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Synthesis and Anticancer Activity of a Novel Series of Tetrazolo[1,5-*a*]quinoline Based 1,2,3-Triazole Derivatives

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Abstract—We report here the synthesis of a novel series of tetrazolo[1,5-*a*]quinoline based 1,2,3-triazoles from acetaniline via the Vilsmeier–Haack reaction, the Claisen–Schmidt condensation and 1,3-dipolar Click reaction. The newly synthesized compounds have been tested for their anticancer activity against a panel of three cell lines such as cervix (SiHa), brest (MDA-MB-231) and pancreatic carcinoma (PANC-1). Most of the synthesized compounds possess potent anticancer activity.

Keywords: tetrazole, 1,2,3-triazole, quinoline, chalcone, anticancer activity

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INTRODUCTION

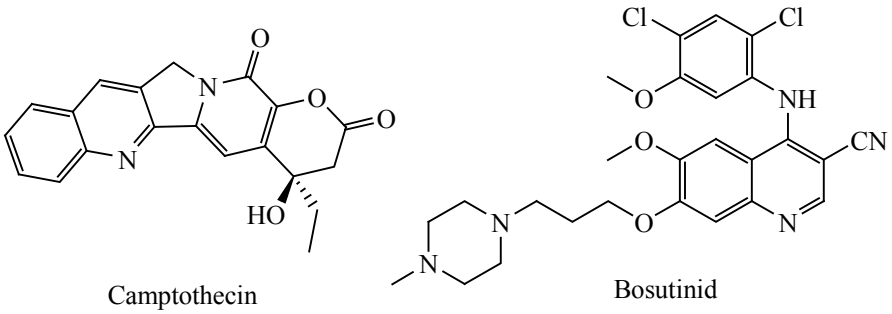
Heterocycles are the core components of many marketed anticancer drugs [1, 2]. Synthetic quinoline derivatives such as Camptothecin [3] and Bosutinid [4] are the most important quinolone containing drugs used in treatment of cancer (Fig. 1).

1,2,3-Triazole and tetrazole derivatives [5, 6] and chalcones [7, 8] are also well known for their anticancer activity. Based on the above, we have developed our ongoing research [9–12] in synthesis of a novel series of (*E*)-1-{2/3/4-[(1-aryl-1*H*-1,2,3-triazol-4-yl)methoxy]-phenyl}-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one derivatives **7a**–**7l** (Schemes 1, 2) and evaluated their anticancer activity.

RESULTS AND DISCUSSION

Synthesis of the title compounds started with the Vilsmeier–Haack reaction of acetanilide **1** with DMF and $POCl_3$ that gave 2-chloroquinoline-3-carbaldehyde (**2**), the following treatment of which with sodium azide in the presence of *p*-toluenesulphonic acid gave tetrazole derivative **3**. The Claisen–Schmidt condensation of tetrazole aldehyde **3** with a propargylated acetophenones **4a**–**4c** in the presence of KOH afforded the corresponding chalcones **5a**–**5c**. The target 1,2,3-triazole derivatives were prepared by the Huisgen 1,3-dipolar cycloaddition of propargylated chalcones **5a**–**5c** with aromatic azides **6a**–**6d** in the presence of copper sulphate pentahydrate and sodium ascorbate in DMF:water medium with high yields.

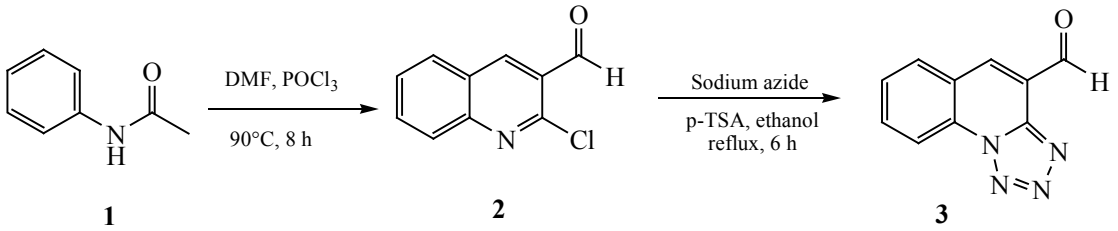
Anticancer activity. Tetrazolo[1,5-*a*]quinoline based triazoles were evaluated for their anticancer activity against three human tumor cell lines: cervix (SiHa), brest (MDA-MB-231) and pancreatic carcinoma (PANC-1) using the Sulforhodamine B assay method (Table 1). Doxorubicin was used as a standard. The compounds **7d**,



