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A facile route to new potential helicating ligands

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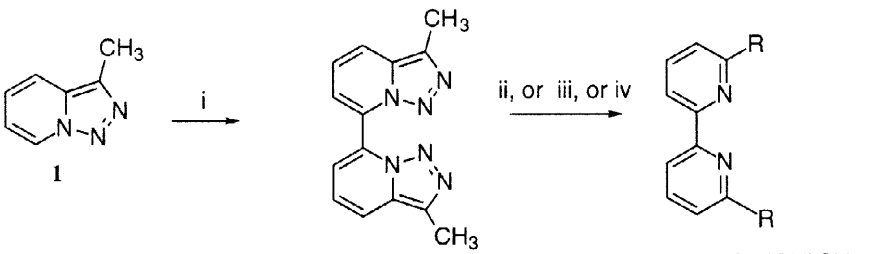
Abstract

The synthesis of new bitriazolopyridines, 6,6'-disubstituted-2,2'-bipyridines and 2-pyridyl-6-(2-pyridyl carbonyl)-2-pyridylmethanone potential helicating ligands from 3-(2-aryl)-[1,2,3]triazolo[1,5-*a*]pyridines is described.

Keywords: Triazolpyridines, lithium derivatives, dimerization, helicates.

1. Introduction

The synthetic chemical mimicry of the double-helix structural motif is an interesting area of research with intense activity in recent years.[1] The formation of helicates and helices incorporating metal ions has become an important synthetic tool. Oligopyridines and related compounds are more useful helicating ligands.[1,2] As we have recently discovered[3] a new route to 6,6'-disubstituted-2,2'-bipyridines **2** from triazolopyridine **1** (Scheme 1), we thought that this methodology could generate ligands which could form helicates. We wish to report here the use of the cited strategy to synthesise eight potential helicating ligands from 3-(2-aryl)-[1,2,3]triazolo[1,5-*a*] pyridines.



i) LDA, THF, -70°C, ii) H_2SO_4, H_2O, 95°C,
iii) AcOH, 118°C, iv) SeO_2, xylene, boil

2 a) R= $CH(OH)CH_3$
b) R= $CH(OAc)CH_3$
c) R= $COCH_3$

Scheme 1

2. Results and discussion

In order to have a multiple helical system in bipyridine derivatives, there should be a non-planar arrangement about the C-C bond between coordinated and non-coordinated rings. This will only be achieved if some of the potential donor atoms are coordinated to the metal and with this in mind, we designed ligands **6b-c**. Compounds **6** can be easily accessible by the sequence in scheme 2 (route a), if the route summarized above is general. Using compounds **3a-c**, we have tested the generality of the lithiation reaction to give bitriazolopyridine derivatives.

