An efficient one-pot synthesis of 1,3,5-substituted-1*H*-pyrazoles derivatives

Alireza Hassanabadi[a]*, Mohammad R. Hosseini-Tabatabaei[a], Mehdi Shahraki[a], Mohammad Anary-Abbasinejad[b] and Saeedeh Ghoroghchian[c]

[a]*Department of Chemistry, Islamic Azad University, Zahedan Branch, PO Box 98135-978, Zahedan, Iran*

[b]*Young Researchers Club, Islamic Azad University, Anar Branch, Anar, Iran*

[c]*Department of Chemistry, Islamic Azad University, Yazd Branch, PO Box 89195-155, Yazd, Iran*

One-pot reaction between dialkyl acetylendicarboxylates and different hydrazide acids in the presence of triphenylphosphine leds to 1,3,5-substituted-1*H*-pyrazoles derivatives in excellent yields. In the absence of triphenylphosphine, dialkyl acetylenedicarboxylate adds to hydrazide acids to produce dialkyl 2-(arylhydrazono)succinates were obtained in good yields.

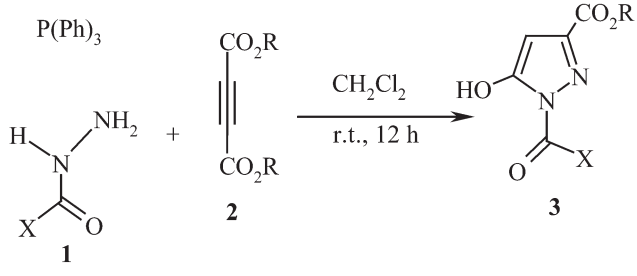
Keywords: substituted pyrazoles, acetylenic esters, hydrazide acids, triphenylphosphine

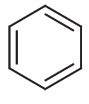
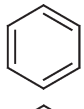
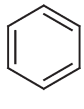
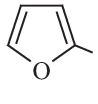
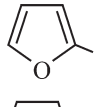
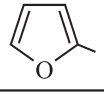
Heterocycles are popularly known for displaying a wide range of biological properties.[1] The recent success of pyrazole based COX-II inhibitors and their application in medicinal chemistry have amplified the importance of pyrazoles to even a greater extent.[2] Several pharmaceutical drugs including celecoxib[2] and rimonabant[3] utilise the pyrazole as their core molecular entity,[4,5] Pyrazoles are often synthesised by the 1,3-dipolar cycloaddition reaction of nitrilimines with alkynes,[6] alkyne surrogates,[7] or alkenes,[5,6] and other methods.[4] Special attention is warranted toward the synthetic design and development of pyrazoles because of their high demand in academic and pharmaceutical sectors. Three-component reaction between triphenylphosphine, acetylenic esters and an organic acidic compounds has been reported to produce phosphorus ylides which may further undergo intramolecular Wittig reaction to produce unsaturated hetro-or carbocyclic compounds in a one-pot process.[8–11] Similar reactions have been developed for the synthesis of a variety of carbocycles and heterocycles, using N–H, O–H, S–H and C–H acidic compounds in order to trap the zwitterionic intermediate produced by the addition of triphenylphosphine to dialkyl acetylenedicarboxylates (DAAD). In continuation of our previous works on the reaction of PPh_3–DAAD zwitterion with organic acidic compounds, [12–17] we now report the results of our study on the reaction between DAADs and triphenylphosphine in the presence of hydrazides in order to trap the PPh_3–DAAD zwitterion.

Results and discussion

Treatment of diethyl acetylenedicarboxylate (DEAD) with triphenylphosphine and benzohydrazide in dichloromethane at room temperature for 12 h, after column chromatography afforded ethyl 1-benzoyl-5-hydroxy-1H-pyrazole-3-carboxylate (**3a**) in 85% yield (Scheme 1).