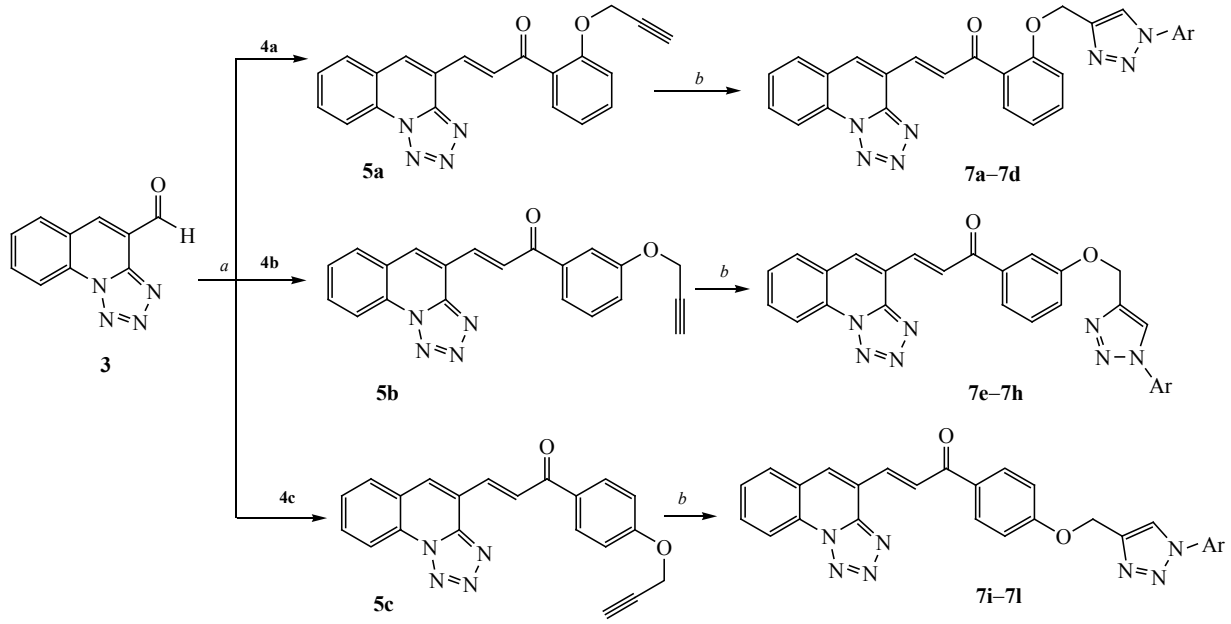
Camptothecin Bosutinid

Fig. 1. Quinoline based anticancer drugs.

Scheme 1. Synthesis of tetrazolo[1,5-*a*]quinoline-4-carbaldehyde (**3**).



Scheme 2. Synthesis of (*E*)-1-{2/3/4-[(1-aryl-1*H*-1,2,3-triazol-4-yl)methoxy]phenyl}-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-ones (**7a**–**7l**).



Reaction conditions: *a*: 2,3,4-propagylated acetophenone (**4a**–**4c**), KOH, methanol, room temperature, 3–4 h; *b*: aryl azides (**6a**–**6d**), $CuSO_4 \cdot 5H_2O$, NaASc, DMF : water.
1-[2-(prop-2-yn-1-yloxy)phenyl]ethanone (**4a**), 1-[3-(prop-2-yn-1-yloxy)phenyl]ethanone (**4b**), 1-[4-(prop-2-yn-1-yloxy)phenyl]ethanone (**4c**); Ar = azidobenzene (**6a**), 1-azido-4-chlorobenzene (**6b**), 1-azido-4-nitrobenzene (**6c**), 1-azido-4-nitrobenzene (**6d**).