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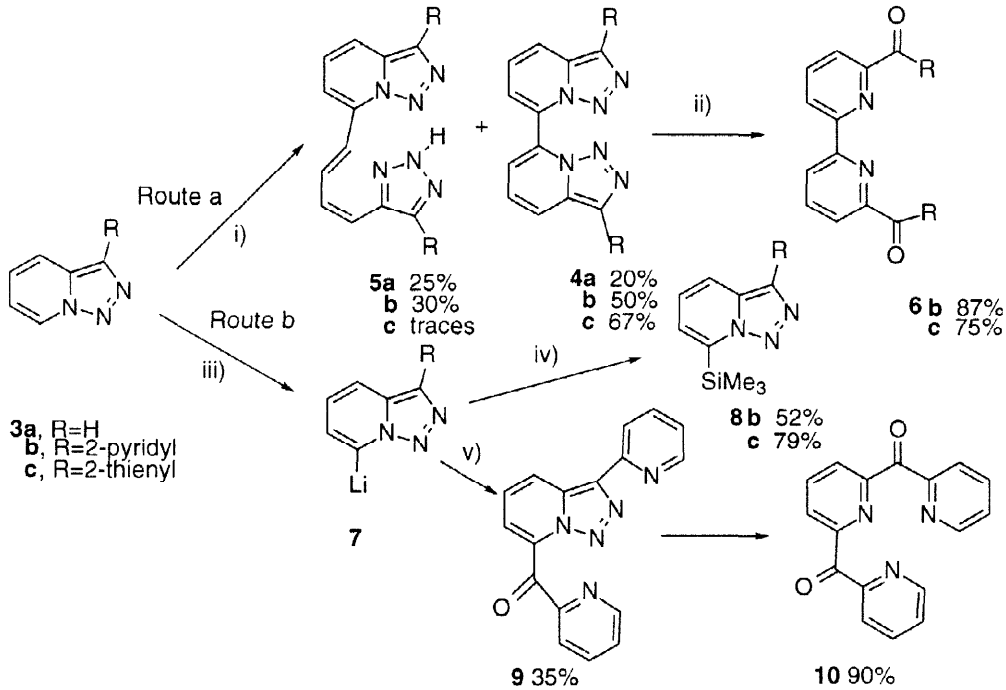
[1,2,3]Triazolo[1,5-*a*]pyridine **3a**[4] and 3-(2-pyridyl)-[1,2,3]triazolo[1,5-*a*]pyridine **3b** have been synthesized previously.[5,6] 3-(2-Thienyl)-[1,2,3]triazolo[1,5-*a*]pyridine **3c** is a new compound. It was synthesized from 2-pyridyl-2-thienylmethanone[7] by Bower's method.[4]



i) LDA, THF, -70°C, ii) H_2SO_4, H_2O, 95°C, or SeO_2 or AcOH
iii), LDA, THF, -40°C, iv) $ClSiMe_3$, v) PyCHO

Scheme 2

The lithiation of compounds **3** resulted in interesting reactions which were strongly temperature dependent. Dimers **4** were formed as the unique reaction compounds, in moderate yields, when the reactions were carried out at -70°C in THF as solvent and hydrolysed at the same temperature. The dienes **5** were also formed in these reactions when the mixture was allowed to rise to room temperature before hydrolysis. The lithiation reaction of triazolopyridine **3a** had been described at -40°C giving a 7-lithio derivative **7a**,[8] trapped by electrophiles. We now found that compounds **3b,c** also give the usual reaction at -40°C and 7-lithio derivatives **7b,c** were formed, which reacted with $ClSiMe_3$ to give **8b,c**. With **3c** the same results were found at 0-5°C. No competitive *ortho* directed lithiation by the 2-pyridyl group[9] to the C4 position was found with **3b** and **3c** did not lithiate in the thiophene ring. The 7-lithio derivative **7b** reacted with 2-pyridine carbaldehyde to form unstable diarylmethyl alkoxide intermediate which provide rapid access to ketone **9** by spontaneous air oxidation in work-up. Ring opening reactions of **4b,c** and **9** with sulfuric acid, selenium dioxide, or acetic acid gave directly the diketones **6b,c** and **10** respectively. Potentially compound **9** can also be used to prepare new quatertriazolopyridines.

Compounds **4b-c** have very low solubility in most common organic solvents. The compounds **4b,c**, **5**, **6**, **9** and **10** have interesting ligand structures and should be able to form polynuclear complexes with numerous metal ions. We are at present investigating the complexation of Cu(I) ions. The coordination chemistry of copper(I) salts with polydentate ligands has become an important research area with respect to the search for model compounds that can either mimic or even ideally duplicate one or more of the important specific physical and chemical properties of copper proteins, in which the metal center exists

in the reduced Cu(I) state. The Cu(I) complexes of **4b** and **6b** have been prepared by adding $Cu(NO_3)_2.6H_2O$ to a solution/suspension of the ligand in a methanol-water mixture (1:1), followed by excess $NaBF_4$. Reduction with a slight excess of sodium ascorbate results in the immediate appearance of the deep orange-red color characteristic of $[Cu(I)(bipyridine)_2]^+$ and related species.[10] The structure and properties of these complexes are currently under investigation and more accurate data must await crystal structure determination.

