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Traceless Synthesis of 3-N-Substituted-2-Thioxoquinazoline-4-Ones on a Soluble Polymeric Support

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Abstract: The synthesis of 3-N-substituted-2-thioxoquinazoline-4-ones is described with a traceless linker strategy using poly(ethylene glycol) (PEG) as a soluble polymeric support. Staudinger-Aza-Wittig reaction of PEG-supported azide with Ph_3P and CS_2 gave the corresponding PEG-supported phenyl isothiocyanate. Treatment of the phenyl isothiocyanate with different primary amines led, *via* intramolecular cyclization and simultaneous cleavage from PEG, to 2-thioxoquinazoline-4-ones with of moderate to excellent yields.

Keywords: Poly (ethylene glycol), support, liquid phase synthesis, 2-thioxoquinazoline-4-ones, Aza-Wittig reaction.

INTRODUCTION

In recent years, the design and synthesis of pharmacologically relevant heterocyclic molecules using soluble polymers as supports has greatly attracted the interest of organic chemists in the field of combinatorial chemistry [1]. This approach benefits from the advantages of both homogeneous solution chemistry (high reactivity and simple analytical procedures) and solid-phase methods (use of excess reagents, easy isolation and purification of products). In particular, among the soluble supports, poly(ethylene glycol) (PEG) is an ideal support and has been used intensively for polymer-supported solution synthesis because of its unique solubility properties [2].

2-Thioxoquinazoline-4-ones have attracted considerable attention, because they possess an array of biological activities and provide an incentive for future exploration of this class of compounds as potential drug precursors [3]. However, to the best of our knowledge, little effort has been made toward the synthesis of this class of bioactive compounds [4]. Makino *et al.* [4b, c] reported the solid-phase synthesis of quinazoline-2-thioxo-4-ones by reacting SynPhase™ lantern supported derivatized amines with isothiocyanate, which required careful selection of the solid support linkers to avoid *S*-alkylation.

Based on our research on the PEG-supported synthesis of substituted heterocyclic compounds [5], we report herein the traceless linker synthesis of substituted 2-thioxoquinazoline-4-ones for the first time by using PEG as a soluble support. One of the key steps in our synthetic approach is the Aza-Wittig reaction [6].

CHEMISTRY

As described in Scheme 1, PEG (MW=4000, dihydroxy) was coupled with *o*-azidobenzoic acid **1** using 2 equiv of DCC and 0.15 equiv of DMAP in CH_2Cl_2 to give the PEG–bound *o*-azido ester **2** through ester linkage. The disappearance of absorption for the hydroxy group at 3469 cm^{-1} and the appearance of an ester group and an azido group at 1748 cm^{-1} and 2107 cm^{-1}, respectively, in the IR spectrum was clear evidence for the formation of **2**. Ph_3P reacted with **2** to form almost quantitatively the PEG-bound phosphorane **3**. This compound underwent an Aza-Wittig reaction with CS_2 in dry CH_2Cl_2 at 40 ℃ for 24 h, which produced PEG-bound phenyl isothiocyanate **4** quantitatively. The completion of the reaction was indicated by the disappearance of the azido group and appearance of the N=C=S group at 2117 cm^{-1}. Compound **4** reacted with 2 equiv of the primary amines and 0.3 equiv of Et_3N in CH_3CN to give the desired 3-N-substituted-2- thioxoquinazoline-4-ones **6** through the intermediate N, N-disubstituted thioureas **5**. This cyclization-cleavage strategy thus combined linker cleavage and ring formation in single reaction step.

RESULTS AND DISCUSSION

A variety of 3-N-substituted-2-thioxo- quinazoline-4-ones were synthesized using this procedure. Primary aliphatic amines and common aromatic amines gave good results, while sterically hindered amines such as *tert*- butyl amine and cyclohexylamine and aromatic amines with a strong electron-withdrawing group (such as 2-chloroaniline, 2,5-dichlo- roaniline, 2-nitroaniline and 4-nitroaniline) failed to give the target compound. The stepwise formation of various 3-N-substituted-2- thioxoquinazoline-4-ones is illustrated in Scheme 1, and the results are shown in Table **1**. The yields were moderate to excellent.

It is worth noting that, in each step of the sequence, the PEG-bound products were precipitated selectively, and the excess low molecular reagents and the by-products were removed by simple filtration. TLC was used to confirm the complete removal of the excess reagents and the soluble by-products. Compared to solid phase organic synthesis [4b, c] and besides above advantages, the PEG-bound intermediates were easily analyzed to confirm the structures by using routine analytical methods (UV, IR, HNMR) without resorting to cleave-and-analyze techniques followed by spectroscopic analysis.

