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

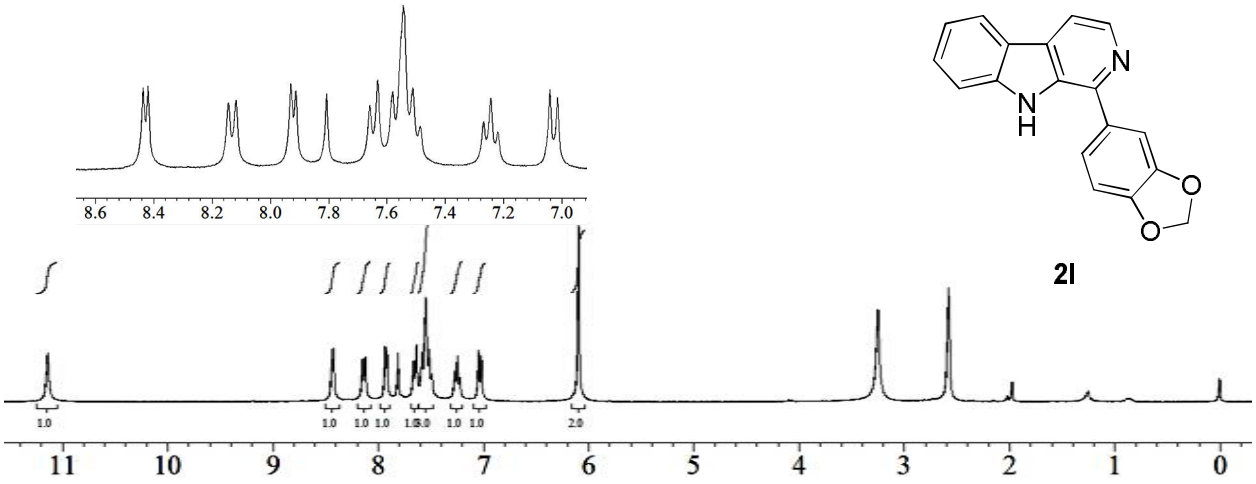


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

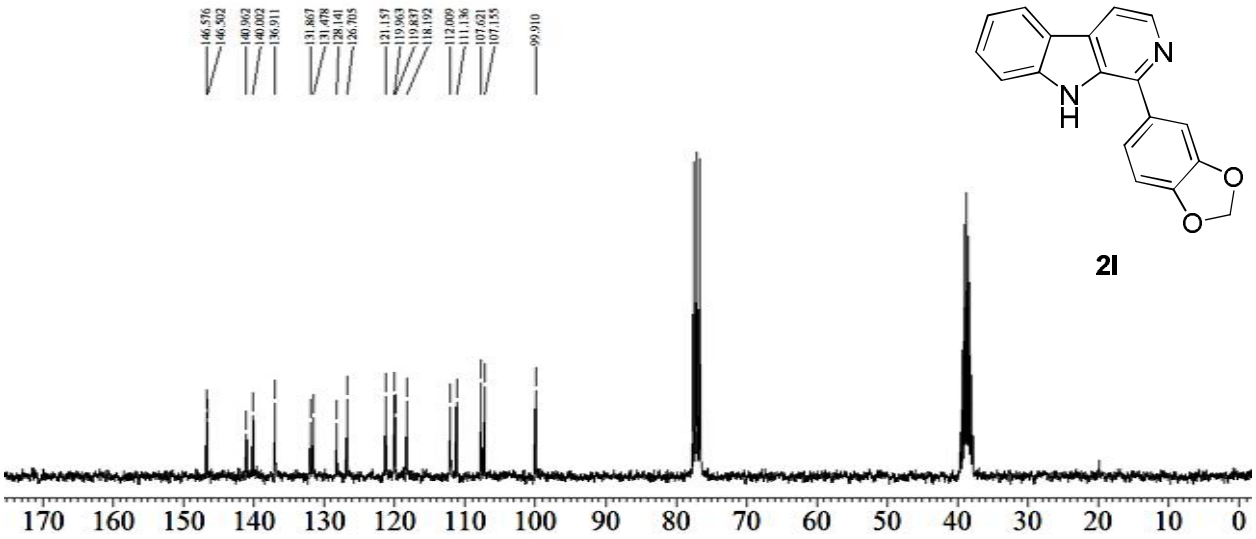


1-(Benzo[*d*][1,3]dioxol-5-yl)-9*H*-pyrido[3,4-*b*]indole (2l)

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO−d_6)

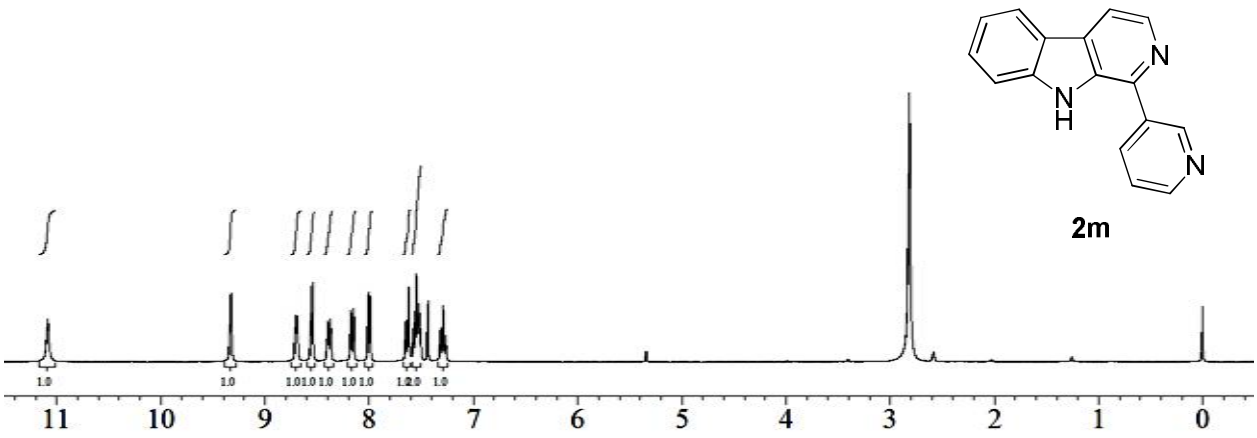


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

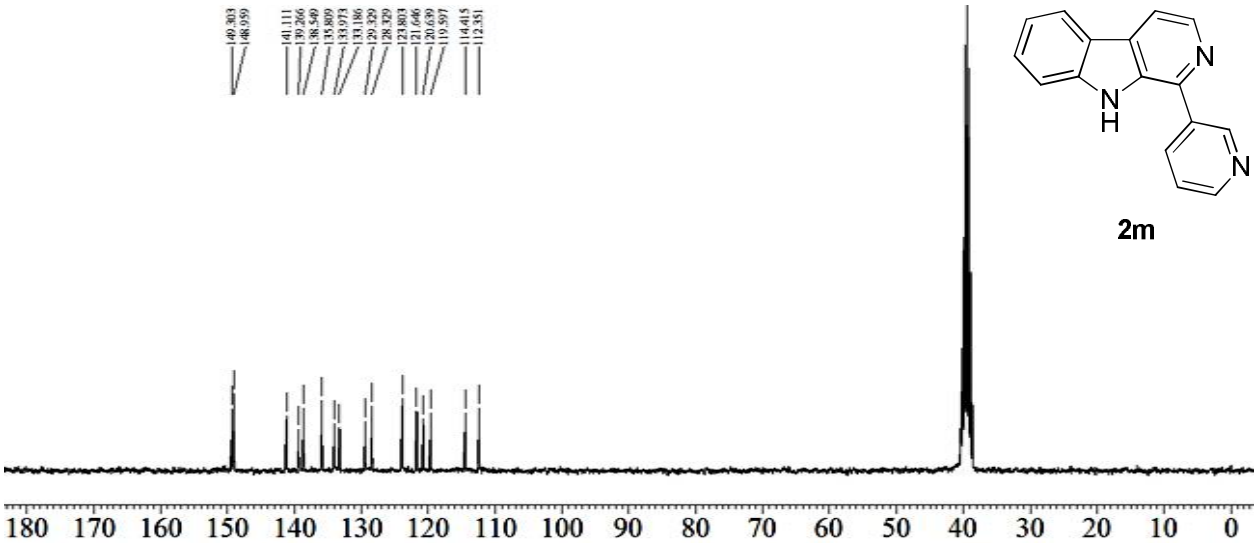


1-(Pyridin-3-yl)-9*H*-pyrido[3,4-*b*]indole (2m)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

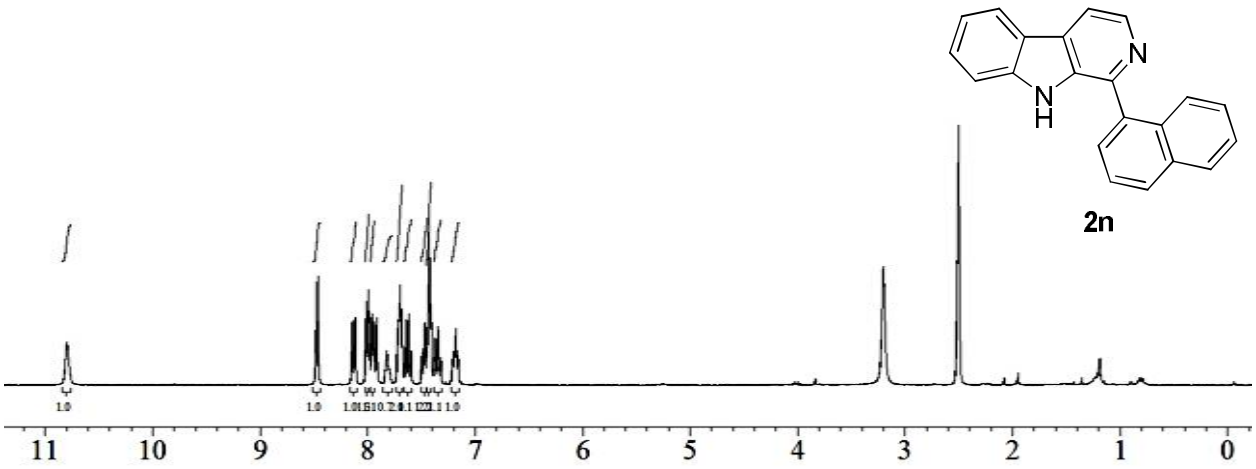


^{13}C NMR (75 MHz, DMSO−d_6)

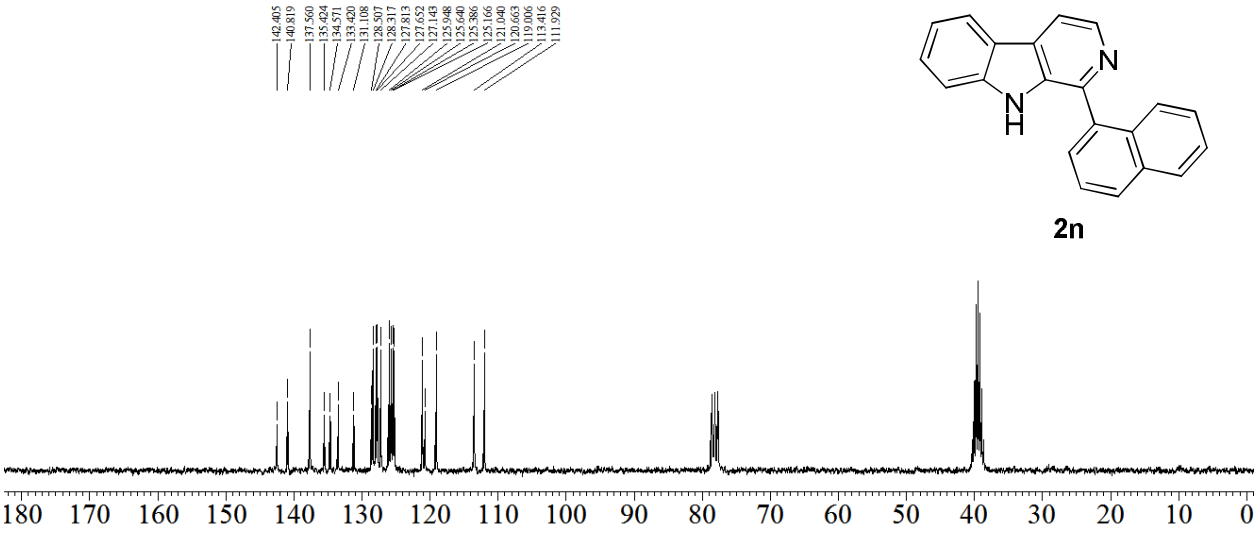


1-(Naphthalen-1-yl)-9*H*-pyrido[3,4-*b*]indole (2n)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