3. Experimental

Melting points were determined on a heated stage and are uncorrected. Nmr spectra were recorded on a Bruker AC250MHz instrument. HRMS (EI) determinations were made using a VG Autospec Trio 1000 (Fisons). Ir spectra were recorded as KBr discs.

[1,2,3]Triazolo[1,5-a]pyridine ***3a***.-
Prepared as described.[4]

3-(2-Pyridyl)-[1,2,3]triazolo[1,5-a]pyridine ***3b***.-
When it was prepared as described,[6] the yield was 26%. By manganese oxide oxidation of the intermediate hydrazone the yield was 86%. m.p. 125-127°C (hexane/ethyl acetate) (lit. 123-125°C hexane/ethyl acetate).

3-(2-Thienyl)-[1,2,3]triazolo[1,5-a]pyridine ***3c***.-
A mixture of 2-pyridyl-2-thienylmethanone (4.65g, 200mmol) and hydrazine (35% solution in water) (12ml, 200mmol) was heated for 1h under reflux. Then a solution of sodium hydroxide (30ml, 10% in water) was added and extracted with ether. The organic layer was dried and evaporated to give the corresponding hydrazone (4.27g, 85%), which was dissolved in chloroform without purification, and heated to reflux for 2h with manganese oxide (3.65g, 400mmol). Then was filtered and the solvent evaporated to give almost pure **3c** as a solid, crystallised from hexane (3.5g, 83%). m.p. 114-115°C. HRMS found for M^+ 201.0353; $C_{10}H_7N_3S$ requires 201.0361. Nmr ^1H ($CDCl_3$) δ 8.70(d, J=7.0Hz, 1H); 7.96(d, J=9.1Hz, 1H); 7.55(d, J=3.6Hz, 1H); 7.38(d, J=5.1Hz, 1H); 7.34(dd, J_1=7.0Hz, J_2=9.1Hz, 1H); 7.16(dd, J_1=5.1Hz, J_2=3.6Hz, 1H); 7.05(d, J=7.0Hz, 1H). Nmr ^{13}C ($CDCl_3$) δ, 133.28(C); 132.72(C); 129.68(C); 127.75(CH); 125.90(CH); 125.41(CH); 125.11(CH); 124.03(CH); 118.21(CH); 115.85(CH). ν_{max} 3081, 1622, 1520, 1378, 1210, 722 cm^{-1}.

General procedure for lithiation reactions of compounds 3 at -70°C.-
A solution of *n*-butyllithium in hexane (1.6M) was added to an equimolar amount of diisopropylamine, freshly distilled from KOH, at -70°C under argon. Equimolar amount of a solution of the appropiate compound **3** in anhydrous THF was added with stirring. A deep red colour developed. The mixture was kept at -70°C (10h), and then at room temperature (48h), during which time the solution became yellow. Then was hydrolysed with a saturated solution of ammonium chloride. The isolation and purification procedures are given for each compound.

7,7'-Bi[1,2,3]triazolo[1,5-a]pyridine ***4a*** *and 1-([1,2,3]triazolo[1,5-a]pyridin-7-yl)-4-(1H-[1,2,3]triazol-4-yl)-1,3-butadiene* ***5a***.-
The reaction was carried out with **3a** (1g, 8.4mmol). Extraction with dichloromethane gave, after drying and evaporation of the organic solvent, a residue which was purified by chromatography. Elution with ethyl acetate/hexane (8:2) gave starting material (0.3g).