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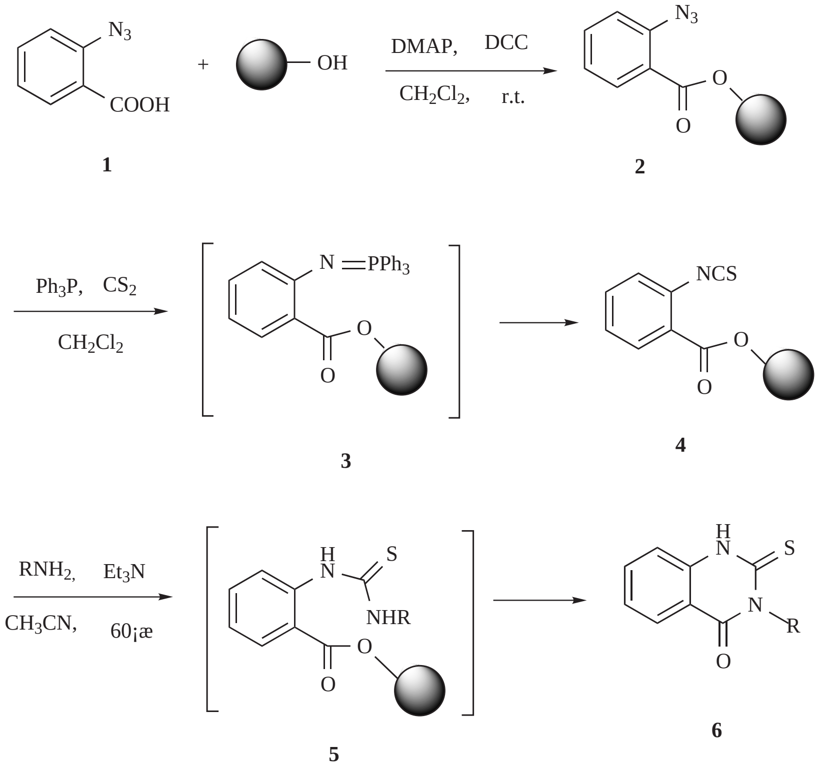

Scheme 1. Synthetic route to 3-N-substituted-2- thioxoquinazoline-4-ones.

CONCLUSIONS

In conclusion, we have demonstrated the use of PEG-bound azide compounds for the efficient traceless synthesis of 3-N-substituted-2-thioxoquinazoline-4-ones by means of the Aza-Wittig reaction. As shown in the experimental and results, this method is simple, the reaction conditions are mild and the yield is moderate to excellent.

EXPERIMENTAL

Preparation of PEG-Bound *o*-Azidobenzoic Acid 2

To a solution of PEG (12 g) in dry CH_2Cl_2 (30 mL) was added *o*-azidobenzoic acid (1.96 g, 12 mmol), DCC (2.47 g, 12 mmol) and DMAP (0.12 g 0.9 mmol). The mixture was stirred at r.t. for 18 h and then filtered to remove the resulting urea. The mixture was concentrated, and the residue was precipitated with cold Et_2O (250 mL). Then the precipitate was filtered, washed with cold Et_2O (30×3mL), and dried under vacuum to give white PEG-bound *o*-azidobenzoic acid **2** (12.2 g, 98.4%), TLC showed no residual low molecular weight reagents or by-products. IR (KBr): 1728 (CO), 2115 ($Ar\text{-}N_3$) cm^{-1}. ^1H NMR (600Mz, $CDCl_3$): δ=7.82~7.23 (m, 4H, ph-H), 3.83~3.25(m, $-OCH_2CH_2O-$).

Preparation of PEG-Bound Phenyl Isothiocyanate 4

A solution of **2** (12.2 g), Ph_3P (1.54 g, 5.88 mmol) and CS_2 (6 mL) in dry CH_2Cl_2 (30 mL) was stirred at 40℃ for 24 h. After evaporation of the solvent at reduced pressure, the

Table 1. Synthesis of 3-N-Substituted-2-Thioxoquinazoline-4-Ones 6 Using PEG as Solid Support

Entry	R	Overall Yield *(%)	Entry	R	Overall Yield* (%)
6a	propyl	76	**6l**	phenyl	90
6b	isopropyl	83	**6m**	2,3-dimethylphenyl	88
6c	butyl	82	**6n**	2,4-dimethylphenyl	85
6d	isobutyl	76	**6o**	2,5-dimethylphenyl	78
6e	sec-butyl	80	**6p**	2,6-dimethylphenyl	81
6f	pentyl	88	**6q**	3,4-dimethylphenyl	88
6g	hexyl	83	**6r**	4-chlorophenyl	76
6h	heptyl	78	**6s**	3,4-dichloro phenyl	89
6i	n-dodecyl	90	**6t**	4-bromophenyl	75
6j	n-hexadecyl	89	**6u**	4-(trifluoromethoxy) phenyl	68
6k	n-octadecyl	90	**6v**	3-(trifluromethyl) phenyl	50

*Based on the loading capacity of PEG.

residue was precipitated with cold Et_2O (250 mL), filtered, washed with cold Et_2O (30 mL×3) and dried under vacuum to give PEG-bound phenyl isothiocyanate **4** (11.6 g, 95.1%). IR (KBr): 1717 (CO), 2117 (NCS) cm^{-1}. ^{1}H NMR (600Mz, $CDCl_3$): δ=7.89~7.41 (m, 4H, ph-H), 3.77~3.33 (m, -OCH_2CH_2O-).