7g, and **7h** demonstrated potent activity against Cervix (SiHa) cell lines. The products **7b**, **7d**, and **7f** exhibited high activity against MDA-MB-231 cell lines. The compounds **7c**, **7g**, **7Ij**, and **7k** demonstrated maximum activity on pancreatic carcinoma (PANC-1).

EXPERIMENTAL

Progress of the reactions was monitored by TLC (Silica gel, aluminium sheets 60 F254, Merck). IR (KBr) spectra were recorded on a Perkin-Elmer FT-IR-8400s. ^{1}H NMR spectra were measured on a Bruker Avance II 400 spectrometer using $CDCl_3$ as a solvent and TMS as an internal standard. Mass spectra were measured on a SHIMADZU LCMS 2020 mass spectrometer.

Synthesis of (*E*)-1-[2/3/4-(prop-2-yn-1-yloxy)-phenyl]-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (5a–5c). A mixture of tetrazolo[1,5-*a*]quinoline-4-carbaldehyde (**3**) (1 mmol) with a propargylated acetophenone **4a**–**4c** (1 mmol) and KOH in dry acetone was refluxed for 3–4 h. The reaction mixture was poured into ice cold water. The solid precipitated, and it was filtered off, washed with water, dried, and purified by column chromatography using n-hexane:ethyl acetate (9 : 1) to afford the corresponding pure product **5a**–**5c**.

(*E*)-1-[2-(Prop-2-yn-1-yloxy)phenyl]-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (5a). Yield 88%. IR spectrum, ν, cm^{-1}: 2112 (acetylene C–H), 1658 (C=O), 1572 (C=N). ^{1}H NMR spectrum, δ, ppm:

Table 1. Anticancer activity of tetrazolo[1,5-*a*]quinoline based triazoles (GI_{50} values)

Compound	GI_{50}, two dimension			Compound	GI_{50}, two dimension		
	SiHa	MDA-MB-231	PANC-1		SiHa	MDA-MB-231	PANC-1
7a	0.51	0.70	1.39	**7h**	1.88	0.44	1.78
7b	1.22	0.84	2.03	**7i**	1.06	0.38	1.16
7c	1.56	0.55	2.29	**7j**	1.19	0.76	2.31
7d	1.74	1.01	1.99	**7k**	1.23	0.69	2.67
7e	0.98	0.67	1.56	**7l**	0.96	0.58	2.06
7f	1.39	0.91	2.13	Doxorubicin	2.31	1.15	3.10
7g	2.01	0.51	2.55				

3.56–3.57 t (1H, acetylene), 4.98 d (2H, CH_2), 7.15–7.19 t (1H, Ar-H), 7.32–7.34 t (1H, Ar-H), 7.59–7.65 m (2H, Ar-H), 7.78–7.87 m (2H, Ar-H), 8.01–8.05 t (1H, Ar-H), 8.22–8.24 d (1H, Ar-H), 8.42–8.46 d (1H, Ar-H), 8.64–8.66 d (1H, Ar-H), 8.70 s (1H, quinoline-H). MS: 355 $[M + H]^+$.

(*E*)-1-[3-(Prop-2-yn-1-yloxy)phenyl]-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (5b). Yield 84%. IR spectrum, ν, cm^{-1}: 2108 (acetylene C–H), 1657 (C=O), 1569 (C=N). ^1H NMR spectrum, δ, ppm: 3.64–3.65 t (1H, acetylene), 4.99 d (2H, CH_2), 7.36–7.38 t (1H, Ar-H), 7.60–7.66 m (2H, Ar-H), 7.77–7.79 d (1H, Ar-H), 7.85–7.89 t (1H, Ar-H), 8.03–8.08 d (2H, Ar-H), 8.24–8.26 d (1H, Ar-H), 8.66–8.68 d (1H, Ar-H), 8.75–8.79 d (1H, Ar-H), 8.84 s (1H, quinoline-H). MS: 355 $[M + H]^+$.