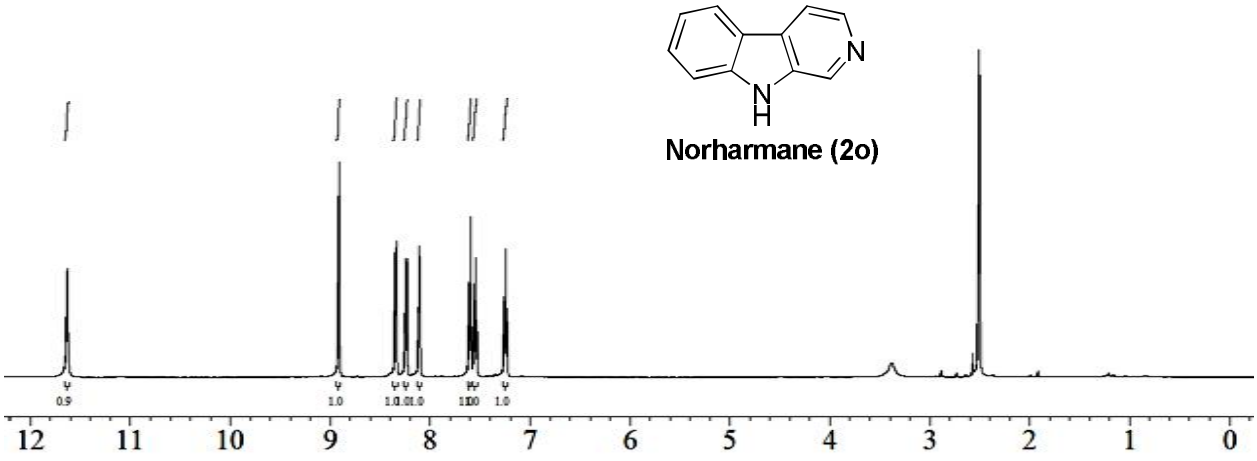


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

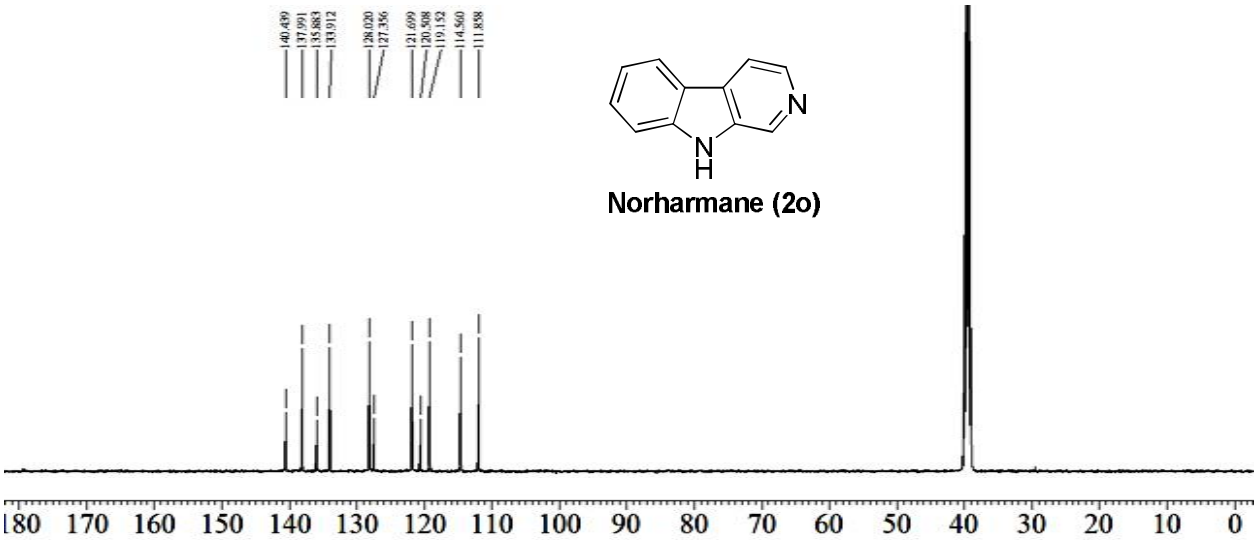


Norharmane (2o)

^{1}H NMR (500 MHz, DMSO−d_6)

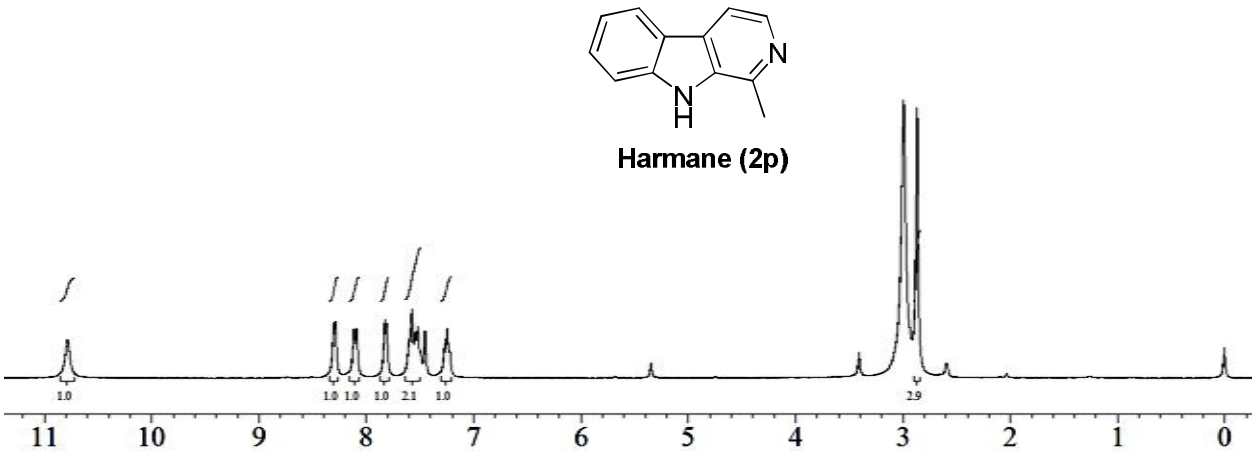


^{13}C NMR (125 MHz, DMSO−d_6)

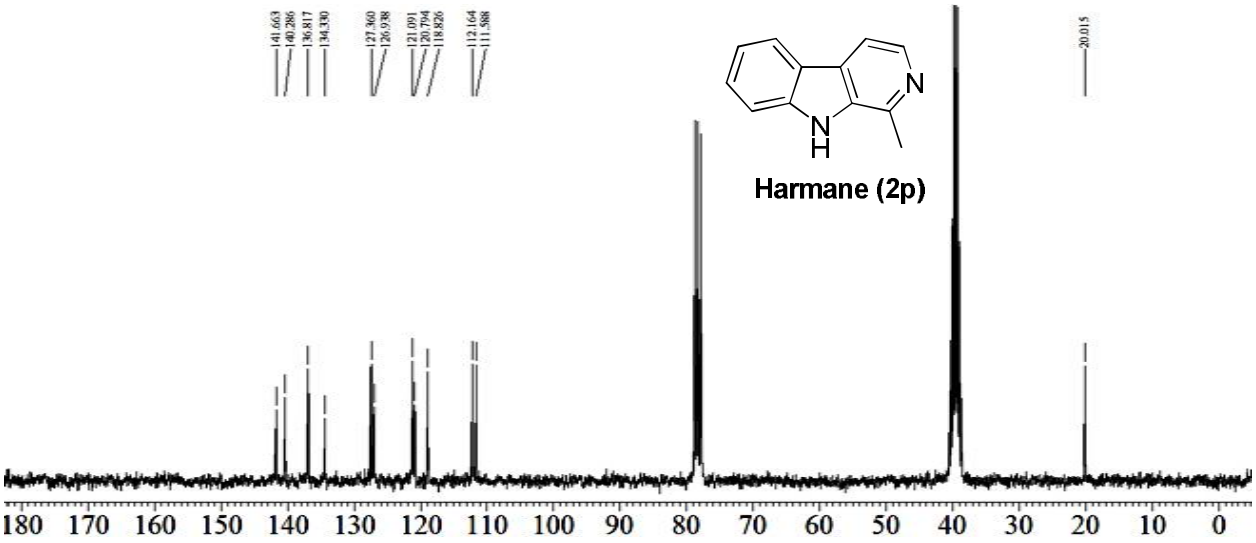


Harmane (2p):

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

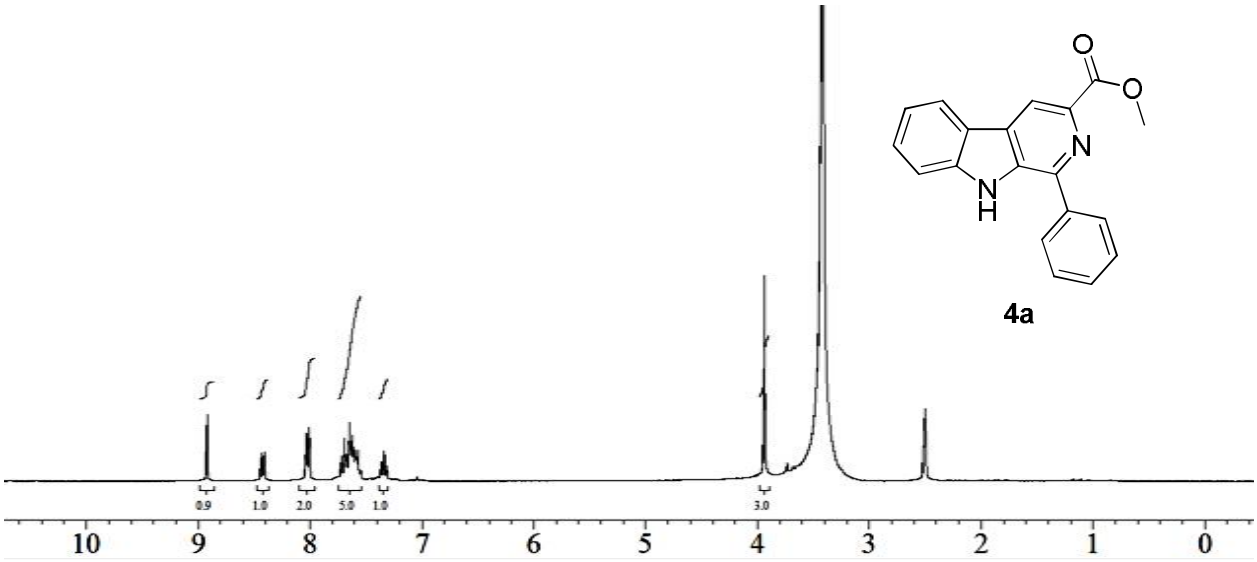


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO–d_6)

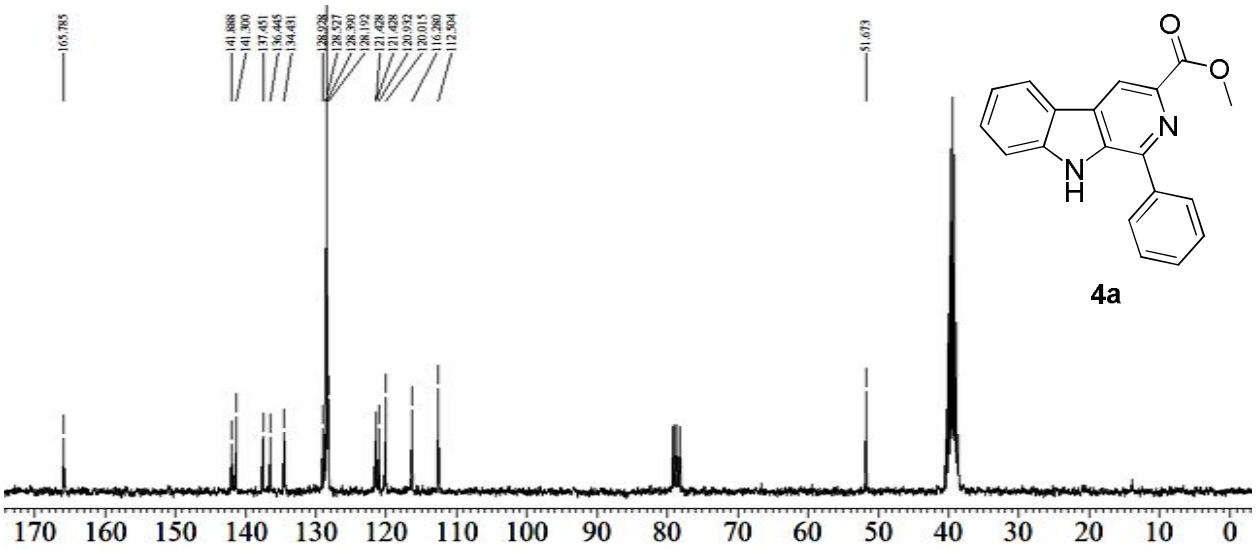


Methyl 1-phenyl-9*H*-pyrido [3, 4-*b*] indole-3-carboxylate (4a)

^{1}HNMR (300 MHz, DMSO−d_6)

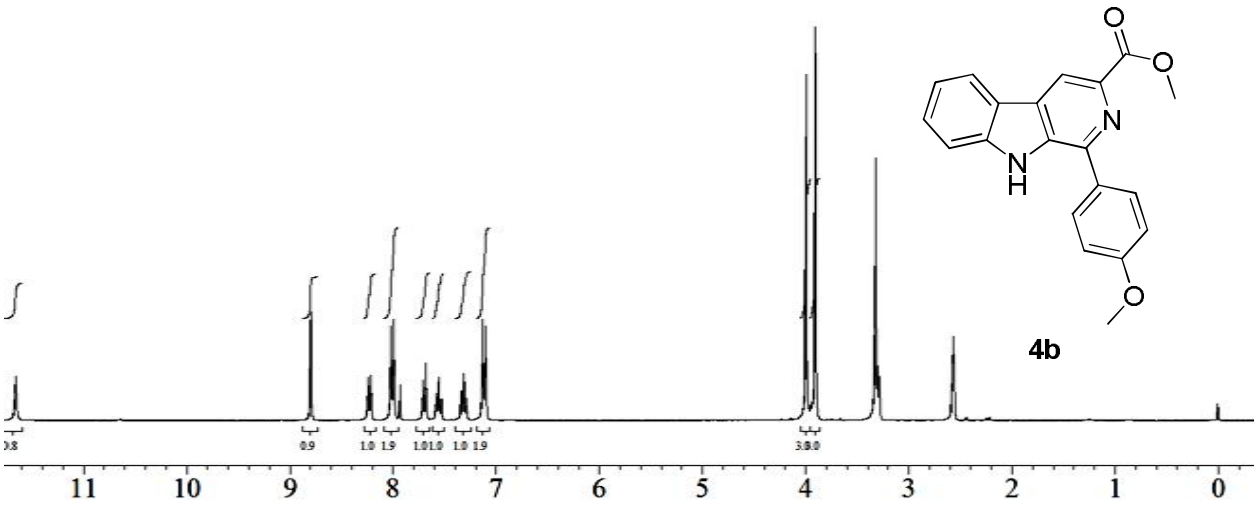


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

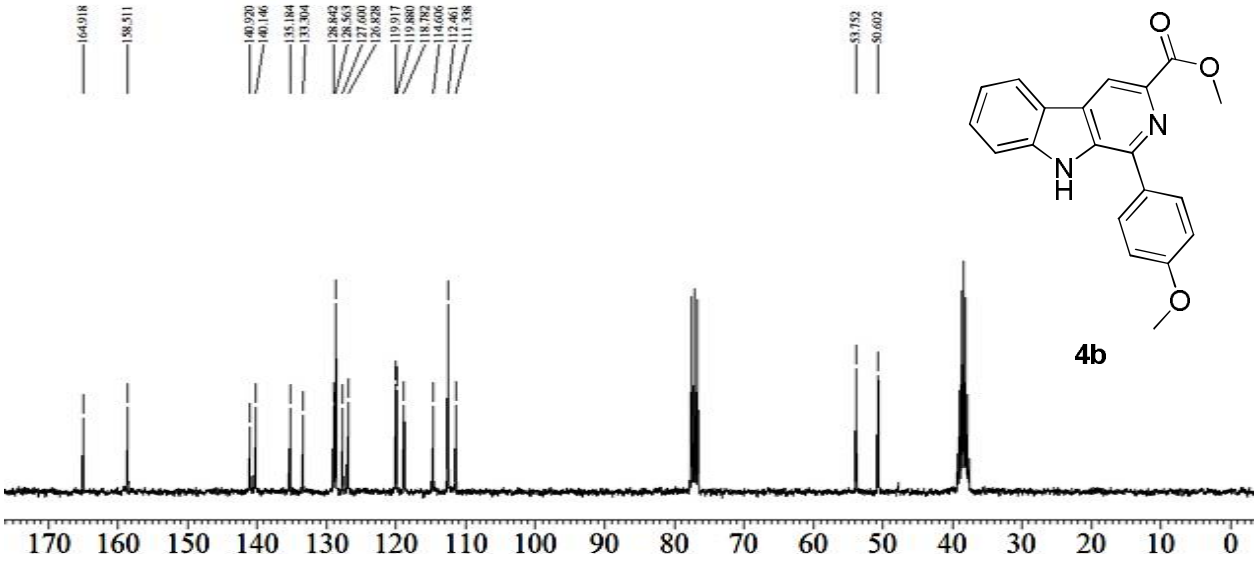


Methyl 1-(4-methoxyphenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4b)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

