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Short Communication

A convenient ultrasound-promoted regioselective synthesis of fused polycyclic 4-aryl-3-methyl-4,7-dihydro-1*H*-pyrazolo[3,4-b]pyridines

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ABSTRACT

Fused polycyclic 4-aryl-3-methyl-4,7-dihydro-1*H*-pyrazolo[3,4-b]pyridines were obtained in a three-component regioselective reaction of 5-amino-3-methyl-1*H*-pyrazole, 2*H*-indene-1,3-dione and arylaldehydes in ethanol under ultrasound irradiation. This rapid method produced the products in short reaction times (4–5 min) and excellent yields (88–97%).

1. Introduction

Pyrazolo-fused compounds have been of interest due to their wide range of biological and pharmacological properties [1–3]. In particular pyrazolopyridines have attracted many interests in recent years as possible anti-viral agent, [4,5] potent p38 kinase inhibitors [6], HIV reverse transcriptase inhibitors [7], and inhibitors of cGMP degradation, together with several herbicidal and fungicidal activities [8,9]. Some derivatives exhibit potential antimalarial properties [10,11]. Others show intense fluorescence in the bluegreen region and have been considered for applications as fluorescence standards and luminophores in organic light emitting diodes [12], bactericidal activity [13], and also used as vasodilators [14] or evaluated for CCR1 antagonists and enzymatic inhibitory activities [15,16]. Therefore several protocols for the synthesis of pyrazolopyridines in recent years have been reported respect to their different structures [17–19].

On the other hand, green chemistry has become a major driving force for organic chemists to develop environmentally benign routes for the preparation of organic compounds of synthetic and biological values. For example, the possibility of performing reactions under ultrasound irradiations to enhance the reaction efficiency from both economic and ecological points of view has given to this kind of procedures a remarkable synthetic value and received a great attention [20].

2. Results and discussion

As part of our program aimed at developing new selective and environmentally friendly methodologies for the preparation of heterocyclic compounds [21], we describe here an efficient method for the regioselective synthesis of new derivatives of pyrazolopyridines.

Multicomponent reactions of 1*H*-pyrazol-5-amines, aldehydes and CH-acid compounds due to formation of different condensation products depending on the specific conditions have recently attracted the interest of many chemists [22]. In this report we have devised an efficient one-pot three-component reaction for the synthesis of novel fused tetracyclic derivatives of pyrazolopyridines (**4a–l**) from 5-amino-3-methyl-1*H*-pyrazole (**1**), 2*H*-indene-1,3-dione (**2**) and aldehydes (**3a–l**) under ultrasound irradiation (Scheme 1).

Equimolar amounts of reactants **1**, **2** and **3** (Table 2 entries 1–3, 0.5 eq.) in ethanol were placed into Pyrex-glass open vessel and irradiated at 60 °C by ultrasound (45 kHz) to produce desired pyrazolopyridine derivatives (**4a–l**) in 4–5 min with 88–97% yields (Tables 1 and 2). A control reaction on substrates **3a**, **3e** and **3j** in refluxing ethanol, using the same ratio of the reactants furnished the related pyrazolopyridines in lower yields and much longer reaction times (60–75 min) (Tables 1 and 2).

Interestingly this one-pot multicomponent approach also afforded an efficient protocol for the synthesis of bis-pyrazolopyridines **4j–l** (Fig. 1) in high yields (90–92%) (Table 2). The structures of all the products were fully characterized by spectroscopic (IR, ^{1}H NMR, ^{13}C NMR) and elemental analyses. In this study the main

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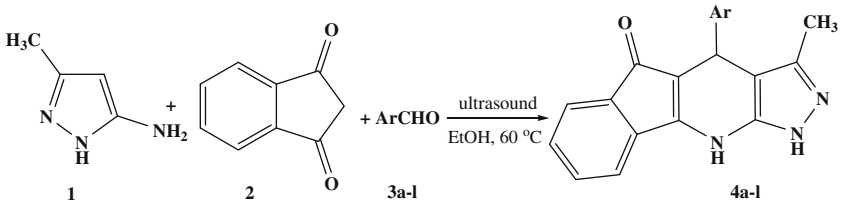


Ar = *o*-$NO_2C_6H_4$, *m*-$NO_2C_6H_4$, *p*-$NO_2C_6H_4$, *m*-BrC_6H_4, *m*-ClC_6H_4, *p*-ClC_6H_4, 2,4-$Cl_2C_6H_3$, 3-indoyl, 5-NO_2-2-furyl, etc.