(*E*)-1-[4-(Prop-2-yn-1-yloxy)phenyl]-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (5c). Yield 90%, mp 82–85°C. IR spectrum, ν, cm^{-1}: 2122 (acetylene C–H), 1660 (C=O), 1588 (C=N). ^1H NMR spectrum, δ, ppm: 3.67–3.68 t (1H, acetylene), 4.97 d (2H, CH_2), 7.22–7.25 d (2H, Ar-H), 7.85–7.89 t (1H, Ar-H), 8.01–8.06 m (2H, Ar-H), 8.13–8.15 t (1H, Ar-H), 8.23–8.25 d (1H, Ar-H), 8.65–8.67 d (1H, Ar-H), 8.78–8.82 m (3H, Ar-H & quinoline-H). MS: 355 $[M + H]^+$.

Synthesis of 1,2,3-triazoles (7a–7l). A mixture of a (*E*)-1-(2/3/4-(prop-2-yn-1-yloxy)phenyl)-3-(tetrazolo-[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (**5a**–**5c**) (1 mmol) with aryl an azide **6a**–**6d** (1.1 mmol) and catalytic amount of copper iodide in DMF:water (3 mL) was stirred for 8–10 h at room temperature. After completion of the reaction, the organic compound was extracted with ethyl acetate (2×20 mL), and the combined organic extracts were washed with water, brine solution and dried over sodium sulphate. The solvent was evaporated under reduced pressure, and the product was purified with column chromatography using *n*-hexane : ethyl acetate (7 : 3) to afford the corresponding pure 1,2,3-triazole derivatives **7a**–**7l**.

(*E*)-1-{2-[(1-Phenyl-1*H*-1,2,3-triazol-4-yl)methoxy]-phenyl}-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (7a). Yield 84%. IR spectrum, ν, cm^{-1}: 1645 (C=O), 1590 (C=N). ^1H NMR spectrum, δ, ppm: 5.44 s (2H, CH_2), 7.15–7.18 t (1H, Ar-H), 7.28–7.29 d (2H, Ar-H), 7.48–7.59 m (3H, Ar-H), 7.62–7.83 m (4H, Ar-H), 7.98–8.01 t (1H, Ar-H), 8.14–8.15 d (1H, Ar-H), 8.40–8.44 d (1H, Ar-H), 8.51–8.53 d (1H, Ar-H), 8.60–8.71 m (3H, Ar-H). MS: 474 $[M + H]^+$.

(*E*)-1-(2-{[1-(4-Chlorophenyl)-1*H*-1,2,3-triazol-4-yl]methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7b). Yield 84%. IR spectrum, ν, cm^{-1}: 1644 (C=O), 1588 (C=N). Yield 83%. IR spectrum, ν, cm^{-1}: 1649 (C=O), 1590 (C=N). ^1H NMR spectrum, δ, ppm: 5.43 s (2H, CH_2), 7.15–7.18 t (1H, Ar-H), 7.17–7.18 d (2H, Ar-H), 7.47–7.51 m (3H, Ar-H), 7.63–8.72 m (3H, ArH), 7.80–7.83 t (1H, Ar-H), 7.98–8.02 t (1H, Ar-H), 8.11–8.13 d (1H, Ar-H), 8.36–8.40 d (1H, Ar-H), 8.47–8.49 d (1H, ArH), 8.56 s (1H, Ar-H), 8.67 s (1H, Ar-H). MS: 508 $[M + H]^+$.