Further elution with ethyl acetate gave the bitriazolopyridine **4a** as a yellow solid (0.14g, 20%). m.p. >350°C. HRMS found for M^+ 236.0818; $C_{12}H_8N_6$ requires 236.0810. Nmr ^1H δ ($CDCl_3$) 8.24(s, 2H); 7.94(d, J=9.1Hz, 2H); 7.86(d, J=7.0Hz, 2H); 7.43(dd, J_1=9.1, J_2=7.0Hz, 2H). Nmr ^{13}C δ ($CDCl_3$) 134.41(C); 128.52(C); 126.64(CH); 124.85(CH); 119.56(CH); 118.64(CH). ν_{max} 3100, 2950, 1620, 1120, 780, 720 cm^{-1}. Acidification with 10% HCl of the aqueous phase, extraction with dichloromethane gave the butadiene **5a** (0.17g, 25%) almost pure. m.p. 190-191°C. HRMS found for M^+ 238.0963; $C_{12}H_{10}N_6$ requires 238.0967. Nmr ^1H δ (DMSO) 8.29(s, 1H); 8.09(bs, 1H); 7.98(dd, J_1=10.9Hz, J_2=15.7Hz, 1H); 7.88(d, J=8.0Hz, 1H); 7.45-7.39(m, 3H), 7.24(dd, J_1=10.9Hz, J_2=15.7Hz, 1H); 6.98(d, J=15.7 Hz, 2H). Nmr ^{13}C δ (DMSO) 143.35(C); 136.46(CH); 134.66(C); 134.46(C); 130.87(CH); 128.75(CH); 126.26(CH); 126.02(CH); 124.93(CH); 122.36(CH); 116.72(CH); 114.30(CH). ν_{max} 3600-2400, 1500, 980, 800 cm^{-1}.

7,7'-Bi-3-(2-pyridyl)[1,2,3]triazolo[1,5-a]pyridine ***4b*** *and 1-[3-(2-pyridyl)[1,2,3] triazolo [1,5-a]pyridin-7-yl)-4-[5-(2-pyridil)-1H-[1,2,3]triazol-4-yl)]-1,3-butadiene* ***5b****.-*

The reaction was done with **3b** (1g, 6mmol). A yellow solid was formed, filtered and the solid identified as the bitriazolopyridine **4b** (0.35g, 50%). m.p. >350°C. HRMS found for M^+ 390.1340; $C_{22}H_{14}N_8$ requires 390.1341. Nmr ^1H δ (CF_3CO_2D) 9.07(d, J=6.9Hz, 2H); 8.63-8.45(m, 8H); 8.00(dd, J_1=7.0Hz, J_2=8.0Hz, 2H); 7.62(dd, J_1=J_2=7.0Hz, 2H). Nmr ^{13}C δ (CF_3CO_2D) 146.17(C); 145.30(CH); 144.14(C); 133.07(C); 132.72(CH); 129.54(C); 126.83(CH); 124.79(CH); 122.70(CH); 121.09(CH); 118.55(CH). ν_{max} 3068, 1572, 1420, 783 cm^{-1}. The filtrate was extracted with dichlomethane. Drying and evaporation of the solvent gave a residue purified by silica chromatography. Elution with ethyl acetate/hexane (9:1) gave starting material (0.3g). Further elution with ethyl acetate gave the butadiene **5b** (0.21g, 30%). m.p. 200-202°C (ethyl acetate/hexane). HRMS found for M^+ 392.1501; $C_{22}H_{16}N_8$ requires 392.1498. Nmr ^1H δ (DMSO) 9.19(d, J=6.9Hz, 1H); 8.75(m, 2H); 8.12-8.04(m, 2H); 7.96-7.87(m, 2H); 7.81-7.51(m, 4H), 7.43-7.29(m, 4H); 7.00(d, J=15Hz, 1H). Nmr ^{13}C δ (DMSO) 154.95(C); 151.54(C); 150.79(C); 149.77(CH); 137.92(CH); 137.47(CH); 136.35(C); 134.08(CH); 132.83(CH); 132.17(CH); 131.54(C); 128.23(CH); 128.00(C); 126.32(CH); 123.12(CH); 122.61(C); 121.61(CH); 120.98(CH); 120.48(CH); 118.64(CH); 116.92(CH); 115.47(CH). ν_{max} 3600-2400, 1600, 1500, 980, 715 cm^{-1}.