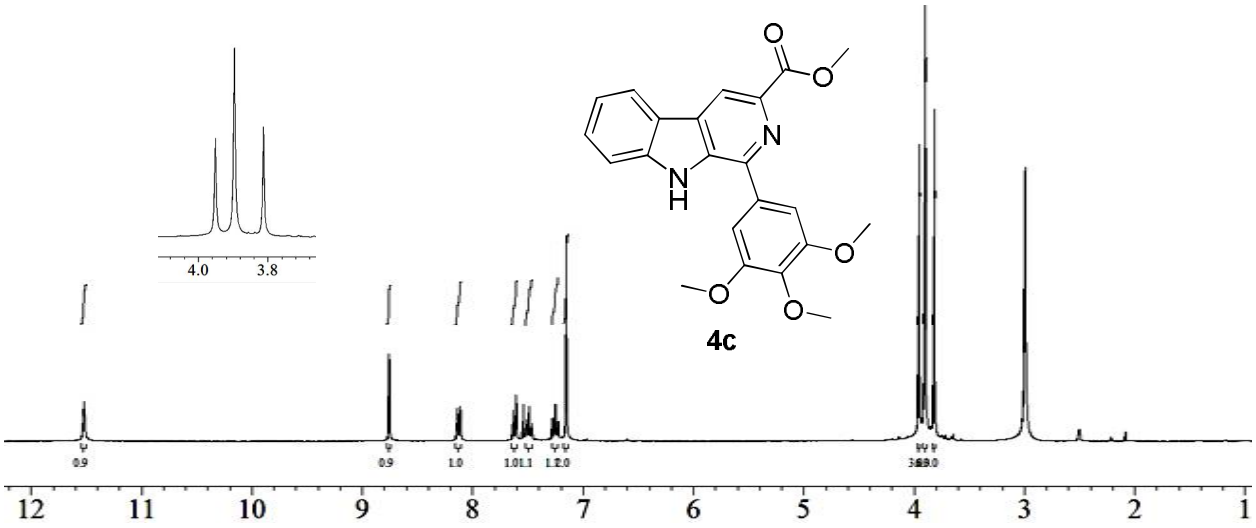


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

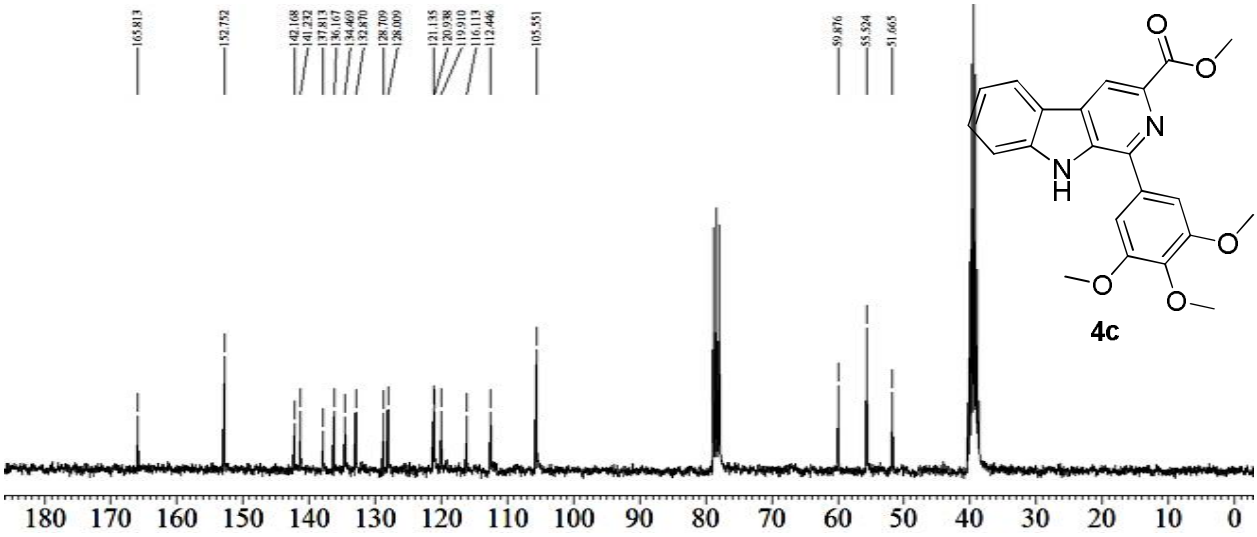


Methyl 1-(3,4,5-trimethoxyphenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4c)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

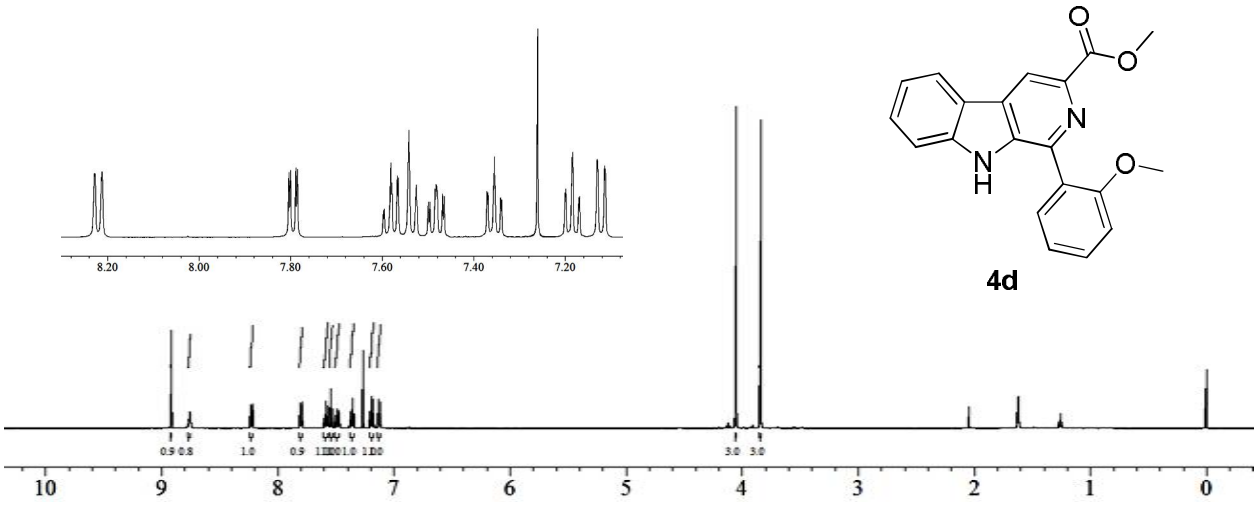


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

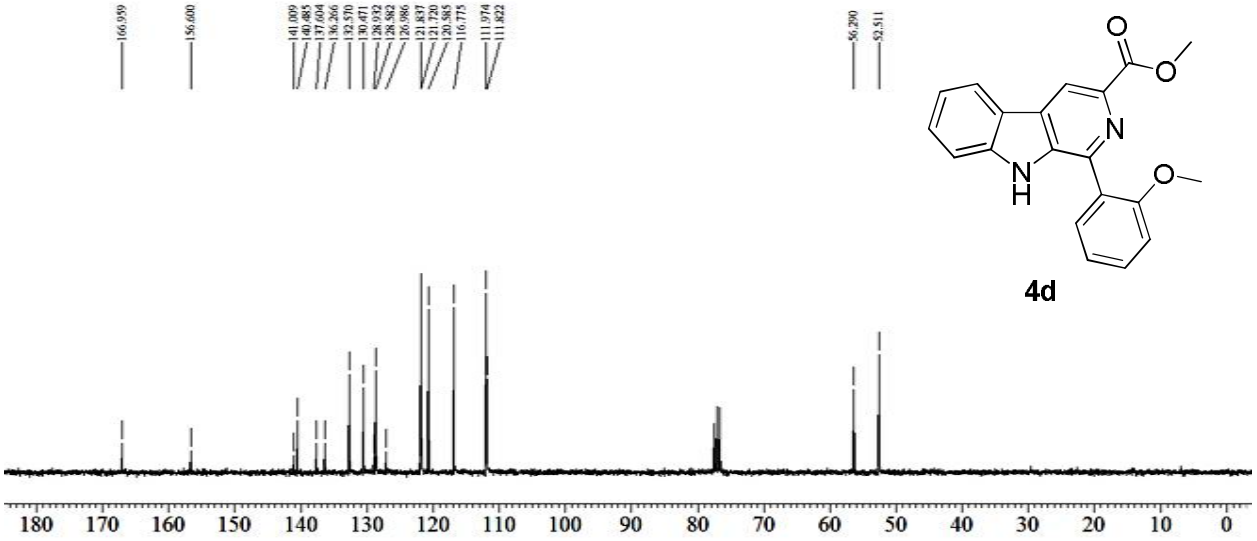


Methyl 1-(2-methoxyphenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4d)

^{1}H NMR (500 MHz, $CDCl_3$)

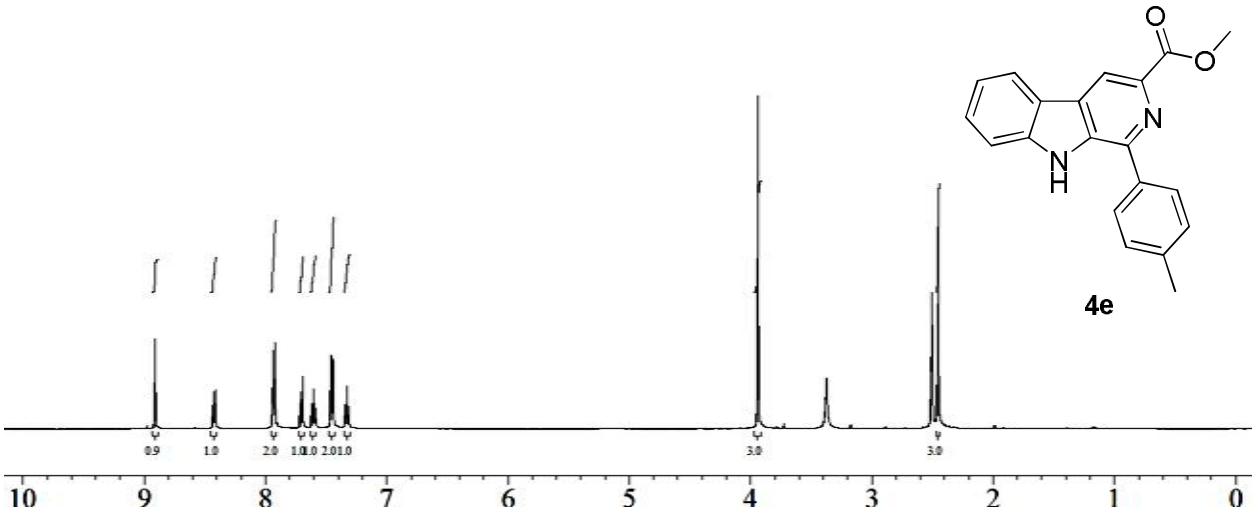


^{13}C NMR (75 MHz, $CDCl_3$)

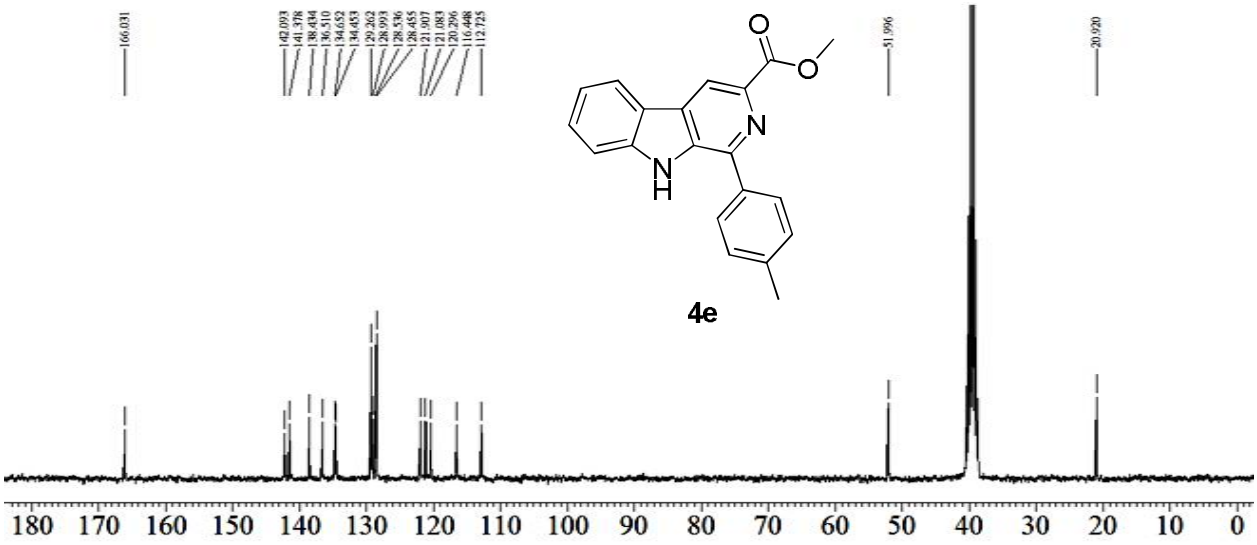


Methyl 1-(p-tolyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4e)

^{1}H NMR (500 MHz, DMSO−d_6)