Scheme 1. Synthesis of fused polycyclic 4-aryl-3-methyl-4,7-dihydro-1*H*-pyrazolo[3,4-b] pyridines.

Table 1
Synthesis of 4-aryl-3-methyl-4,7-dihydro-1*H*-pyrazolo[3,4-b]pyridines (**4a–i**) under ultrasound irradiation.

Entry	Reagents	Time (min)	Product	Yield (%)[a,b]
1	**1** + **2** + 2-$NO_2C_6H_4CHO$	4 (60)[c]	**4a**	97 (85)[c]
2	**1** + **2** + 3-$NO_2C_6H_4CHO$	5	**4b**	93
3	**1** + **2** + 4-$NO_2C_6H_4CHO$	4	**4c**	96
4	**1** + **2** + 3-BrC_6H_4CHO	5	**4d**	88
5	**1** + **2** + 2-ClC_6H_4CHO	5 (65)[c]	**4e**	91 (78)[c]
6	**1** + **2** + 3-ClC_6H_4CHO	5	**4f**	88
7	**1** + **2** + 4-ClC_6H_4CHO	5	**4g**	89
8	**1** + **2** + 2,4-$Cl_2C_6H_3CHO$	4	**4h**	95
9	**1** + **2** + 5-NO_2–furyl–CHO	4	**4i**	94

[a] Isolated yields.
[b] Identified by spectroscopic analyses (IR, ^{1}H NMR,^{13}C NMR).
[c] Equimolar amounts of 5-amino-3-methyl-1*H*-pyrazole 1 (10 mmol), 2*H*-indene-1,3-dione **2** (10 mmol) and aldehyde **3** (10 mmol) were refluxed in EtOH (15 mL) in the absence of sonication.

features of pyrazolodihydropyridine formation are the appearance of two separate singlets for NH protons in downfields and characteristic CH signal (^{1}H NMR: 4.82–5.56 ppm; ^{13}C NMR: 26.2–35.2 ppm). In this method no trace of pyridine products which could have been the result of oxidation of the formed pyrazolodihydropyrindes (**4a–l**) could be detected.

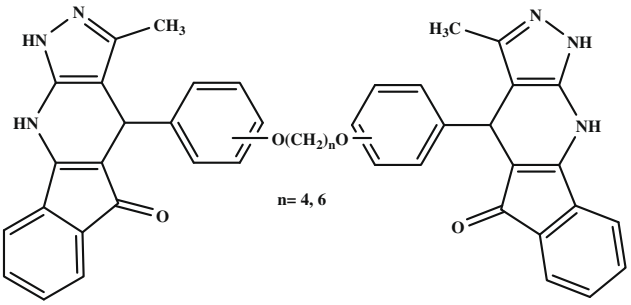
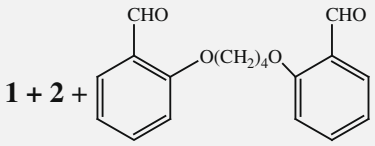
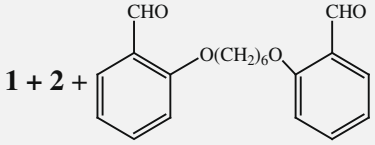
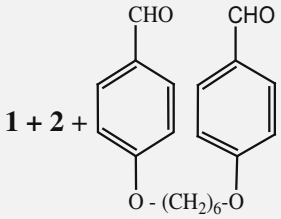


Fig. 1. Bis-pyrazolopyridines **4j–l**.