7,7'-Bi-3-(2-thienyl)[1,2,3]triazolo[1,5-a]pyridine ***4c****.-*

The reaction was done with **3c** (0.75g, 3.73mmol). A yellow solid was formed, filtered and the solid identified as the bitriazolopyridine **4c** (0.175g). The filtrate was extracted with dichlomethane, after drying and evaporation of the solvent, gave a residue purified by silica chromatography. Elution with ethyl acetate/hexane (1:1) gave starting material (0.45g). Further elution with ethyl acetate gave more **4c** (0.025g, total yield 67%). m.p.>350°C. HRMS found for M^+ 400.0584; $C_{20}H_{12}N_6S_2$ requires 400.0565. Nmr ^1H δ ($CDCl_3$) 8.18(d, J=8.8Hz, 2H); 7.94(d, J=7.3Hz, 2H); 7.62(d, J=3.7Hz, 2H); H); 7.52(dd, J_1=7.3Hz, J_2=8.8Hz, 2H); 7.41(d, J=5.1Hz, 2H); 7.20(dd, J_1=3.7Hz, J_2=5.1Hz, 2H). Nmr ^{13}C δ (CF_3CO_2D) 131.70(C); 131.60(CH); 131.15(C); 130.74(CH); 129.42(CH); 128.59(CH); 127.12(CH); 126.53(C); 122.43(CH); 120.23(C). ν_{max} 3015, 1619, 1218, 717 cm^{-1}. Further elution gave traces of **5c**, detected by ^1H nmr.

General procedure for lithiation reactions of compounds ***3*** *at -40°C.-*

A solution of *n*-butyllithium in hexane (1.6M) was added to an equimolar amount of diisopropylamine, freshly distilled from KOH, at -40°C under argon. Equimolar amount of a solution of the appropiate compound **3** in anhydrous THF was added with stirring. A deep red colour developed. The mixture was kept at -40°C (4h), and then a solution of the corresponding electrophile in dichloromethane was added. The mixture was kept at room

temperature overnight. Hydrolysis with a saturated solution of ammonium chloride and extraction with dichlomethane gave, after drying and evaporation of the organic solvent, a residue which was purified by chromatography on silica gel.

Trimethyl[3-(2-pyridyl)[1,2,3]triazolo[1,5-a]pyridin-7-yl]silane ***8b.-***

The reaction was done with **3b** (200mg, 1.02mmol) and trimethylsilyl chloride (1.0ml, 1M solution). Elution with ethyl acetate/hexane (2:1) gave a white solid (68mg, 52%) identified as **8b**. m.p. 108-110°C (AcOEt / hexane). HRMS found for M^+ 268.1143; $C_{14}H_{16}N_4Si$ requires 268.1144. Nmr ^1H δ ($CDCl_3$) 8.61(d, J=9.1Hz, 1H); 8.57(d, J=5.1Hz, 1H); 8.27(d, J=8.0Hz, 1H); 7.69(dd, J_1=8.0Hz, J_2=7.6Hz, 1H); 7.22(dd, J_1=6.6Hz, J_2=9.1Hz, 1H); 7.10(dd, J_1= 5.1Hz, J_2=7.6Hz, 1H); 7.01(d, J=6.6Hz, 1H); 0.45(s, 9H). Nmr ^{13}C δ ($CDCl_3$) 152.33(C); 149.20(CH); 140.82(C); 136.70(C); 136.46(CH); 131.27(C); 125.55(CH); 122.82(CH); 121.67(CH); 121.48(CH); 120.15(CH); -2.24 (CH_3). ν_{max} 2947, 1598, 1252, 1090, 1031, 807, 747 cm^{-1}. Further elution gave starting material (100mg).