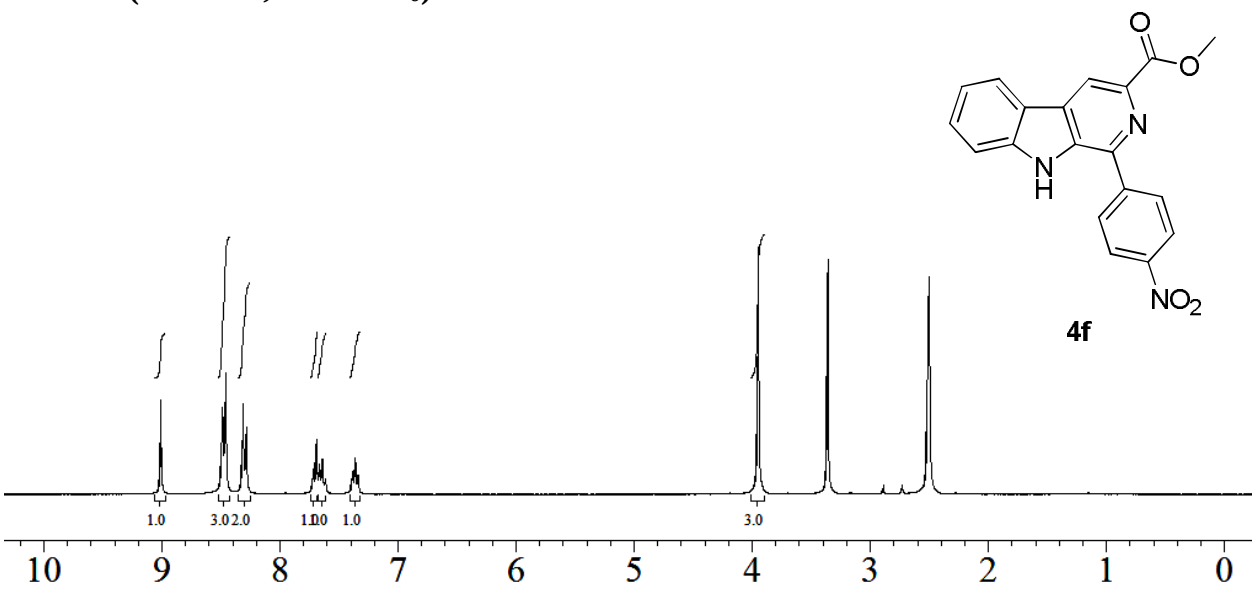


^{13}C NMR (500 MHz, DMSO−d_6)

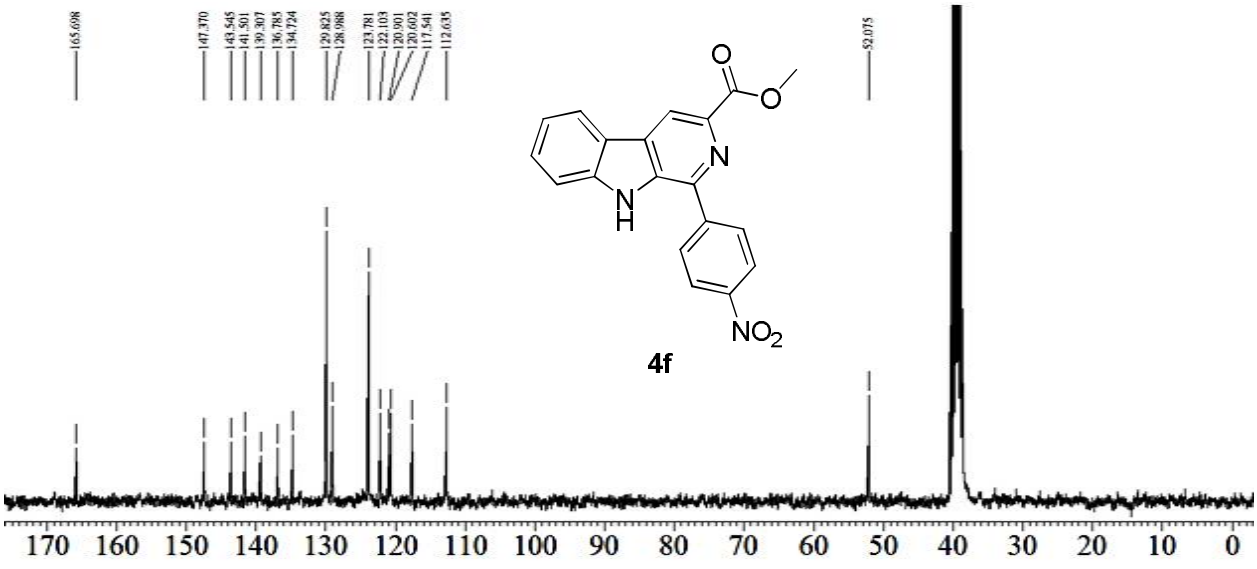


Methyl 1-(4-nitrophenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4f)

^{1}H NMR (300 MHz, DMSO−d_6)

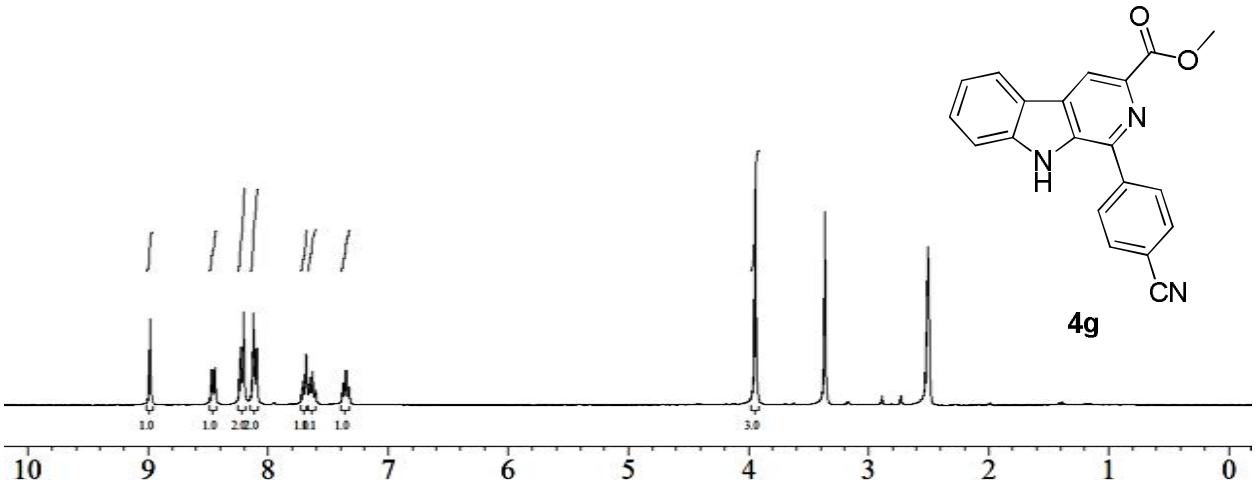


^{13}C NMR (75 MHz, DMSO−d_6)

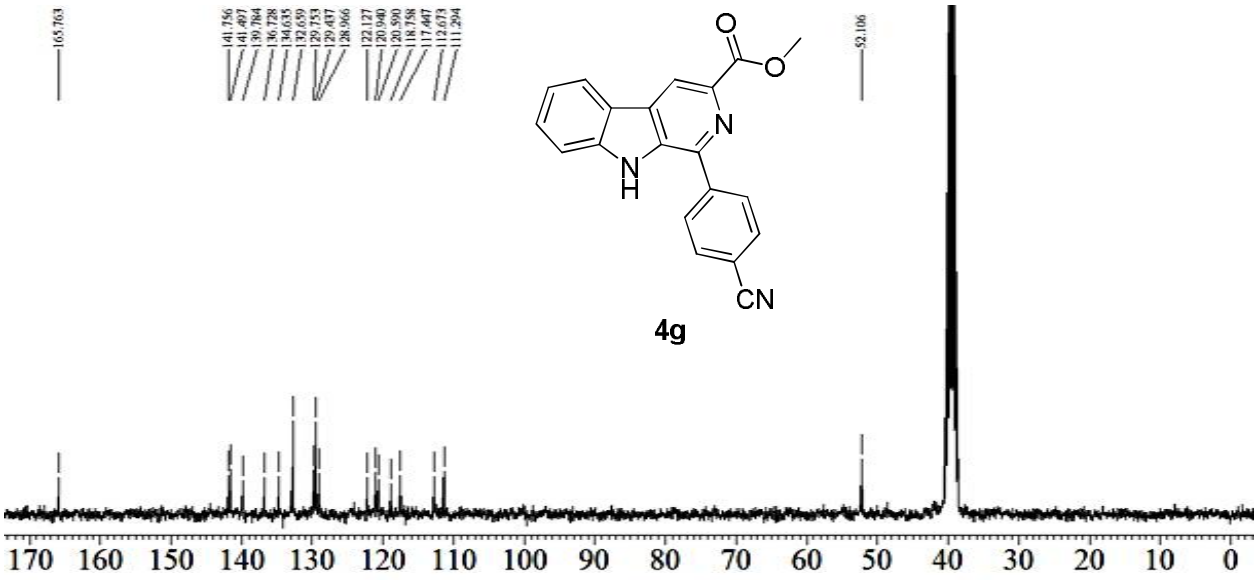


Methyl 1-(4-cyanophenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4g)

^{1}H NMR (300 MHz, DMSO−d_6)

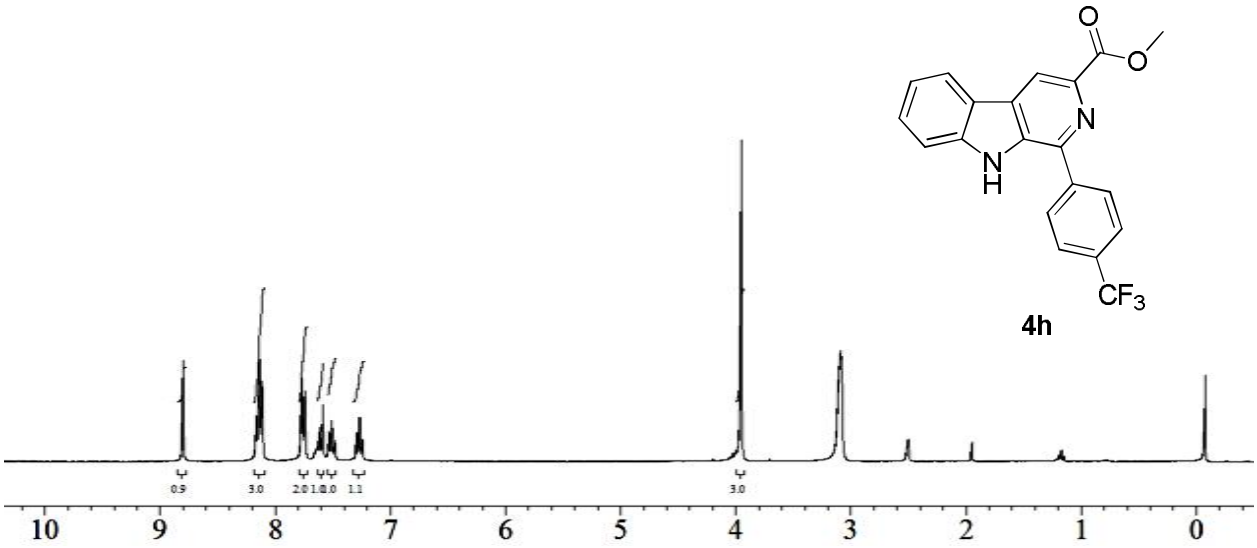


^{13}C NMR (75 MHz, DMSO−d_6)

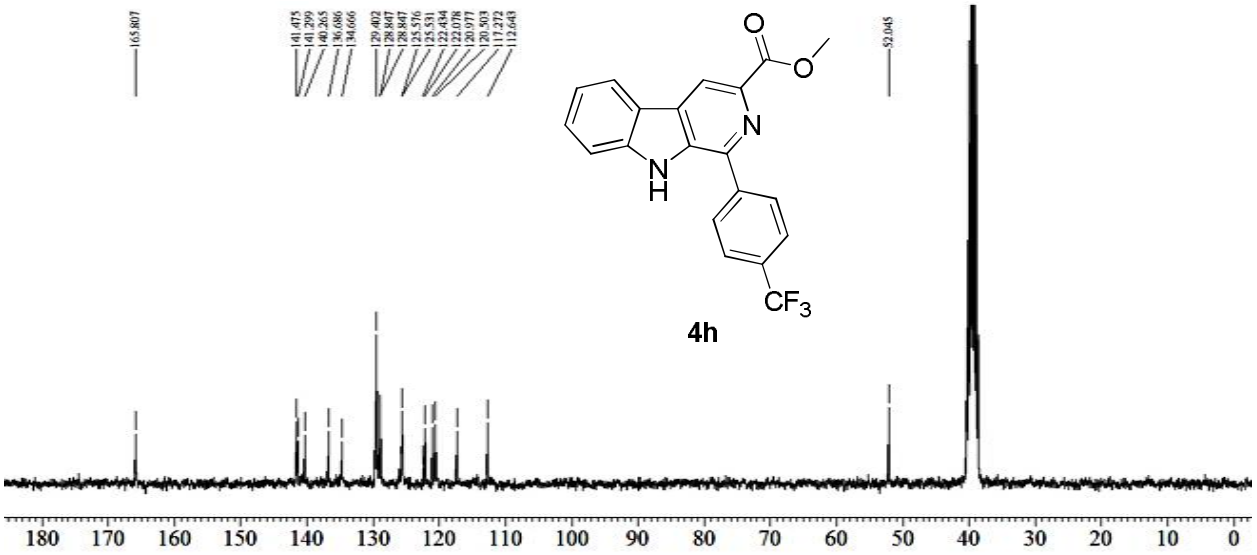


Methyl 1-(4-(trifluoromethyl)phenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4h)

^{1}H NMR (300 MHz, DMSO−d_6)

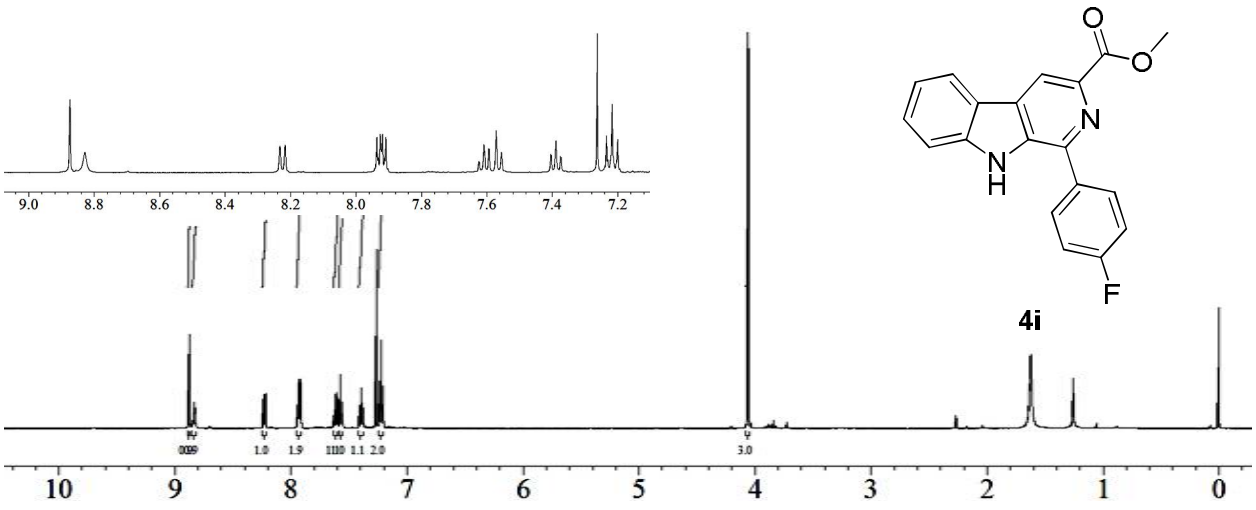


^{13}C NMR (75 MHz, DMSO−d_6)