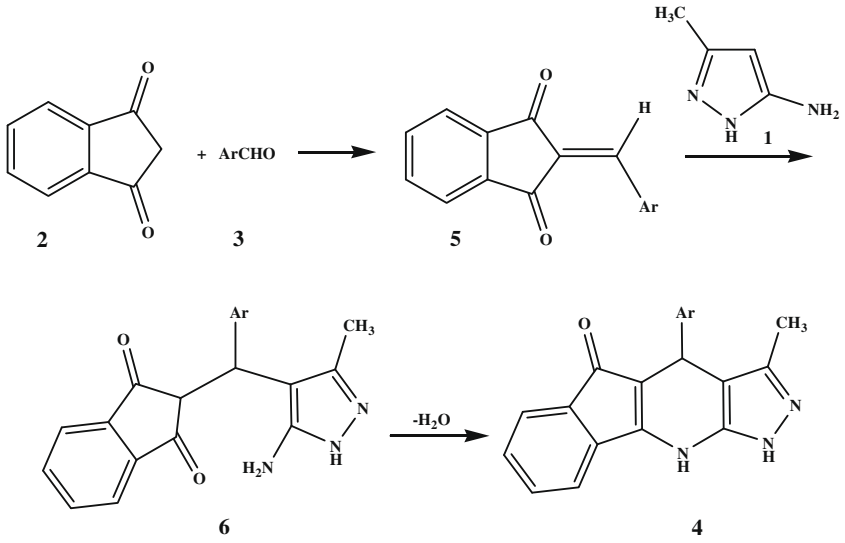
Mechanistically the formation of the products (**4a–l**) can be visualized by initial Knoevenagel condensation of aryl aldehyde (**3**) and 2*H*-indene-1,3-dione (**2**), followed by a Michael type nucleophilic addition of C-4 of the pyrazole ring to the enone intermediate **(5)** and subsequent cyclodehydration to furnish the desired compounds (Scheme 2).

In conclusion, this one-pot three component protocol under ultrasound irradiation provides a regioselective, fast and practical method for the preparation of fused polycyclic pyrazolo[3,4-b]pyridines from 5-amino-3-methyl-1*H*-pyrazole, 2*H*-indene-1,3-dione

Table 2
Synthesis of bis-pyrazolopyridines **4j–l** under ultrasound irradiation.

Entry	Reagents	Time (min)	Product	Yield (%)[a,b]
1		5 (75)[c]	**4j**	92 (80)
2		5	**4k**	90
3		5	**4l**	91

[a] Isolated yields.
[b] Identified by spectroscopic analyses (IR, ^{1}H NMR,^{13}C NMR).
[c] A solution of 5-amino-3-methyl-1*H*-pyrazole 1 (10 mmol), 2*H*-indene-1,3-dione **2** (10 mmol) and aldehyde **3** (5 mmol) were refluxed in EtOH (15 mL) in the absence of sonication.



Scheme 2. Mechanism of formation of pyrazolopyridines **4a–l.**

and various aryl aldehydes in short reaction times and in excellent yields, which pave the way for assessment of pharmalogical activities of these novel pyrazolopyridine derivatives.

3. Experimental

3.1. General

Melting points were measured on an Electrothermal 9100 apparatus. For the ultrasound reactions, ultrasound apparatus Astra 3D (9.5 L, 45 kHz frequency, input power with heating, 305 W, number of transducers, 2) from TECNO-GAZ was used. IR spectra were determined on a Shimadzo IR-470 spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a 500 MHz Bruker DRX-500 in $CDCl_3$ as solvent and TMS as an internal standard. Chemicals were purchased from Merck and Fluka. Elemental analyses were done on a Carlo-Erba EA1110CNNO-S analyser and agreed with the calculated values. All solvents used were dried and distilled according to standard procedures.

General Procedure for the synthesis of Fused polycyclic 4-aryl-3-methyl-4,7-dihydro-1*H*-pyrazolo[3,4-b]pyridines under ultrasound irradiation.

A solution of 5-amino-3-methyl-1*H*-pyrazole **1** (10 mmol), 2*H*-indene-1,3-dione **2** (10 mmol) and aldehyde **3** (10 mmol) (Table 2, entries 1–3, 5 mmol) in EtOH (15 mL) were placed into Pyrex-glass open vessel and irradiated in a water bath under silent condition by ultrasound (45 kHz) at 60 °C for the required reaction times (Tables 1 and 2). When the irradiation was stopped, the amount of solvent was reduced to a minimum under vacuum, to afford **4a–l** as crystalline products in 88–97% yields (Tables 1 and 2).