The mass spectrum of compound **3a** showed the molecular ione peak and the base peak at 260 and 105 (Ph–C≡O$^+$), respectively. The peaks at *m/z* = 147 and 77 were attributed respectively to the fragments Ph–CO–N$^+$–N=CH_2 and Ph^+. The ^{1}H NMR spectrum of compound **3a** exhibited a D_2O-exchangeable broad signal at 11.22 ppm for OH proton. Ethyl protons were observed as a triplet ($^3J_{HH}$ = 7 H_Z) at 1.29 ppm and a quartet at 4.26 ppm. Two triplets and a doublet were observed respectively at 7.41, 7.54 and 7.94 ppm for aromatic protons. The ^{13}C NMR spectrum of compound **3a** showed eleven signals in agreement with the proposed structure. The IR spectrum of compound **3a** also supported the suggested structure, strong absorption bands were observed at 3180, 1730 and 1662 cm^{-1} respectively for the OH, ester and amide carbonyl groups. To explore the scope of the reaction, benzohydrazide and furan-2-carbohydrazide acid were reacted with acetylene diesters DEAD, dimethyl acetylenedicarboxylate (DMAD) and di-tert-butyl acetylenedicarboxylate (DTAD) in the presence of triphenylphosphine and corresponding pyrazole derivatives **3a–f** were obtained in good yields. In all the above reactions triphenylphosphine oxide was also isolated as the by-product. To prove the key role of PPh_3 in the above reaction, DMAD was treated with benzohydrazide in the absence of PPh_3. After 3 h stirring at room temperature the



3	X	R	* %Yield
a		C_2H_5	85
b		CH_3	88
c		*t*-Bu	90
d		CH_3	82
e		C_2H_5	80
f		*t*-Bu	84

*Isolated yields

Scheme 1 Synthesis of pyrazoles by reaction between hydrazide acids and DAADs in the presence of triphenylphosphine.

* Correspondent. E-mail: ar_hasanabadi@yahoo.com

reaction was completed and dimethyl 2-(benzoylhydrazono) succinate **4a** was isolated in nearly quantitative yield. The ^{1}H NMR spectrum of compound **4a** showed two single signals at 3.61 and 3.73 ppm for two methoxy carbonyl groups supported by the strong absorption bands at 1727 and 1671 cm^{-1} in the IR spectrum. The two protons of the methylene group was absorbed a single signal at 3.87 ppm. ^{13}C NMR spectrum of compound **4a** exhibited three signals at aliphatic region (33.3, 53.0 and 53.3), which are due to two methyl and a methylene groups. The above reaction was also carried out between furan-2-carbohydrazide acid and acetylene diesters DMAD, DEAD and DTAD and the related hydrazone derivative **4b–e** were obtained in good yields (Scheme 2).

A reasonable mechanism for the formation of compounds **3** is presented at Scheme 3. It is well known that the reaction between triphenylphosphine and activated acetylenes in the presence of NH-acidic compounds afforded phosphorus ylides. Similar ylides **5** may be produced from three component reaction of DAAD, PPh_3 and hydrazide acid, which then cyclises to intermediate **6**. Hydrolysis of ylide **6** which is accompanied with elimination of triphenylphosphinoxide follows by the air oxidation and tautomerisation gives pyrazole **3**. In the absence of triphenylphosphline benzohydrazide acid to DMAD to produce compounds **4a.** Compounds **4a–e** are stable derivatives and do not cyclise to pyrazole **3** even in refluxing acetonitrile. This may be attributed to the *Z* geometry of N=C double bond which is stabilised by intramolecular hydrogen bond. This geometry has not the suitable orientation for the NH group to attack the ester carbonyl group to afford the five membered ring of pyrazole derivative.

In summary 1,3,5-substituted-1*H*-pyrazole derivatives may be prepared by a simple, one-pot reaction between dialkyl acetylenedicarboxylates, hydrazides and triphenylphosphine in good yields. In the absence of triphenylphosphine hydrazides add to acetylenic esters to produce dialkyl 2-(arylhydraz ono)succinates in excellent yields. The present method carries the advantage that the reaction is performed under neutral conditions and starting materials can be mixed without any activation or modification.