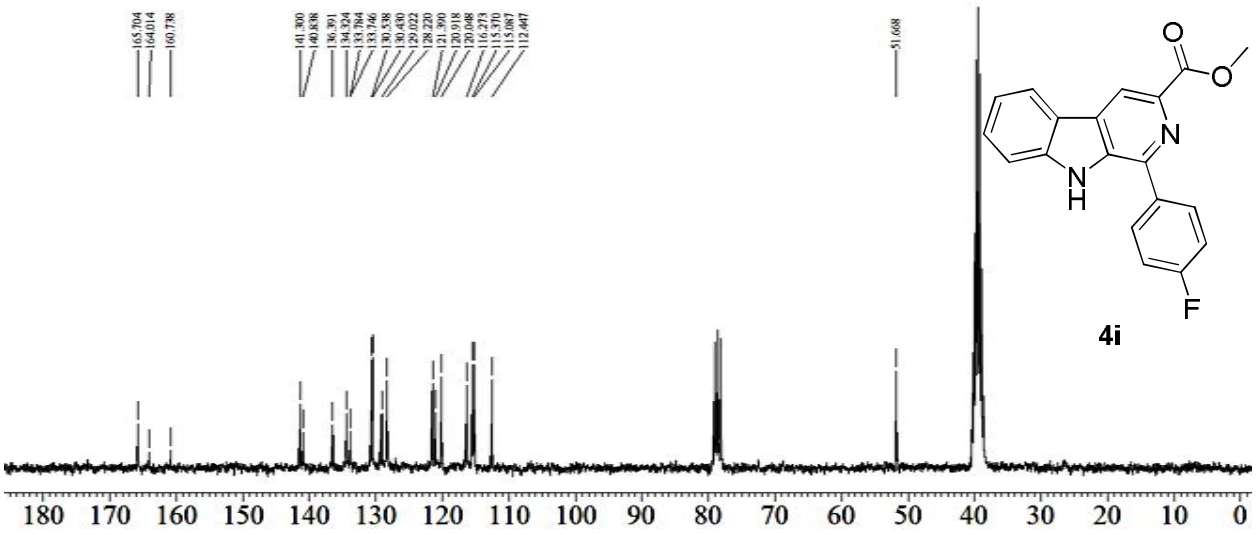


Methyl 1-(4-fluorophenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4i)

^{1}H NMR (500 MHz, $CDCl_3$ + DMSO−d_6)

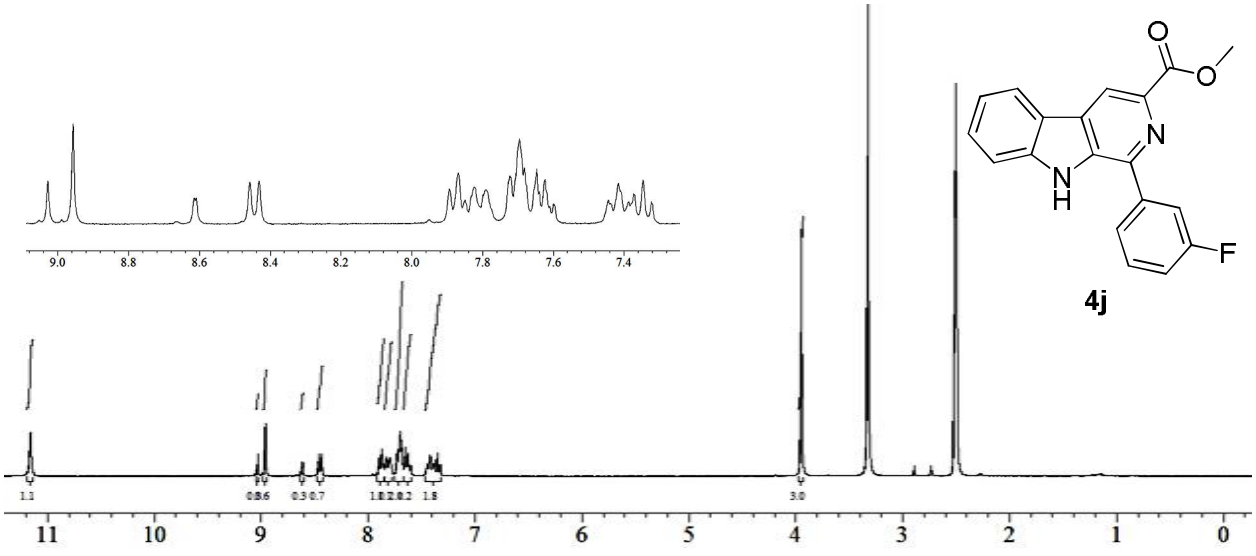


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

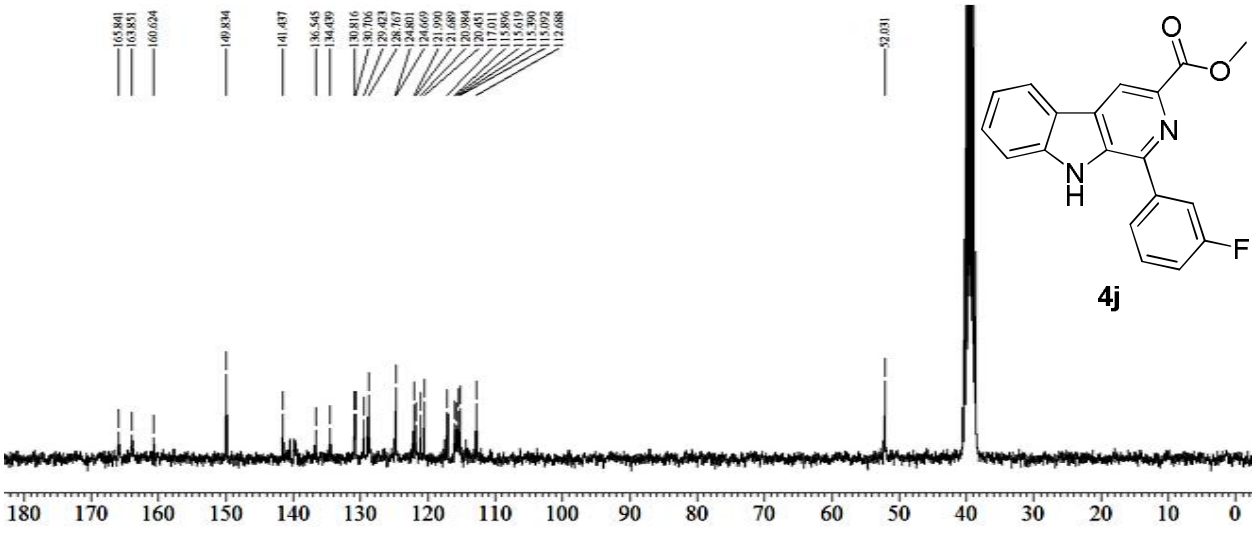


Methyl 1-(3-fluorophenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4j)

^{1}H NMR (300 MHz, DMSO−d_6)

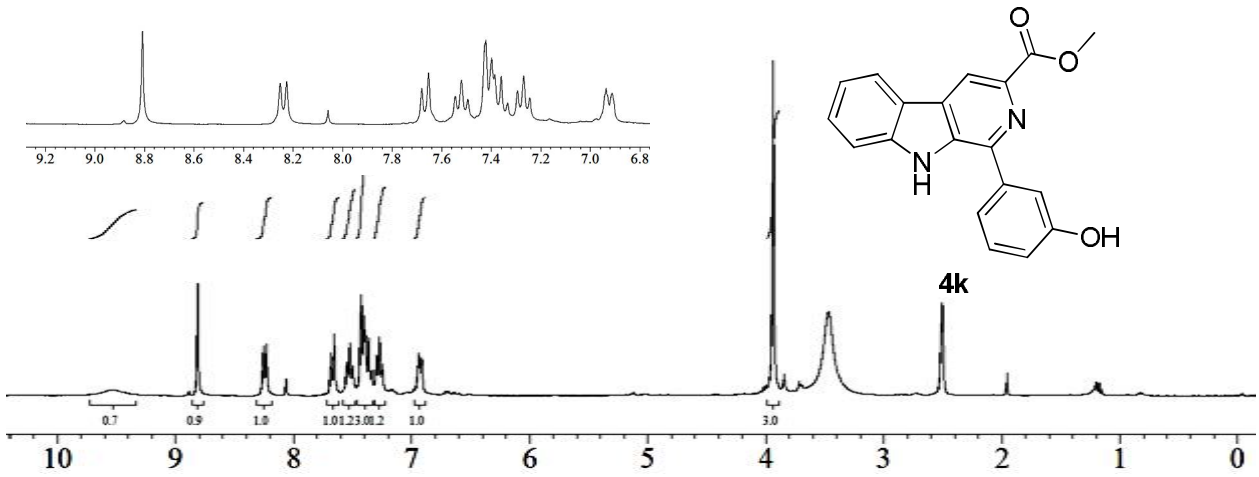


^{13}C NMR (75 MHz, DMSO–d_6)

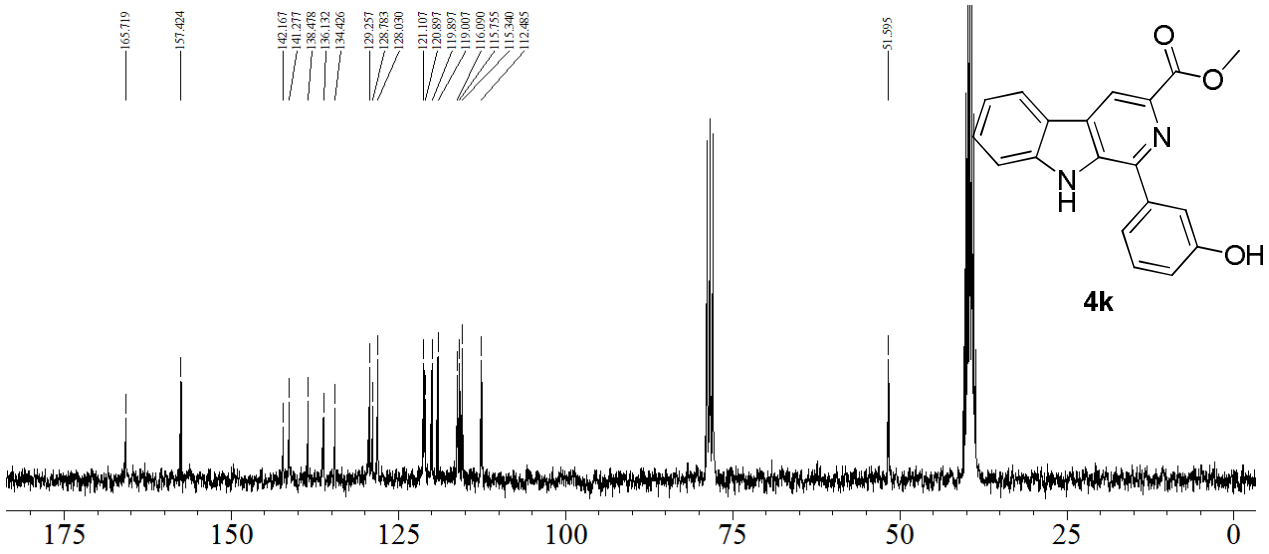


Methyl 1-(3-hydroxyphenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4k)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

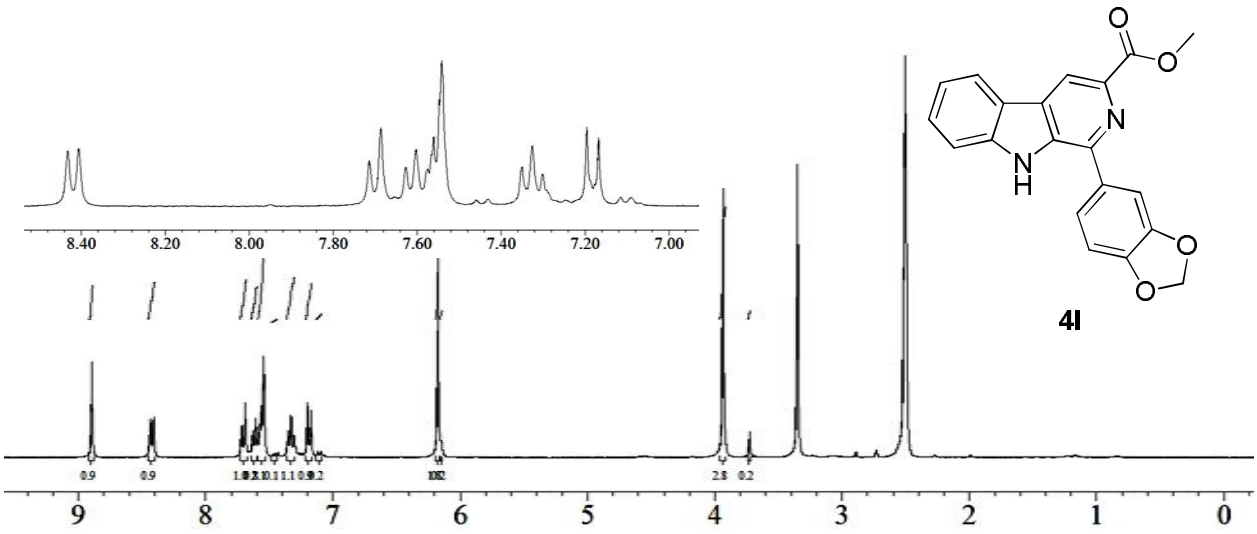


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

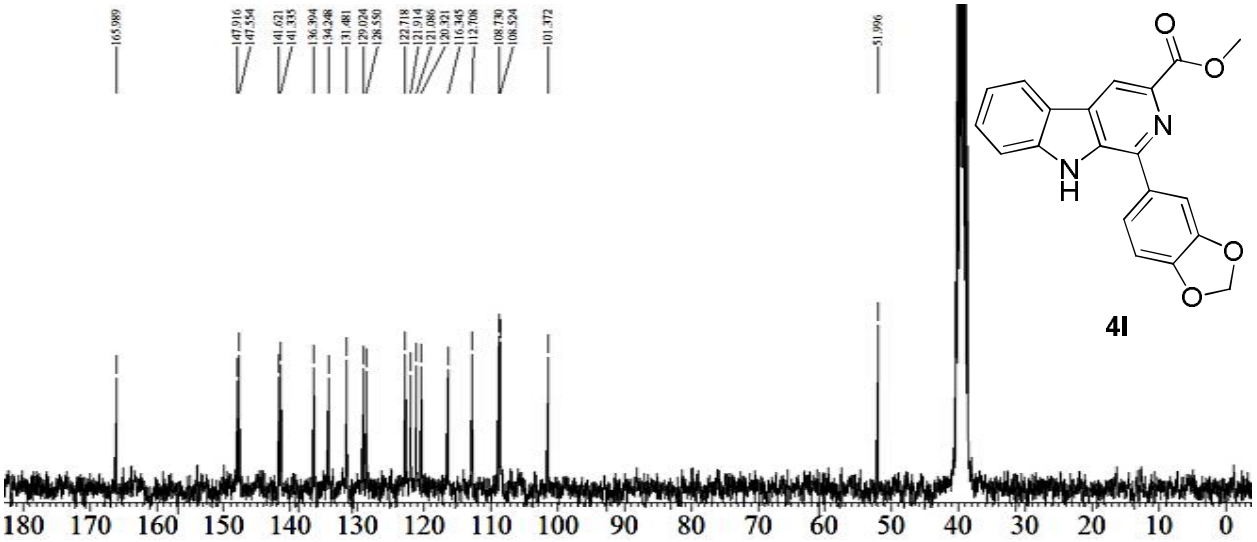


Methyl 1-(benzo[*d*][1,3]dioxol-5-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4l)