3.1.1. 4a: purple solid

mp 247–250 °C; IR (KBr, cm^{-1}) ν_{max} 3320, 3168, 3056, 1680, 1616, 1558, 1551, 1350; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.85 (3H, s), 5.56 (1H, s), 7.10 (1H, d, J = 6.96 Hz), 7.24–7.26 (2H, t, J = 7.71 Hz), 7.30–7.35 (2H, m), 7.47–7.50 (1H, m), 7.62 (1H, d, J = 7.16 Hz), 7.76 (1H, d, J = 7.60 Hz), 11.31 (1H, s), 12.20 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.0, 157.6, 149.5, 148.4, 140.4, 137.2, 135.5, 133.9, 132.5, 131.9, 131.1, 128.0, 124.2, 120.5, 120.1, 105.4, 103.6, 29.8, 10.2 ppm. Anal. Calc. for $C_{20}H_{14}N_4O_3$: C, 67.04; H, 3.93; N, 15.63. Found: C, 67.15; H, 3.88; N, 15.75.

3.1.2. 4b: red solid

mp 300–302 °C; IR (KBr, cm^{-1}) ν_{max} 3392, 3188, 3064, 1695, 1558, 1488, 1541, 1340; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.82 (3H, s), 5.15 (1H, s), 7.15 (1H, d, J = 6.96 Hz), 7.27–7.29 (1H, t, J = 7.23 Hz), 7.35–7.38 (1H, t, J = 7.56 Hz), 7.49–7.52 (1H, t, J = 7.92 Hz), 7.64 (1H, d, J = 7.22 Hz), 7.67 (1H, d, J = 7.76 Hz), 7.97 (1H, dd, J = 8.10, 1.44 Hz), 8.02 (1H, s), 11.32 (1H, s), 12.17 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.3, 158.1, 149.0, 148.5, 148.0, 137.6, 137.1, 135.6, 135.4, 132.0, 131.1, 130.5, 122.9, 122.0, 120.6, 120.1, 105.0, 104.0, 34.9, 10.4 ppm. Anal. Calc. for $C_{20}H_{14}N_4O_3$: C, 67.04; H, 3.94; N, 15.63. Found: C, 67.20; H, 3.80; N, 15.55.

3.1.3. 4c: red solid

mp 304–306 °C; IR (KBr, cm^{-1}) ν_{max} 3380, 3193, 3064, 1670, 1616, 1566, 1527, 1348; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.82 (3H, s), 5.10 (1H, s), 7.15 (1H, d, J = 6.94 Hz), 7.27 (1H, t, J = 7.6 Hz), 7.35 (1H, t, J = 7.5 Hz), 7.7 (2H, d, J = 8.54 Hz), 7.66 (1H, d, J = 7.13 Hz), 8.08 (2H, d, J = 8.52 Hz), 11.32 (1H, s), 12.17 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.2, 158.2, 154.4, 147.9, 146.6, 137.6, 137.2, 135.6, 131.9, 131.1, 129.8, 124.3, 120.5, 120.1, 104.9, 104.0, 35.2, 10.4 ppm. Anal. Calc. for $C_{20}H_{14}N_4O_3$: C, 67.04; H, 3.94; N, 15.63. Found: C, 67.12; H, 3.85; N, 15.70.

3.1.4. 4d: red solid

mp 240–242 °C; IR (KBr, cm^{-1}) ν_{max} 3317, 3244, 3040, 1701, 1620, 1550, 1506, 1080; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.67 (3H, s), 4.92 (1H, s), 6.99–7.04 (2H, m), 7.05–7.08 (1H, td, J = 6.74, 2.00 Hz), 7.17–7.20 (1H, t, J = 7.90 Hz), 7.36 (1H, d, J = 7.10 Hz), 7.50 (1H, d, J = 7.02 Hz), 7.74 (1H, d, J = 8.16 Hz), 7.85 (1H, s), 10.66 (1H, s), 11.42 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 192.8, 144.3, 140.4, 138.9, 137.9, 137.0, 136.9, 133.7, 132.8, 132.2, 132.2, 129.1, 131.4, 127.9, 124.2, 124.0, 123.3, 106.1, 105.6, 34.7, 12.0 ppm. Anal. Calc. for $C_{20}H_{14}BrN_3O$: C, 61.24; H, 3.59; N, 10.71. Found: C, 61.32; H, 3.69; N, 10.78.