Experimental

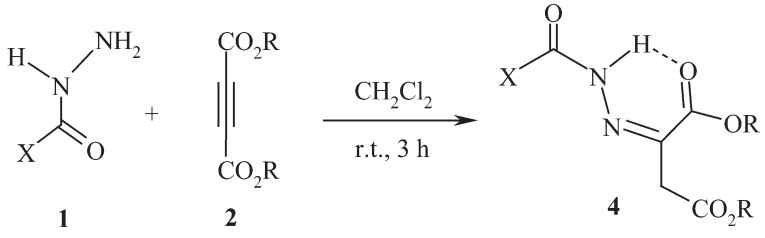
Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed at the analytical laboratory of Science and Researches Unite of Islamic Azad University. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer.^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-500 Avance spectrometer at solution in $CDCl_3$ using TMS as internal standard. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

Preparation of compounds **3a–f***; general procedure*

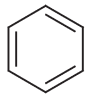
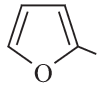
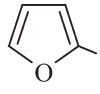
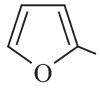
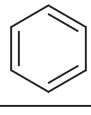
To a magnetically stirred solution of hydrazides (2 mmol) and triphenylphosphine (2 mmol) in 10 mL dichloromethane was added dropwise a mixture of dialkyl acetylenedicarboxylate (2 mmol) in 5 mL dichloromethane at room temperature. The reaction mixture was then stirred for 12 h. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography using hexane-ethyl acetate as eluent. The solvent was removed under reduced pressure to afford the product.

Preparation of compounds **4a–e***; general procedure*

To a magnetically stirred solution of hydrazides (2 mmol) in 20 mL dichloromethane was added dropwise a mixture of dialkyl acetylenedicarboxylate (2 mmol) in 5 mL dichloromethane at room temperature. The reaction mixture was then allowed to stir for 12 h. The solvent was evaporated at reduced pressure and the residue was

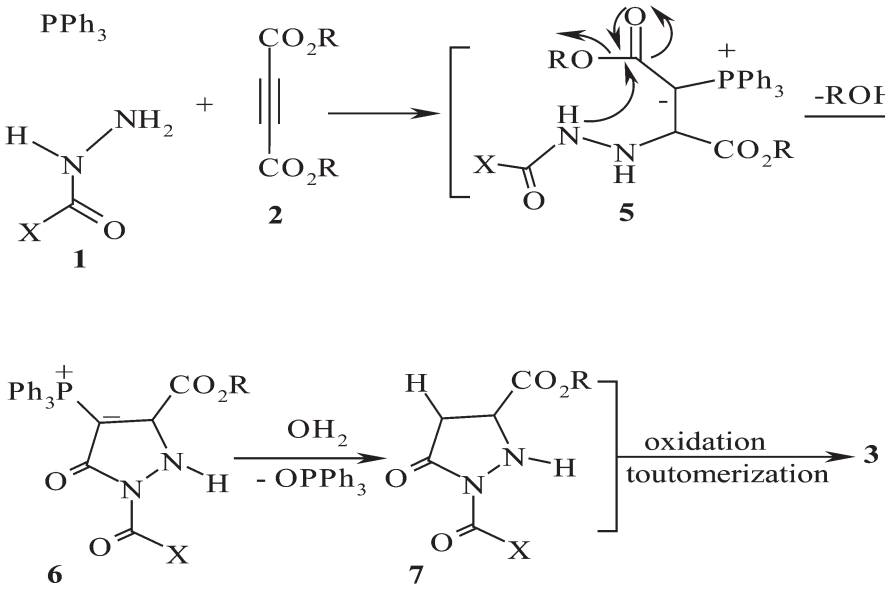


1 **2** **4**

4	X	R	* %Yield
a		CH_3	95
b		CH_3	90
c		C_2H_5	91
d		*t*-Bu	88
e		*t*-Bu	90

* Isolated yields

Scheme 2 Synthesis of dialkyl 2-(arylhydrazono)succinates by reaction between hydrazides and DAADs.



Scheme 3 Suggested mechanism for formation of compound **3**.

purified by silica gel column chromatography using hexane-ethyl acetate (1:2) as eluent. The solvent was removed under reduced pressure to afford the product.