Trimethyl[3-(2-thienyl)[1,2,3]triazolo[1,5-a]pyridin-7-yl]silane ***8c.-***

The reaction was done with **3c** (500mg, 2.48mmol) and trimethylsilyl chloride (3.2ml, 1M solution). Elution with ethyl acetate/hexane (3:2) gave a yellow solid (0.54g, 79%) identified as **8c**. m.p. 110-113°C (AcOEt / hexane). HRMS found for M^+ 273.0769; $C_{13}H_{15}N_3SSi$ requires 273.0756. Nmr ^1H δ ($CDCl_3$) 7.95(dd, J_1=1.1Hz, J_2=9.1Hz, 1H); 7.56(dd, J_1=0.7Hz, J_2=3.6Hz, 1H); 7.36(dd, J_1= 5.1Hz, J_2=1.1Hz, 1H); 7.27(dd, J_1=6.5Hz, J_2=9.1Hz, 1H); 7.17(dd, J_1=3.6Hz, J_2=5.1Hz, 1H); 7.07(dd, J_1=1.1Hz, J_2=6.5Hz, 1H); 0.54(s, 9H). Nmr ^{13}C δ ($CDCl_3$) 141.25(C); 133.87(C); 132.80(C); 128.87(C); 127.60(CH); 124.90(CH); 124.52(CH); 123.24(CH); 122.20(CH); 118.42(CH); -2.34 (CH_3). ν_{max} 2957, 1608, 1239, 846, 753 cm^{-1}. Whith *n*-butyllithium at 0°C the same results were obtained.

2-Pyridyl[3-(2-pyridyl)[1,2,3]triazolo[1,5-a]pyridin-7-yl]methanone ***9.-***

The reaction was done with **3b** (1.5g, 7.65mmol) and 2-pyridylcarbaldehyde (0.86g, 7.65mmol) in THF solution. Elution with ethyl acetate/hexane (2:3) gave starting material (170mg). Further elution with ethyl acetate/hexane (5:3) gave compound **9** (700mg, 35%) as a white solid. m.p 194-195°C (AcOEt / Hexane). HRMS found for M^+ 301.0971. $C_{17}H_{11}N_5O$ requires 301.0963. Nmr ^1H δ ($CDCl_3$) 8.80(d, J=4.0Hz, 1H); 8.72(d, J=6.9Hz, 1H); 8.53(dd, J_1=1.4Hz, J_2=7.3Hz, 1H); 8.22(d, J=8.8Hz, 1H); 8.09-7.89(m, 4H); 7.54 (ddd, J_1=1.1Hz, J_2=4.7Hz, J_3=7.6Hz, 1H); 7.17(dd, J_1=7.6Hz, J_2=6.9Hz, 1H); 6.99(ddd, J_1=1.1Hz, J_2=6.9Hz, J_3=6.9Hz, 1H). Nmr ^{13}C δ ($CDCl_3$), 192.90(CO); 155.06(C); 153.26(C); 151.28(C); 148.98(CH); 137.46(CH); 136.63(CH); 132.24(C); 126.45(CH); 126.02(CH); 125.17(CH); 125.06(CH); 123.21(CH); 123.09(CH); 121.06(CH); 115.90(CH). ν_{max} 3100, 2950, 1650, 1590, 1310, 1160, 740 cm^{-1}.

General procedure for ring opening reactions of triazolopyridines.-

A solution of the triazolopyridine (*ca.* 200mg) and selenium dioxide (2 equivalents) in aqueous sulfuric acid (10ml, 2.5M) was heated to reflux for 10h. Then was neutralized with aqueous saturated solution of sodium bicarbonate and extracted with dichloromethane. The organic solvent was dried, and evaporated. The residue was purified by silica chromatography.