3.1.5. 4e: orange-brown solid

mp 230–232 °C; IR (KBr, cm^{-1}) ν_{max} 3220, 3182, 3060, 1690, 1614, 1568, 1080; ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.79 (3H, s), 5.33 (1H, s), 7.25–7.64 (8H, m), 11.22 (1H, s), 12.06 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.0, 157.3, 148.0, 145.1, 137.4, 137.2, 136.6, 135.7, 132.4, 132.4, 131.1, 130.2, 128.7, 128.4, 120.4, 119.9, 105.7, 104.2, 33.2, 10.2 ppm. Anal. Calc. for

$C_{20}H_{14}ClN_3O$: C, 69.07; H, 4.05; N, 12.08. Found: C, 69.19; H, 4.18; N, 12.16.

3.1.6. 4f: orange solid

mp 235–237 °C; IR (KBr, cm^{-1}) ν_{max} 3309, 3200, 1710, 1535, 1506, 1080; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.84 (3H, s), 4.95 (1H, s), 7.00–7.88 (8H, m), 9.95 (1H, s), 11.23 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 192.9, 158.0, 153.6, 149.4, 143.8, 137.4, 137.3, 135.7, 135.0, 132.0, 131.4, 129.8, 122.8, 122.5, 120.5, 120.0, 105.4, 104.4, 35.0, 10.4 ppm. Anal. Calc. for $C_{20}H_{14}ClN_3O$: C, 69.07; H, 4.05; N, 12.08. Found: C, 69.19; H, 4.12; N, 12.14.

3.1.7. 4g: red solid

mp 342–344 °C; IR (KBr, cm^{-1}) ν_{max} 3390, 3189, 3056, 1670, 1625, 1540, 1490, 1080; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.83 (3H, s), 4.93 (1H, s), 7.15 (1H, d, J = 6.95 Hz), 7.20 (2H, d, J = 8.5 Hz), 7.23 (2H, d, J = 8.5 Hz), 7.25–7.28 (1H, t, J = 7.92 Hz), 7.33–7.36 (1H, t, J = 7.26 Hz), 7.62 (1H, d, J = 7.15 Hz), 11.20 (1H, s), 12.09 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.3, 157.9, 147.9, 145.9, 137.3, 135.7, 131.9, 131.2, 131.0, 130.3, 128.8, 120.4, 119.9, 105.7, 104.7, 34.7, 10.4 ppm. Anal. Calc. for $C_{20}H_{14}ClN_3O$: C, 69.07; H, 4.05; N, 12.08. Found: C, 69.18; H, 4.01; N, 12.01.

3.1.8. 4h: red solid

mp 302–304 °C; IR (KBr, cm^{-1}) ν_{max} 3236, 3186, 3060, 1670, 1612, 1558, 1535, 1068; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.80 (3H, s), 5.32 (1H, s), 7.13 (1H, d, J = 6.95 Hz), 7.14–7.24 (2H, m), 7.26 (1H, t, J = 7.2 Hz), 7.33 (1H, t, J = 7.2 Hz), 7.43 (1H, s), 7.64 (1H, d, J = 7.17 Hz), 11.27 (1H, s), 12.10 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.0, 158.4, 148.0, 142.3, 141.5, 137.3, 137.2, 135.7, 133.2, 132.0, 131.9, 131.0, 129.1, 128.5, 120.4, 120.0, 104.8, 104.1, 33.1, 10.2 ppm. Anal. Calc. for $C_{20}H_{13}Cl_2N_3O$: C, 62.84; H, 3.42; N, 10.99. Found: C, 62.72; H, 3.50; N, 10.83.