(*E*)-1-(2-{[1-(4-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (7c). Yield 83%. IR spectrum, ν, cm^{-1}: 1655 (C=O), 1590 (C=N). ^1H NMR spectrum, δ, ppm: 5.44 s (2H, CH_2), 7.16–7.20 t (1H, Ar-H), 7.34–7.42 m (2H, Ar-H), 7.49–7.53 d (1H, Ar-H), 7.65–7.78 m (2H, Ar-H), 7.87–7.91 m (2H, Ar-H), 8.02–8.10 m (3H, Ar-H),

8.25–8.30 m (2H, Ar-H), 8.37–8.41 m (1H, Ar-H), 8.54 s (1H, Ar-H), 8.82 s (1H, Ar-H). MS: 519 $[M + H]^+$.

(*E*)-1-(2-{[1-(2-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7d). Yield 80%. IR spectrum, ν, cm^{-1}: 1651 (C=O), 1593 (C=N). ^1H NMR spectrum, δ, ppm: 5.43 s (2H, CH_2), 7.28–7.34 m (4H, Ar-H), 7.47–7.62 m (3H, Ar-H), 7.65–7.81 m (2H, Ar-H), 7.88–7.92 d (1H, Ar-H), 8.00–8.11 m (2H, Ar-H), 8.32–8.36 m (2H, Ar-H), 8.56 s (1H, Ar-H), 8.82 s (1H, Ar-H). MS: 519 $[M + H]^+$.

(*E*)-1-{3-[(1-Phenyl-1*H*-1,2,3-triazol-4-yl)methoxy]-phenyl}-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (7e). Yield 82%. IR spectrum, ν, cm^{-1}: 1650 (C=O), 1590 (C=N). ^1H NMR spectrum, δ, ppm: 5.41 s (2H, CH_2), 7.21–7.25 t (1H, Ar-H), 7.32–7.44 m (3H, Ar-H), 7.47–7.51 m (2H, Ar-H), 7.55–7.60 m (3H, Ar-H), 7.64–7.79 m (2H, Ar-H), 7.80–7.84 t (1H, Ar-H), 7.89–8.00 m (2H, Ar-H), 8.09–8.15 m (3H, Ar-H), 8.22–8.26 m (3H, Ar-H), 8.64–8.66 d (1H, Ar-H), 8.70–8.74 d (1H, Ar-H), 8.84s (1H, Ar-H), 9.12 s (1H, Ar-H). MS: 474 $[M + H]^+$.

(*E*)-1-(3-{[1-(4-Chlorophenyl)-1*H*-1,2,3-triazol-4-yl]methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7f). Yield 83%. IR spectrum, ν, cm^{-1}: 1648 (C=O), 1587 (C=N). ^1H NMR spectrum, δ, ppm: 5.41 s (2H, CH_2), 7.47–7.49 d (1H, Ar-H), 7.57–7.65 m (3H, Ar-H), 7.74–7.78 t (2H, Ar-H), 7.89–7.90 t (1H, ArH), 7.94–7.96 d (1H, Ar-H), 8.04–8.09 m (3H, Ar-H), 8.24–8.26 d (1H, Ar-H), 8.67–8.69 d (1H, Ar-H), 8.76–8.80 d (1H, Ar-H), 8.85 s (1H, Ar-H), 9.09 s (1H, Ar-H). MS: 508 $[M + H]^+$.

(*E*)-1-(3-{[1-(4-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7g). Yield 85%. IR spectrum, ν, cm^{-1}: 1592 (C=N), 1653 (C=O). ^1H NMR spectrum, δ, ppm: 5.43 s (2H, CH_2), 7.22–7.27 t (1H, Ar-H), 7.34–7.38 m (2H, Ar-H), 7.47–7.52 m (2H, Ar-H), 7.60–7.66 m (2H, Ar-H), 7.74–7.80 m (2H, Ar-H), 7.86–7.92 t (1H, Ar-H), 8.04–8.11 m (2H, Ar-H), 8.23–8.31 m (3H, Ar-H), 8.42–8.52 m (3H, Ar-H), 8.66–8.70 m (2H, Ar-H), 8.85 s (1H, Ar-H), 9.23 s (1H, Ar-H). MS: 519 $[M + H]^+$.