^{1}H NMR (300 MHz, DMSO−d_6)

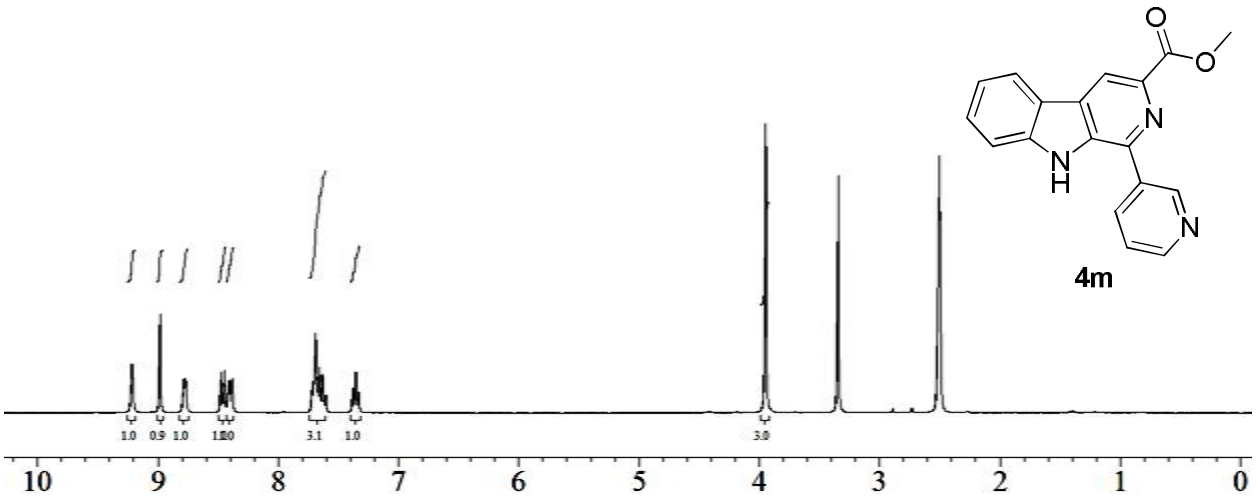


^{13}C NMR (75 MHz, DMSO−d_6)

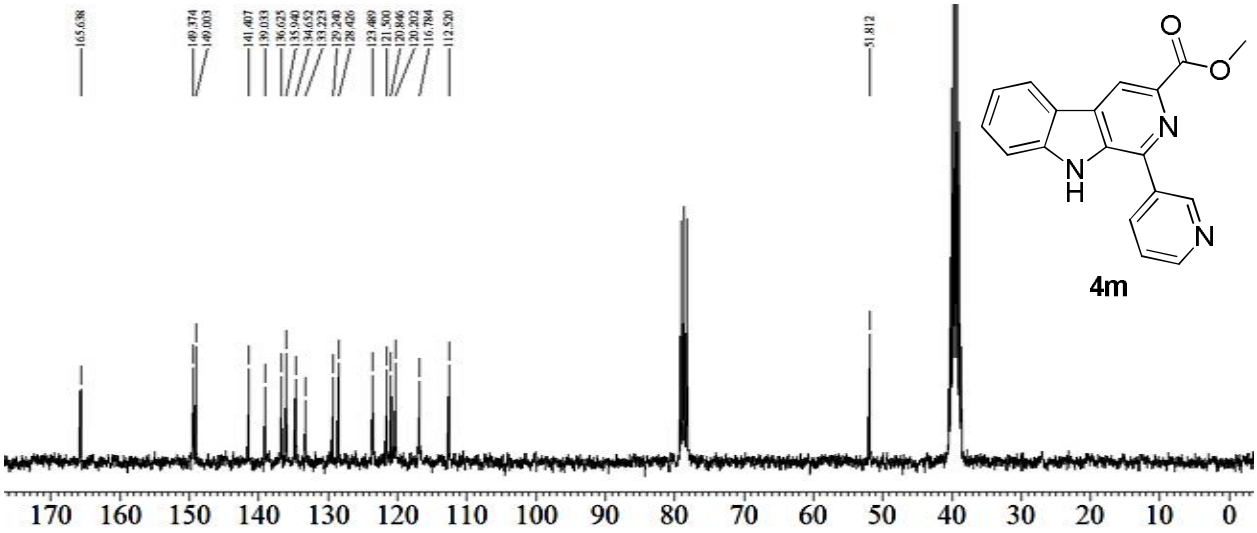


Methyl 1-(pyridin-3-yl)-9*H*-pyrido [3, 4-*b*] indole-3-carboxylate (4m)

^{1}H NMR (300 MHz, DMSO−d_6)

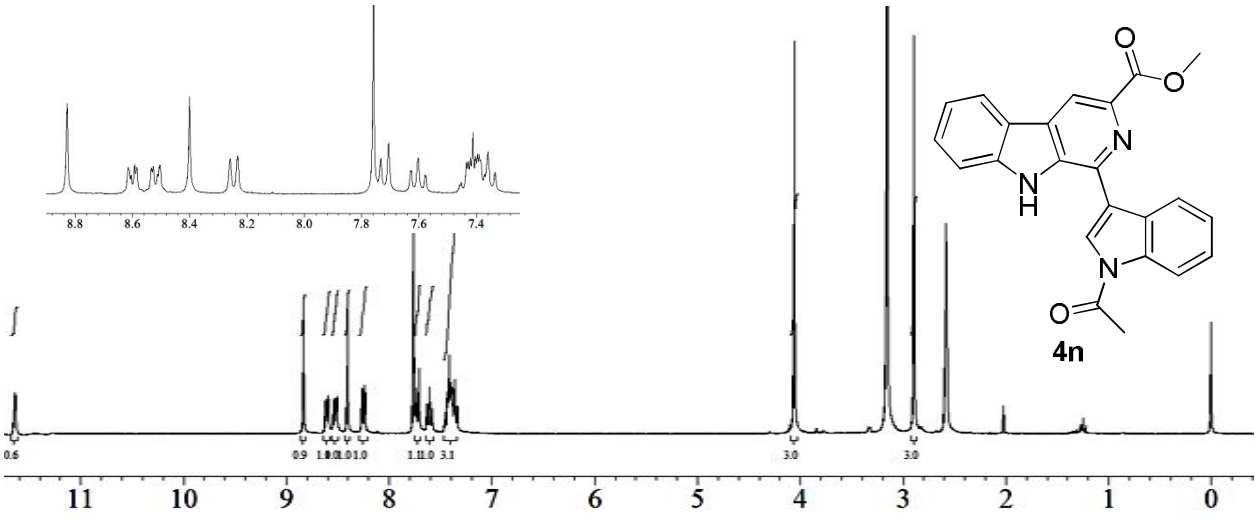


^{13}C NMR (75 MHz, $CDCl_3$ + DMSO−d_6)

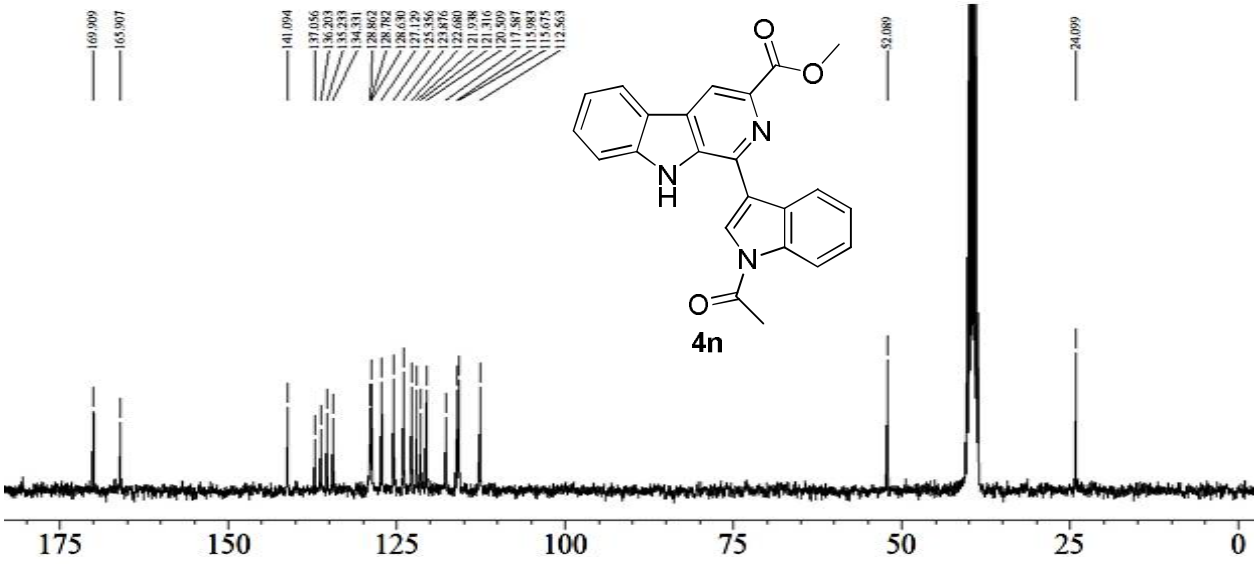


Methyl 1-(1-acetyl-1*H*-indol-3-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4n)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

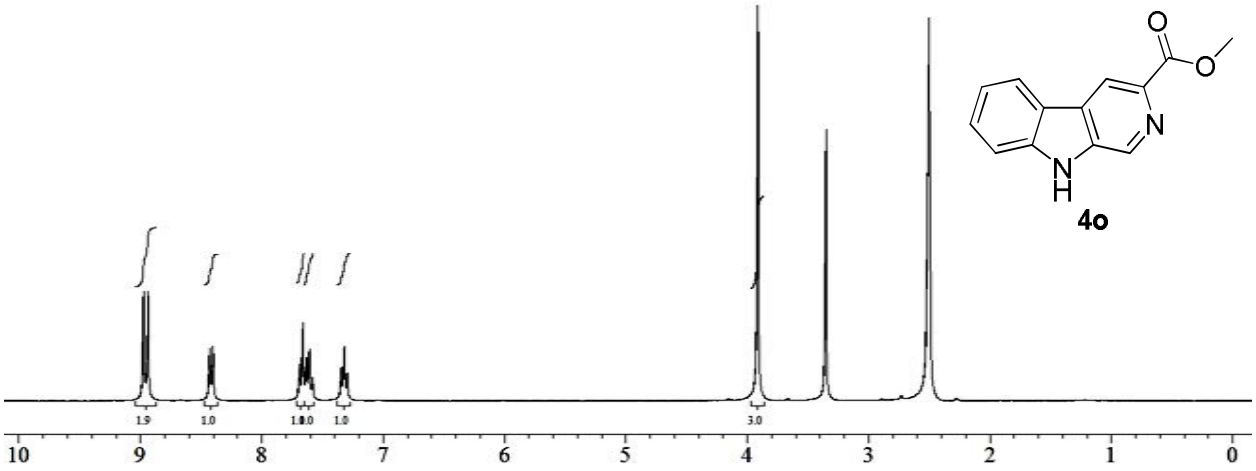


^{13}C NMR (75 MHz, DMSO−d_6)

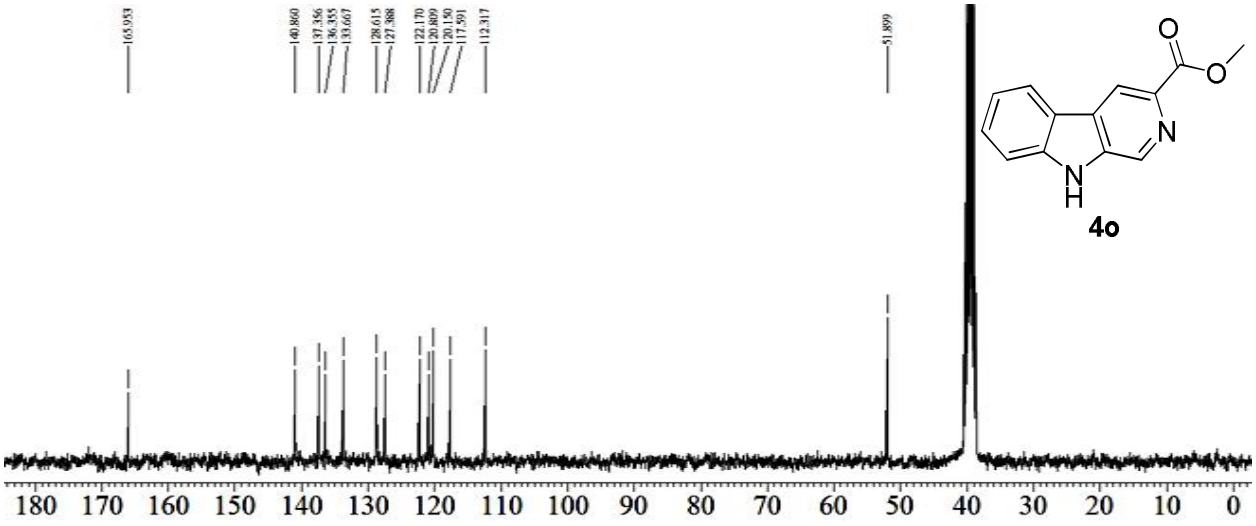


Methyl 9*H*-pyrido [3, 4-*b*] indole-3-carboxylate (4o)