3.1.9. 4i: light red solid

mp > 350 °C; IR (KBr, cm^{-1}) ν_{max} 3415, 3254, 1668, 1616, 1580, 1488, 1545, 1350, 1240; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 2.07 (3H, s), 5.23 (1H, s), 6.57 (1H, d, J = 3.72 Hz), 7.24 (1H, d, J = 6.93 Hz), 7.32 (1H, t, J = 7.4 Hz), 7.38 (1H, t, J = 7.23 Hz), 7.53 (1H, d, J = 3.70 Hz), 7.65 (1H, d, J = 7.12 Hz) 11.40 (1H, s), 12.27 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.0, 162.1, 159.0, 151.5, 147.8, 138.2, 137.0, 135.6, 132.1, 131.3, 120.8, 120.3, 115.3, 110.9, 100.5, 100.4, 29.4, 10.3 ppm. Anal. Calcd. for $C_{18}H_{12}N_4O_4$: C, 62.07; H, 3.47; N, 16.09. Found: C, 62.16; H, 3.35; N, 16.01.

3.1.10. 4j: red solid

mp 262–264 °C; IR (KBr, cm^{-1}) ν_{max} 3419, 3244, 3060, 1680, 1616, 1558, 1494; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.01 (4H, t, J = 7.01 Hz), 1.75 (6H, s), 3.89 (4H, s), 5.17 (2H, s), 6.74 (2H, t, J = 7.36 Hz), 6.84 (2H, d, J = 8.14 Hz), 7.02 (2H, m), 7.09 (2H, d, J = 6.06 Hz), 7.21–7.25 (2H, m), 7.31 (2H, t, J = 7.48 Hz), 7.58 (2H, dd, J = 6.99, 2.56 Hz), 11.00 (2H, s), 11.85 (2H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.4, 158.6, 156.8, 148.0, 137.5, 136.8, 135.9, 131.6, 130.8, 130.3, 127.9, 121.2, 120.3, 119.5, 112.9, 106.0, 105.5, 68.5, 26.2, 19.3, 10.0 ppm. Anal. Calc. for $C_{44}H_{36}N_6O_4$: C, 74.14; H, 5.09; N, 11.79. Found: C, 74.01; H, 5.13; N, 11.68.

3.1.11. 4k: red solid

mp 248–250 °C; IR (KBr, cm^{-1}) ν_{max} 3392, 3197, 3064, 1680, 1616, 1558, 1490, 1250; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.34 (4H, s), 1.59 (4H, s), 1.80 (6H, s), 3.83 (4H, s), 5.21 (2H, s), 6.75 (2H, t, J = 7.36 Hz), 6.82 (2H, d, J = 8.15 Hz), 7.01 (2H, t, J = 6.92 Hz), 7.07 (2H, m), 7.12 (2H, d, J = 6.95 Hz), 7.22 (2H, t, J = 7.31 Hz), 7.31 (2H, t, J = 7.32 Hz), 7.61 (2H, m), 11.07 (2H, s), 11.90 (2H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.2, 158.5, 156.5, 148.1, 137.7, 136.7, 136.0, 135.1, 131.6, 130.7, 130.3, 127.9, 121.1, 120.2, 119.5, 112.9, 106.1, 105.7, 68.7, 26.8, 26.4, 19.4, 10.1 ppm. Anal. Calc. for $C_{46}H_{40}N_6O_4$: C, 74.58; H, 5.44; N, 11.34. Found: C, 74.67; H, 5.32; N, 11.22.

3.1.12. 4l: light orange solid

mp > 350 °C; IR (KBr, cm^{-1}) ν_{max} 3317, 3244, 3040, 1701, 1620, 1550, 1506; ^1H NMR (500 MHz, $CDCl_3$) δ_H: 1.74 (4H, s), 1.81 (6H, s), 1.92 (4H, s), 3.95 (4H, s), 4.82 (2H, s), 6.71 (4H, d, J = 8.5 Hz), 7.05 (4H, d, J = 8.5 Hz), 7.12 (4H, d, J = 6.89 Hz), 7.22 (2H, t, J = 7.05 Hz), 7.31 (2H, t, J = 7.28 Hz), 7.36 (2H, m), 11.02 (2H, s), 12.02 (2H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ_C: 190.2, 160.2, 157.6, 148.2, 139.2, 137.4, 135.8, 131.8, 131.6, 130.8, 129.4, 120.31, 119.7, 114.8, 106.5, 105.4, 67.8, 34.4, 26.4, 26.3, 10.4 ppm. Anal. Calc. for $C_{46}H_{40}N_6O_4$: C, 74.58; H, 5.44; N, 11.34. Found: C, 74.69; H, 5.35; N, 11.26.

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