Ethyl 1-*benzoyl-5-hydroxy*-1*H-pyrazole-3-carboxylate* (**3a**): Yield: 85%; pale white powder, m.p. 130–132 °C, IR (KBr) (ν_{max}/cm^{-1}): 3180 (OH), 1730 and 1662 (C=O). MS, *m/z* (%): 260 (M^{+}, 10). Anal.Calcd for $C_{13}H_{12}N_2O_4$: C, 60.00; H, 4.65; N, 10.76. Found: C, 59.9; H, 4.8; N, 10.9%. ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si): δ 1.29 (3 H, d $^3J_{HH}$ = 7 H_Z, CH_3), 4.27 (2 H, d $^3J_{HH}$ = 7 H_Z, OCH_2), 7.41–7.98 (5 H, m, 5 CH aromatic), 7.93 (1 H, s, CH), 11.23 (1 H, broad s, OH). ^{13}C NMR (125.8 MHz, $CDCl_3$, Me_4Si): δ 14.4 (CH3), 62.3 (CH2), 101.7 (C-4), 140.1 (C-3), 133.3 (C-5), 128.2, 129.0, 129.4 and 132.3 (aromatic), 162.9 (CO).

Methyl 1-*benzoyl-5-hydroxy*-1*H-pyrazole-3-carboxylate* (**3b**): Yield: 88%; pale white powder, m.p. 184–186 °C**,** IR (KBr) (ν_{max}/cm^{-1}): 3185 (OH), 1746 and 1663 (C=O). MS, *m/z* (%): 246 (M^{+}, 12). Anal. Calcd for $C_{12}H_{10}N_2O_4$: C, 58.54; H, 4.09; N, 11.38. Found: C, 58.6; H, 4.0; N, 11.2%. ^{1}H NMR (250.1 MHz, d_6-DMSO): δ 3.84 (3 H, OCH_3), 7.44–7.95 (5 H, m, 5 CH aromatic), 7.87 (1 H, s, CH), 12.14 (1 H, broad s, OH). ^{13}C NMR (62.89 MHz, d_6-DMSO): δ 56.5 (OCH_3), 102.2 (C-4), 140.5 (C-3), 133.0 (C-5), 128.4, 130.1, 130.5 and 132.4 (aromatic), 162.7 (CO).

Tert-butyl 1-*benzoyl-5-hydroxy*-1*H-pyrazole-3-carboxylate* **(3c):** Yield: 90%; pale white powder, m.p. 165–167 °C, IR (KBr) (ν_{max}/cm^{-1}): 3230 (OH), 1710 and 1665 (C=O). MS, *m/z* (%): 288 (M^{+}, 6). Anal. Calcd for $C_{15}H_{16}N_2O_4$: C, 62.49; H, 5.59; N, 9.72. Found: C, 62.6; H, 5.4; N, 9.9%. ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.49 (9 H, 3 CH_3), 7.52–7.90 (5 H, m, 5 CH aromatic), 7.74 (1 H, s, CH), 12.18 (1 H, broad s, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 28.5 (3 CH_3), 82.4 (C), 102.3 (C-4), 140.0 (C-3), 133.5 (C-5), 128.8, 129.4, 133.2 and 135.0 (aromatic), 164.5 (CO).

Methyl 1-*(furan-2-carbonyl)-5-hydroxy*-1*H-pyrazole-3-carboxylate* (**3d**): Yield: 82%; pale white powder, m.p. 180–182 °C**,** IR (KBr) (ν_{max}/cm^{-1}): 3210 (OH), 1739 and 1668 (C=O). MS, *m/z* (%): 236 (M^{+}, 9). Anal. Calcd for $C_{10}H_8N_2O_5$: C, 50.85; H, 3.41; N, 11.86. Found: C, 50.7; H, 3.5; N, 11.7%. ^{1}H NMR (250.1 MHz, d_6-DMSO): δ 3.85 (3 H, OCH_3), 6.59 (1H, m, CH furan), 7.57 (1H, s, CH furan), 7.65 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 7.83 (1 H, s, CH pyrazole), 12.07 (1 H, broad s, OH). ^{13}C NMR (62.89 MHz, d_6-DMSO): δ 55.9 (OCH_3), 116.0 (C-4), 149.3 (C-3), 137.0 (C-5), 111.2, 120.1,140.0 and 142.8 (furan), 167.6 (CO).