NMR (300 MHz, DMSO−d_6)

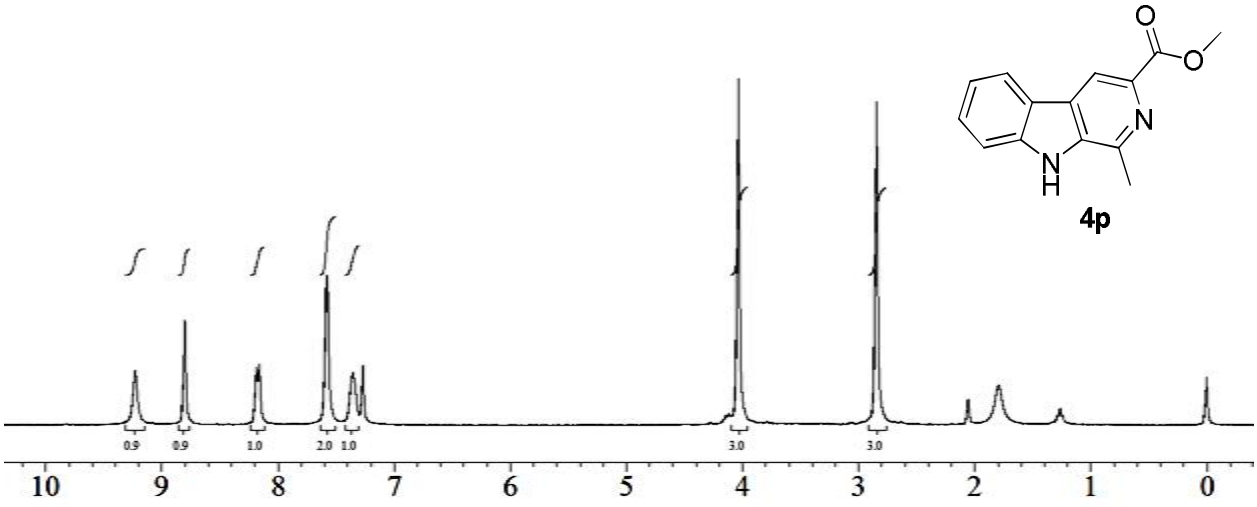


^{13}C NMR (75 MHz, DMSO−d_6)

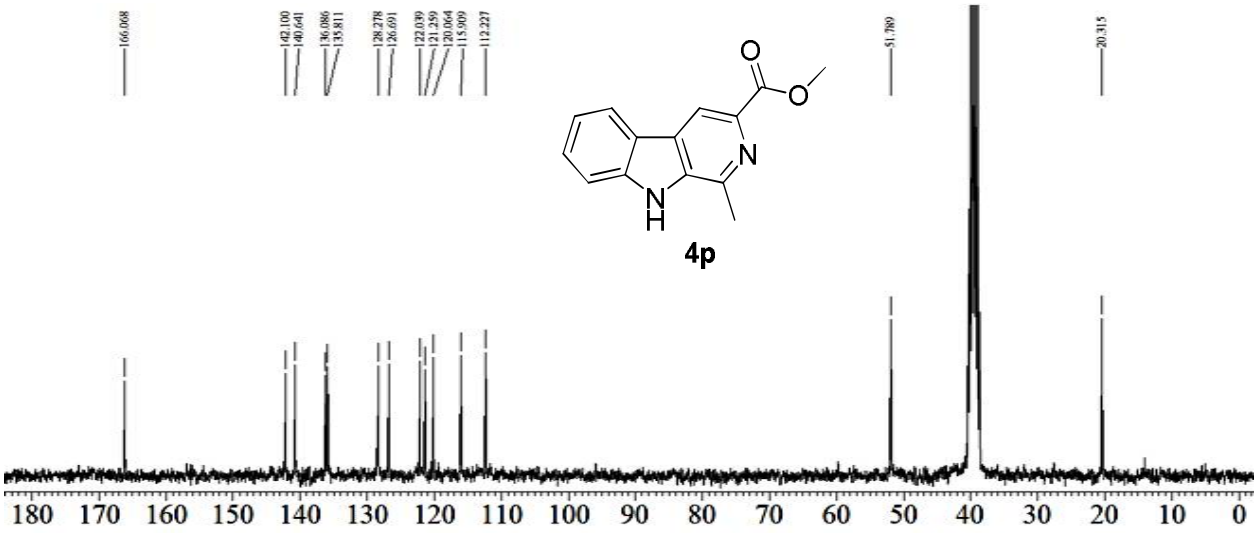


Methyl 1-methyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate (4p)

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

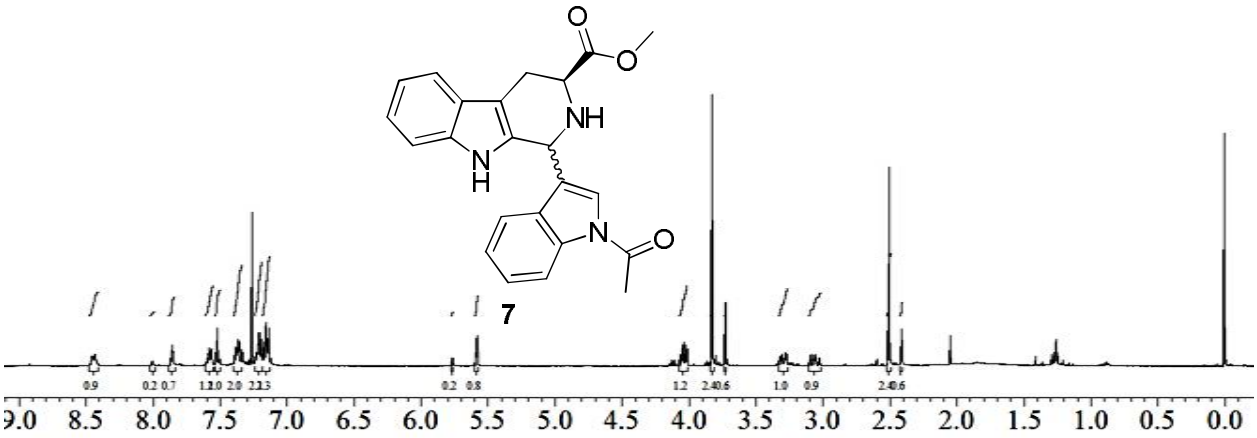


^{13}C NMR (75 MHz, DMSO−d_6)

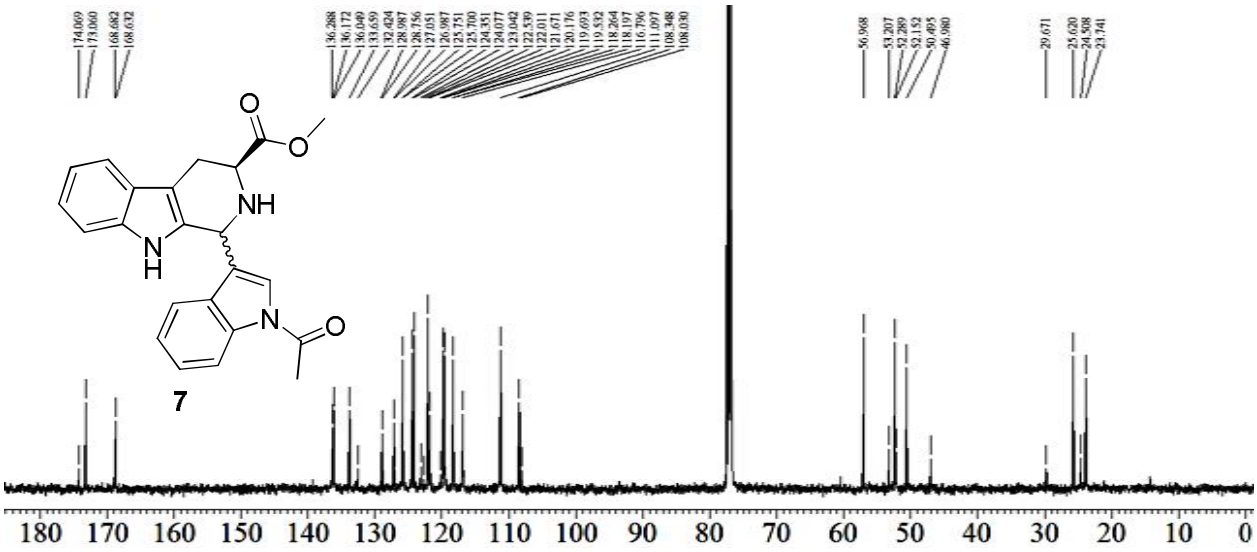


Methyl 1-(1-acetyl-1*H*-indol-3-yl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylate (7)

^{1}H NMR (400 MHz, $CDCl_3$)

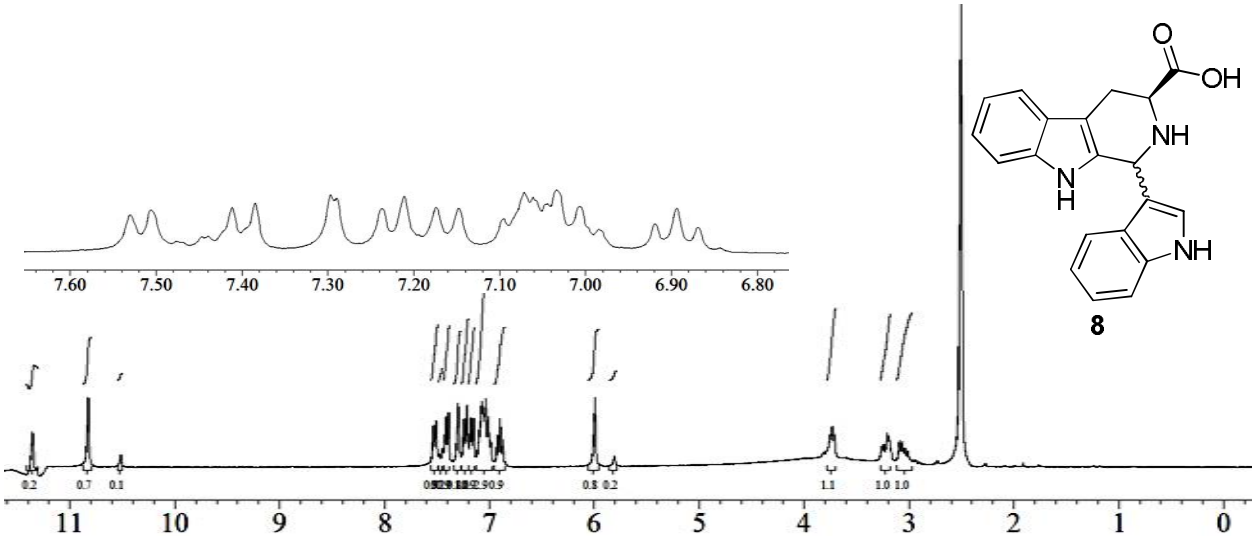


^{13}C NMR (125 MHz, $CDCl_3$)

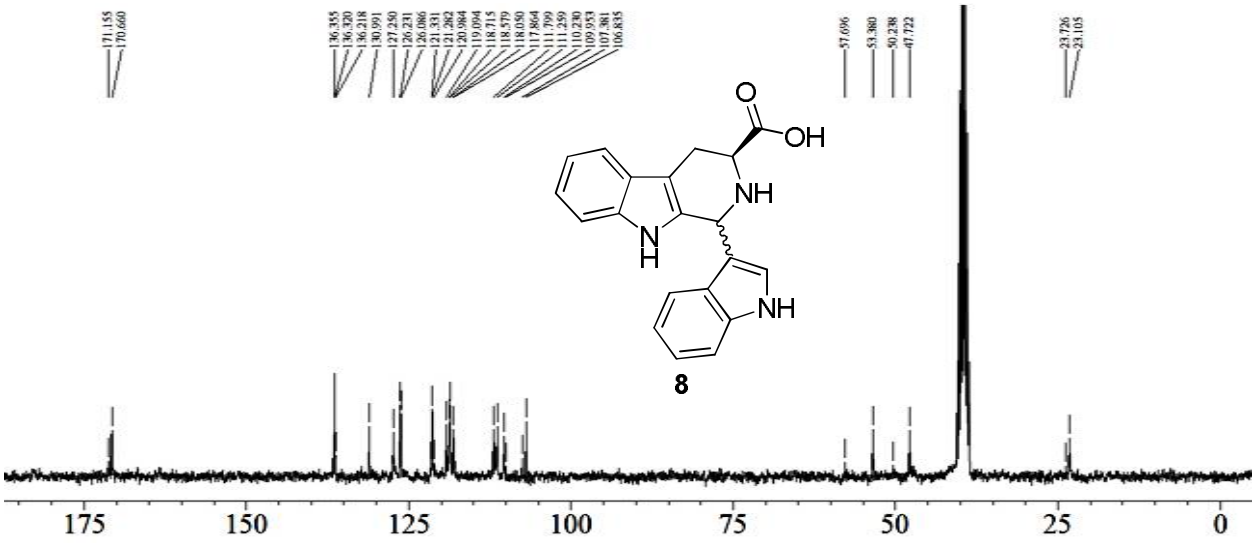


1-(1*H*-Indol-3-yl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylic acid (8)

^{1}H NMR (300 MHz, DMSO−d_6)