2-Pyridyl-6-[6-(2-pyridylcarbonyl)-2-pyridyl]-2-pyridylmethanone ***6b.-***

Elution with ethyl acetate/hexane (3:1) gave **6b** in a 87% yield. m.p. 215-216°C (AcOEt). HRMS found for M^+ 366.1117; $C_{22}H_{14}N_4O_2$ requires 366.1131. Nmr ^1H δ ($CDCl_3$) 8.73(d, J=4.4Hz, 2H); 8.42(dd, J_1=1.0Hz, J_2=7.8Hz, 2H); 8.06(dd, J_1=4.0Hz, J_2=7.0Hz, 2H); 8.05(dd, J_1=1.0Hz, J_2=7.0Hz, 2H); 7.89(dd, J_1=7.8Hz, J_2=7.0Hz, 2H); 7.84(dd, J_1=1.0Hz, J_2=7.0Hz, 2H);

7.45(ddd, J_1=1.0Hz, J_2=7.0Hz, J_3=4.0Hz, 2H). Nmr ^{13}C δ ($CDCl_3$) 192.72(CO); 154.67(C); 154.26(C); 153.42(C); 149.21(CH); 137.68(CH); 136.54(CH); 132.24(C); 126.26(CH); 125.50(CH); 125.26(CH); 123.98(CH). ν_{max} 2940, 2900, 1685, 1580, 1440, 960, 760 cm^{-1}. Compound **6b** was also formed when the ring opening reaction was done with acetic acid (60%) or 2.5M aqueous sulfuric acid (75%).

2-Thienyl-6-[6-(2-thienylcarbonyl)-2-pyridyl]-2-pyridylmethanone ***6c***.-

Elution with ethyl acetate/hexane (1:1) gave **6c** as a yellow solid (75% yield). m.p. 177-180°C (AcOEt). HRMS found for M^+ 376.0334; $C_{20}H_{12}N_2O_2S_2$ requires 376.0340. Nmr ^1H δ ($CDCl_3$) 8.82(d, J=7.7Hz, 2H); 8.34(dd, J_1=4.0Hz, J_2=1.1Hz, 2H); 8.23(d, J=8.0Hz, 2H); 8.06(dd, J_1=7.7Hz, J_2=8.0Hz, 2H); 7.77(dd, J_1=1.1Hz, J_2=5.1Hz, 2H); 7.19(dd, J_1=4.0Hz, J_2=5.1Hz, 2H). Nmr ^{13}C δ ($CDCl_3$) 183.00(CO); 154.56(C); 153.28(C); 138.47(CH); 136.65(CH); 136.53(CH); 127.49(CH); 125.00(CH); 124.23(CH). ν_{max} 2931, 1700, 1628, 1582, 1269, 807 cm^{-1}. Compound **6c** was also formed when the ring opening reaction was done with acetic acid (45%).

2-Pyridyl-6-(2-pyridylcarbonyl)-2-pyridylmethanone ***10***.-

Elution with ethyl acetate/ hexane (8:2) gave **10** (173mg, 90%) as a white solid. m.p. 120-123°C (AcOEt / hexane). HRMS found for M^+ 289.0846; $C_{17}H_{11}N_3O_2$ requires 289.0851. Nmr ^1H δ ($CDCl_3$) 8.69(ddd, J_1=0.7Hz, J_2=1.1Hz, J_3=4.7Hz, 2H).); 8.25(d, J=7.7Hz, 2H); 8.13(ddd, J_1=1.1Hz, J_2=0.7Hz, J_3=8.1Hz 2H); 8.06(t, J=7.7Hz, 1H); 7.74(ddd, J_1=1.1Hz, J_2=8.1Hz, J_3=7.6Hz, 2H); 7.40(ddd, J_1=1.1Hz, J_2=4.7Hz, J_3=7.6Hz, 2H. Nmr ^{13}C δ ($CDCl_3$) 191.58(CO); 153.32(C); 153.18(C); 149.03(CH); 137.63(CH); 136.30(CH); 127.25(CH); 126.28(CH); 125.92(CH). ν_{max} 3070, 1680, 1570, 850 cm^{-1}.

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