Ethyl 1-*(furan-2-carbonyl)-5-hydroxy*-1*H-pyrazole-3-carboxylate* (**3e**): Yield: 80%; Pale white powder, m.p. 143–145 °C, IR (KBr) (ν_{max}/cm^{-1}): 3203 (OH), 1730 and 1668 (C=O). MS, *m/z* (%): 250 (M^{+}, 7). Anal.Calcd for $C_{11}H_{10}N_2O_5$: C, 52.80; H, 4.03; N, 11.20. Found: C, 52.9; H, 4.1; N, 11.1%. ^{1}H NMR (500 MHz, $CDCl_3$, Me_4Si): δ 1.28 (3 H, d $^3J_{HH}$ = 7 H_Z, CH_3), 4.37 (2 H, d $^3J_{HH}$ = 7 H_Z, OCH_2), 6.60 (1H, m, CH furan), 7.58 (1H, s, CH furan), 7.65 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 7.86 (1 H, s, CH pyrazole), 13.43 (1 H, broad s, OH). ^{13}C NMR (125.8 MHz, $CDCl_3$, Me_4Si): δ 14.3 (CH3), 62.7 (CH2), 112.9 (C-4), 145.8 (C-3), 137.3 (C-5), 111.5, 120.3,140.2 and 142.6 (furan), 169.9 (CO).

*Tert-butyl*1-*(furan-2-carbonyl)-5-hydroxy*-1*H-pyrazole-3-carboxyl-ate* (**3f**): Yield: 84%; pale white powder, m.p. 158–160 °C, IR (KBr) (ν_{max}/cm^{-1}): 3220 (OH), 1710 and 1669 (C=O). MS, *m/z* (%): 278 (M^{+}, 7). Anal. Calcd for $C_{13}H_{14}N_2O_5$: C, 56.11; H, 5.07; N, 10.07. Found: C, 56.2; H, 4.9; N, 10.2%. ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.49 (9 H, 3 CH_3), 6.72 (1H, m, CH furan), 7.41 (1H, s, CH furan), 7.67 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 7.98 (1 H, s, CH pyrazole), 12.19 (1 H, broad s, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 28.6 (3 CH_3), 82.4 (C), 113.2 (C-4), 147.6 (C-3), 135.0 (C-5), 110.9, 120.3,140.3 and 142.5 (furan), 162.9 (CO).

Dimethyl 2-(benzoylhydrazono)succinate (**4a**): Yield: 95%;white powder, m.p. 141–143 °C**,** IR (KBr) (ν_{max}/cm^{-1}): 3185 (NH), 1727 and 1671 (C=O). MS, *m/z* (%): 278 (M^{+}, 6). Anal. Calcd for $C_{13}H_{14}N_2O_5$: C, 56.11; H, 5.07; N, 10.07. Found: C, 56.3; H, 4.9; N, 10.2%. ^{1}H NMR (500.1 MHz, d_6-DMSO): δ 3.61 and 3.73 (6H, 2OCH_3), 3.87 (2H, s, CH_2), 7.47–7.81 (5 H, m, 5 CH aromatic), 11.38 (1 H, broad s, NH).^{13}C NMR (125.8 MHz, d_6-DMSO): δ 33.3 (CH_2), 53.0 and 53.3 (2OCH_3), 129.1, 129.6, 133.0 and 133.9 (aromatic), 154.2 (C=N), 165.4, 169.2 and 169.4 (3CO).

Dimethyl 2-[(furan-2-carbonyl)hydrazono]succinate **(4b):** Yield: 90%; white powder, m.p. 133–135 °C**,** IR (KBr) (ν_{max}/cm^{-1}): 3275 (NH), 1735 and 1661 (C=O). MS, *m/z* (%): 268 (M^{+}, 5). Anal. Calcd for $C_{11}H_{12}N_2O_6$: C, 49,26; H, 4.51; N, 10.44. Found: C, 49.1; H, 4.4; N, 10.6%. ^{1}H NMR (500.1 MHz, d_6-DMSO): δ 3.61 and 3.76 (6H, 2OCH_3), 3.81 (2H, s, CH_2), 6.69 (1H, m, CH furan), 7.63 (1H, s, CH furan), 7.96 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 11.45 (1 H, broad s, NH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 32.8 (CH_2), 53.0 and 53.5 (2OCH_3), 113.1, 120.2,145.8 and 147.9 (furan), 154.3 (C=N), 165.2, 168.9 and 169.1 (3CO).