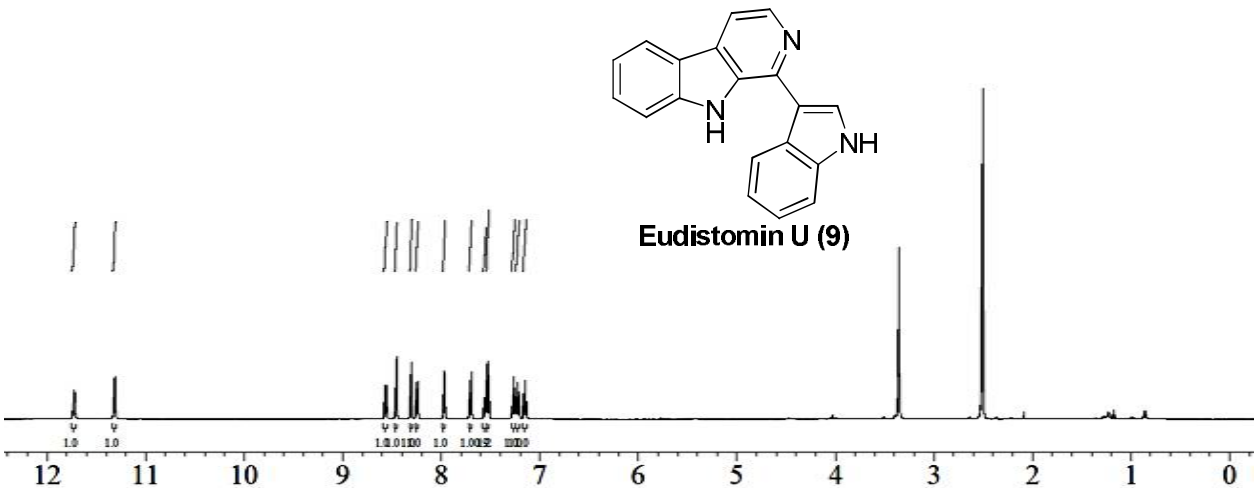


^{13}C NMR (75 MHz, DMSO−d_6)

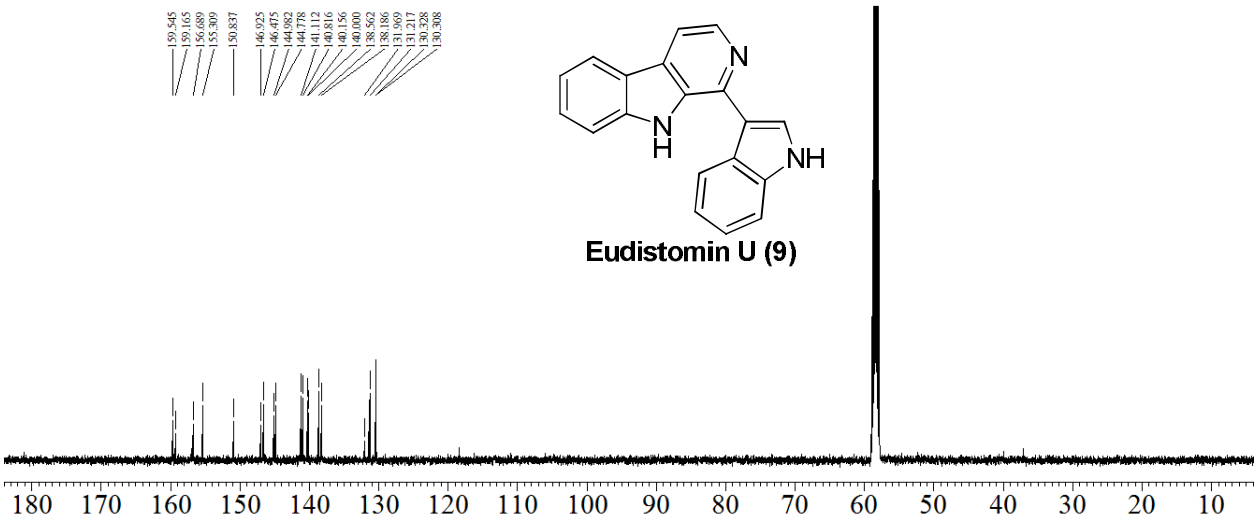


Eudistomin U (9):

[1]HNMR (500 MHz, DMSO−d_6)

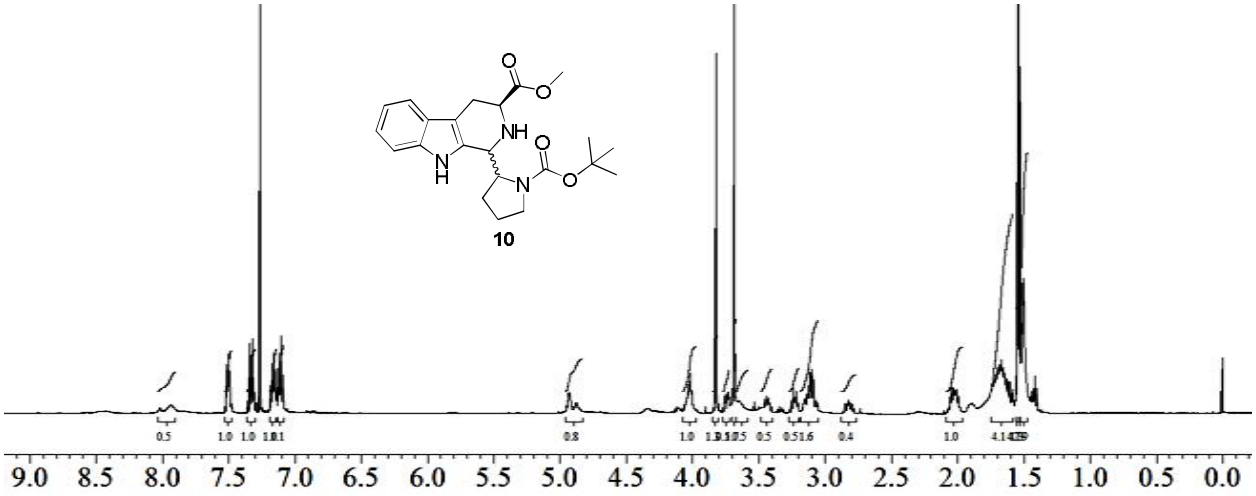


[13]C NMR (125 MHz, DMSO−d_6)

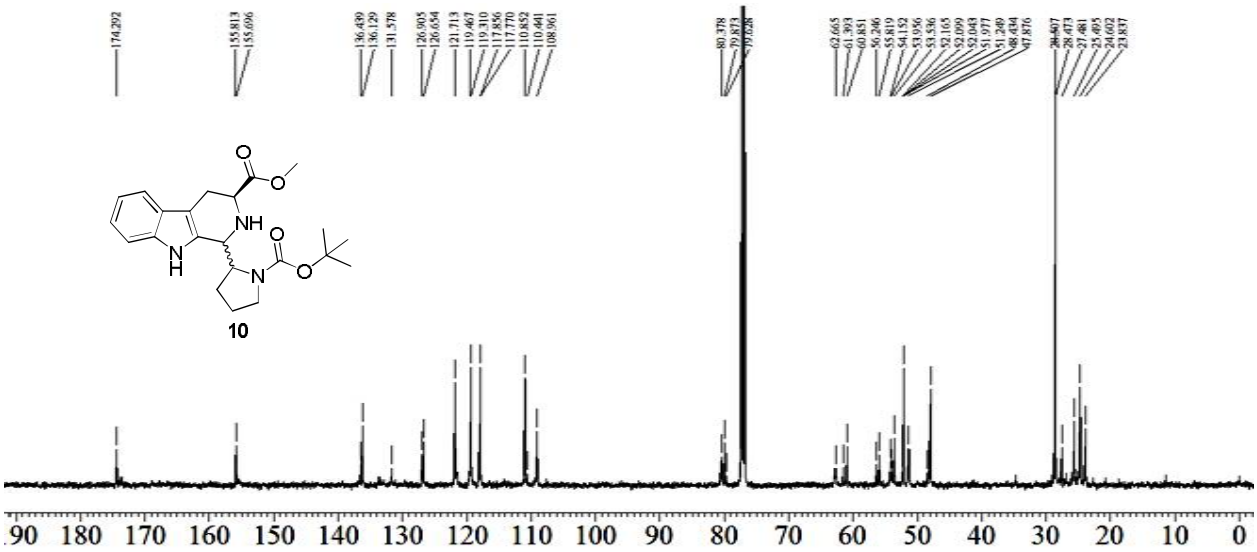


Methyl 1-(1-(*tert*-butoxycarbonyl)pyrrolidin-2-yl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylate (10)

^{1}H NMR (500 MHz, $CDCl_3$)

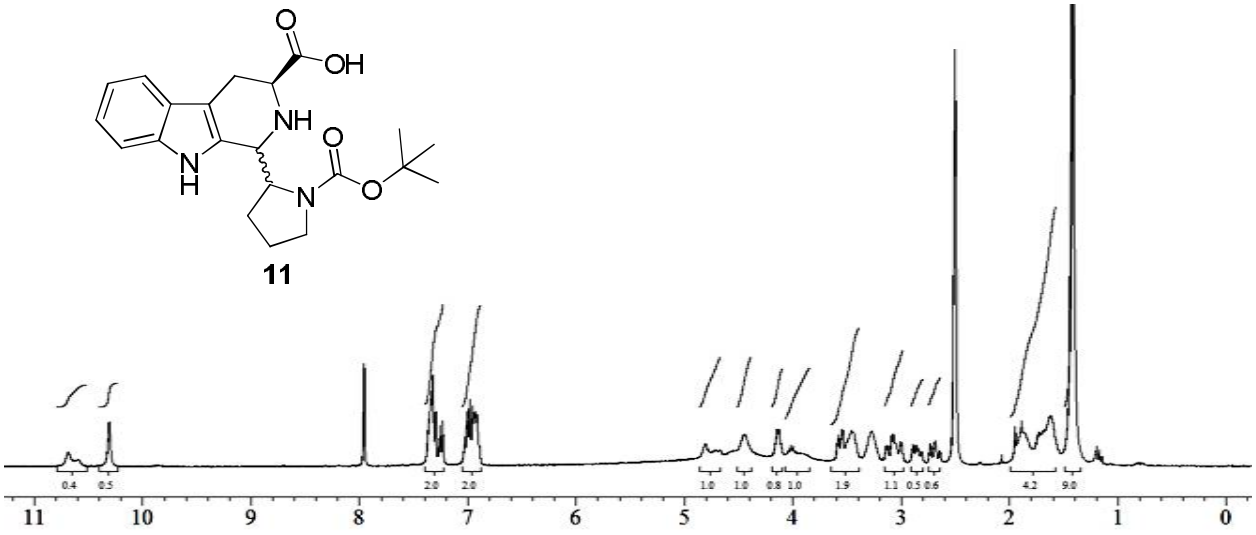


^{13}C NMR (125 MHz, $CDCl_3$)

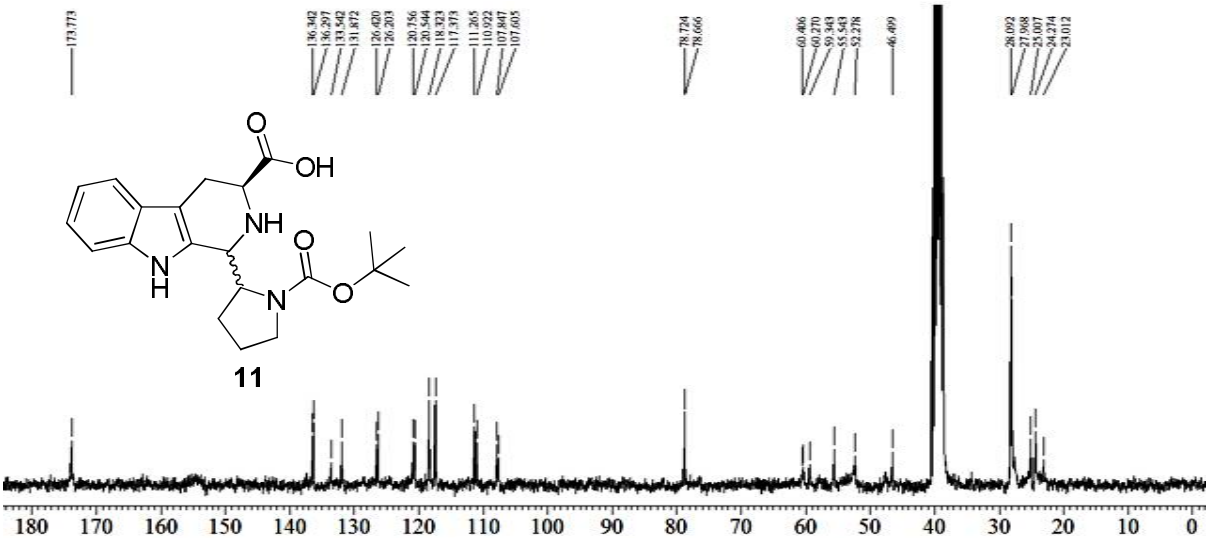


1-(1-(*tert*-butoxycarbonyl)pyrrolidin-2-yl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylic acid (11)

^{1}H NMR (300 MHz, $CDCl_3$ + DMSO−d_6)

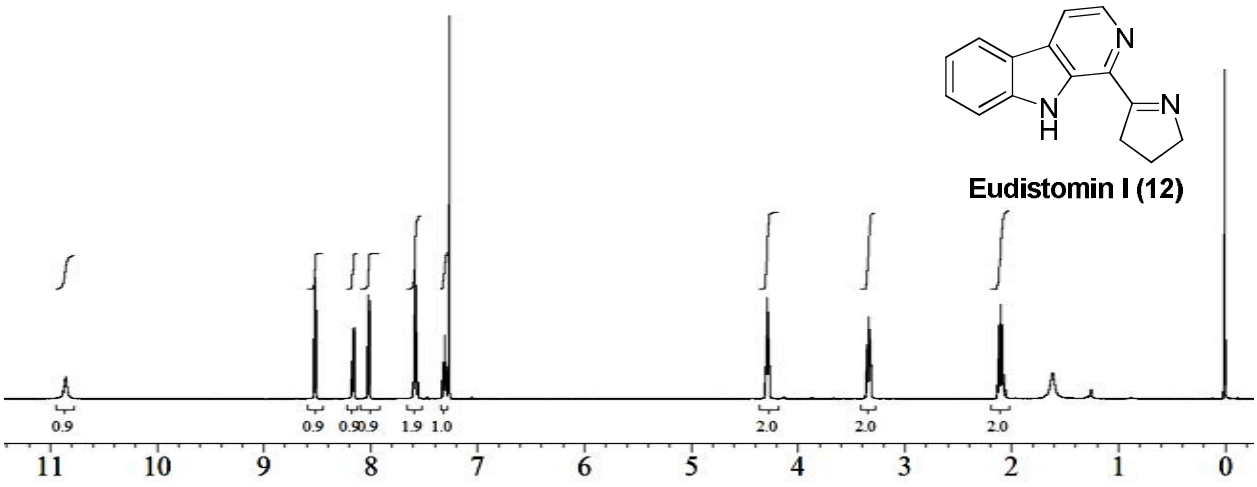


^{13}C NMR (75 MHz, DMSO−d_6)



Eudistomin I (12):

^{1}H NMR (500 MHz, $CDCl_3$)



^{13}C NMR (125 MHz, $CDCl_3$)