(*E*)-1-(3-{[1-(2-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7h). Yield 79%. IR spectrum, ν, cm^{-1}: 1645 (C=O), 1592 (C=N). ^1H NMR spectrum, δ, ppm: 5.41 s (2H, CH_2), 7.47–7.50 d.d (1H, Ar-H), 7.61–7.65 t (1H, Ar-H), 7.68–7.70 m (2H, Ar-H), 7.84–8.09 m (6H, Ar-H), 8.23–8.25 d.d (2H, Ar-H), 8.65–8.67 d (1H, Ar-H), 8.75–8.79 d (1H, Ar-H), 8.84 s (1H, Ar-H), 8.91 s (1H, Ar-H). MS: 519 $[M + H]^+$.

(*E*)-1-{3-[(1-Phenyl-1*H*-1,2,3-triazol-4-yl)methoxy]-phenyl}-3-(tetrazolo[1,5-*a*]quinolin-4-yl)prop-2-en-1-one (7i). Yield 84%. IR spectrum, ν, cm^{-1}: 1649 (C=O), 1590 (C=N). ^1H NMR spectrum, δ, ppm: 5.42 s (2H, CH_2), 7.33–7.36 d (2H, Ar-H), 7.69–7.71 m (4H, Ar-H), 7.88–7.92 d (1H, Ar-H), 7.92–8.06 m (8H, Ar-H), 8.16–8.18 d (2H, Ar-H), 8.24–8.26 d (1H, Ar-H), 8.67–8.69 d (1H, Ar-H), 8.82 s (1H, Ar-H), 9.06 s (1H, Ar-H). MS: 474 $[M + H]^+$.

(*E*)-1-(4-{[1-(4-Chlorophenyl)-1*H*-1,2,3-triazol-4-yl]methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7j). Yield 82%. IR spectrum, ν, cm^{-1}: 1644 (C=O), 1586 (C=N). ^1H NMR spectrum, δ, ppm: 5.42 s (2H, CH_2), 7.34–7.38 m (3H, Ar-H), 7.55–7.63 m (2H, Ar-H), 7.78–7.85 m (1H, Ar-H), 7.94–7.80 m (2H, Ar-H), 8.19–8.25 m (2H, Ar-H), 8.64–8.66 d (1H, Ar-H), 8.82 s (1H, Ar-H), 9.04 s (1H, Ar-H). MS: 508 $[M + H]^+$.

(*E*)-1-(4-{[1-(4-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7k). Yield 86%. IR spectrum, ν, cm^{-1}: 1652 (C=O), 1593 (C=N). ^1H NMR spectrum, δ, ppm: 5.42 s (2H, CH_2), 7.20–7.24 m (2H, Ar-H), 7.33–7.36 m (1H, Ar-H), 7.54–7.58 t (1H, Ar-H), 7.96–8.06 m (8H, Ar-H), 8.22–8.26 m (4H, Ar-H), 8.44–8.48 m (2H, Ar-H), 8.65–8.69 m (2H, Ar-H), 8.82 s (1H, Ar-H), 9.21 s (1H, Ar-H), 12.09 s (1H, Ar-H). MS: 519 $[M + H]^+$.

(*E*)-1-(4-{[1-(2-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methoxy}phenyl)-3-(tetrazolo[1,5-*a*]quinolin-4-yl)-prop-2-en-1-one (7l). Yield 82%. IR spectrum, ν, cm^{-1}: 1645 (C=O), 1591 (C=N). ^1H NMR spectrum, δ, ppm: 5.42 s (2H, CH_2), 7.25–7.32 m (3H, Ar-H), 7.41–7.52 m (1H, Ar-H), 7.59–7.62 t (1H, Ar-H), 7.83–7.87 d (1H, Ar-H), 7.79–8.00 m (5H, Ar-H), 8.22–8.31 d (2H, Ar-H), 8.38–8.43 m (2H, Ar-H), 8.65–8.367 d (1H, Ar-H), 8.80 s (1H, Ar-H), 9.12 s (1H, Ar-H), 12.10 s (1H, Ar-H). MS: 519 $[M + H]^+$.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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