Diethyl 2-[(furan-2-carbonyl)-hydrazono]-succinate **(4c):** Yield: 91%; white powder, m.p. 143–145 °C, IR (KBr) (ν_{max}/cm^{-1}): 3214 (NH), 1728 and 1665 (C=O). MS, *m/z* (%): 296 (M^{+}, 9). Anal.Calcd for $C_{13}H_{16}N_2O_6$: C, 52.70; H, 5.44; N, 9.46. Found: C, 52.8; H, 5.6; N, 9.3%. ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.68 (3 H, d $^3J_{HH}$ = 7 H_Z, CH_3), 1.34 (3 H, d $^3J_{HH}$ = 7 H_Z, CH_3), 3.78 (2H, s, CH_2), 4.17–4.32 (4 H, m, 2OCH_2), 6.63 (1H, m, CH furan), 7.44 (1H, s, CH furan), 7.52 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 13.2 (1 H, broad s, NH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 13.9 and 14.4 (2CH3), 32.6 (CH_2), 61.9 and 62.1 (2OCH2), 113.3, 119.9,145.3 and 147.5 (furan), 154.1 (C=N), 165.6, 169.5 and 169.9 (3CO).

Di t-butyl 2-[(furan-2-carbonyl)-hydrazono]-succinate **(4d):** Yield: 88%;white powder, m.p. 158–160 °C, IR (KBr) (ν_{max}/cm^{-1}): 3223 (NH), 1714 and 1658 (C=O). MS, *m/z* (%): 352 (M^{+}, 6). Anal. Calcd for $C_{17}H_{24}N_2O_6$: C, 57.94; H, 6.86; N, 7.95. Found: C, 57.8; H, 6.7; N,

8.1%. ^{1}H NMR (500 MHz, d_6-DMSO): δ 0.85 (9 H, s, *t*-Bu), 1.47 (9 H, s, *t*-Bu), 3.89 (2H, s, CH_2), 6.61(1H, m, CH furan), 7.38 (1H, s, CH furan), 7.52 (1H, d, $^3J_{HH}$ = 2 H_Z, CH furan), 12.02 (1 H, broad s, NH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 28.6 and 28.8 (6 CH_3 of 2 *t*-Bu), 32.1 (CH_2), 78.4 and 82.7 (2C), 113.7, 119.8,145.0 and 146.9 (furan), 154.6 (C=N), 165.2, 169.0 and 169.6 (3CO).

Di t-butyl 2-(benzoylhydrazono)-succinate **(4e):** Yield: 90%; white powder, m.p. 141–143 °C**,** IR (KBr) (ν_{max}/cm^{-1}): 3260 (NH), 1726 and 1688 (C=O). MS, *m/z* (%): 362 (M^+, 9). Anal. Calcd for $C_{19}H_{26}N_2O_5$: C, 62.97; H, 7.23; N, 7.73. Found: C, 62.8; H, 7.4; N, 7.8%. ^{1}H NMR (500.1 MHz, d_6-DMSO): δ 1.45 (9 H, s, *t*-Bu), 1.54 (9 H, s, *t*-Bu), 3.55 (2H, s, CH_2), 7.44–7.93 (5 H, m, 5 CH aromatic), 13.54 (1 H, broad s, NH).^{13}C NMR (125.8 MHz, d_6-DMSO): δ 28.6 and 28.8 (6 CH_3 of 2 *t*-Bu), 32.3 (CH_2), 79.3 and 82.1 (2 C), 129.3, 129.7, 132.9 and 133.7 (aromatic), 154.5 (C=N), 165.6, 169.4 and 169.9 (3CO).

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