General Preparation of 3-N-Substituted-2-Thioxoquinazoline-4-Ones 6

A mixture of **4** (3.00 g), Et_3N (1 mL) and amines (3.0 mmol) was stirred in CH_3CN (20 mL) at r.t. or refluxed for 30 min to 12 h, depending on the reactivity of the amine. After evaporation of the solvent at reduced pressure, the residue was redissolved in a small amount of CH_2Cl_2. Next, the detached PEG was precipitated by adding cold Et_2O and removed by filtration. Then, the combined filtrate was evaporated to give crude product, which was purified by recrystallization or column chromatography on silica gel (EtOAc: *n*-hexane, 1:4) to give the desired pure product **6** [7].

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[7] **6a**: Mp 195.6~196.6ºC. IR (KBr): υ=3247, 1648cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=1.02 (t, J_1=J_2=7.8Hz, 3H, CH_3), 1.61~1.86 (m, 2H, CH_2CH_3), 4.48(t, J_1=J_2= 7.8Hz, 2H, NCH_2), 7.14~8.14 (m, 4H, ArH), 10.31(s,1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=11.81, 20.74, 49.12, 114.97, 116.78, 125.47, 129.18, 135.91, 138.93, 159.42, 176.23. EIMS: *m/z* 220.4 [M]$^{+\cdot}$.

6b: Mp 233.0~234.0ºC. IR (KBr): υ=3246, 1650 cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ= 1.25 (s, 3H, CH_3), 1.26 (s, 3H, CH_3), 6.10~6.05 (m, 1H, CH), 7.17~8.10 (m, 4H, ArH), 10.75(s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ=18.81 (2C), 114.32, 117.46, 124.98, 128.09, 132.21, 135.15, 138.16, 159.69, 176.60. EIMS: *m/z* 220.4 [M]$^{+\cdot}$.

6c: Mp 177.7~178.1ºC. IR (KBr): υ=3242, 1651cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.99 (t, J_1=J_2= 7.8Hz, 3H, CH_3), 1.44~1.81 (m, 4H, $CH_2CH_2CH_3$), 4.52 (t, J_1=J_2=7.8, 2H, NCH_2), 7.15~8.14 (m, 4H, ArH), 10.40 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=14.26, 20.69, 29.33, 47.45, 114.92, 116.71, 125.42, 129.05, 135.83, 138.86, 159.82, 176.34. EIMS: *m/z* 234.2 [M]$^{+\cdot}$.

6d: Mp 133.6~134.6ºC. IR (KBr): υ=3245, 1650cm^{-1}.^{1}H NMR (600 MHz, $CDCl_3$): δ=0.93 (t, J_1=J_2=7.2Hz, 3H, CH_2CH_3), 1.58(d, J=7.8Hz, 3H, $CHCH_3$), 1.96~2.25(m, 2H, CH_2CH_3), 5.90 (d, *J*=7.8Hz, 1H, NCH), 7.18~8.10(m, 4H, ArH), 10.73 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=11.81, 17.35, 26.58, 60.79, 114.87, 117.80, 125.45, 128.57, 135.80, 138.76, 160.07, 177.79. EIMS: *m/z* 234.2 [M]$^{+\cdot}$.

6e: Mp 213.2~214.0ºC. IR (KBr): υ=3235, 1661cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.98(d, J=6.6Hz, 6H, CH_3), 2.34~2.51(m, 1H, CH), 4.28 (d, *J*=7.2Hz, 2H, NCH_2), 7.32~7.97 (m, 4H, ArH), 12.91 (s, 1H, NH). ^{13}C N MR (150 MHz, $CDCl_3$): δ=19.94, 26.16, 51.73, 115.36, 115.52, 124.41, 127.38, 135.38, 139.03, 159.70, 175.53. EIMS: *m/z* 234.2 [M]$^{+\cdot}$.

6f: Mp 192.0~193.0ºC. IR (KBr): υ=3238, 1668cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.93(d, *J*=6.0Hz, 3H, CH_3), 1.40~1.80(m, 6H, CH_2), 4.50 (t, J_1=J_2=7.8Hz, 2H, NCH_2), 7.11~8.15(m, 4H, ArH), 10.19 (s, 1H, NH).^{13}C NMR (150 MHz, $CDCl_3$): δ=13.99, 22.35, 26.38, 29.01, 47.14, 114.39, 116.16, 124.93, 128.56, 135.34, 138.29, 159.50, 175.78. EIMS: *m/z* 248.1 [M]$^{+\cdot}$.

6g: Mp 173.4~175.9ºC. IR (KBr): υ=3241, 1674cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.90 (t, J_1=J_2=7.2 Hz, 3H, CH_3), 1.33~ 1.83(m, 8H, CH_2), 4.52 (t, J_1=J_2=7.8Hz, 2H, NCH_2), 7.20~8.14 (m, 4H, ArH), 10.93 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=14.26, 23.08, 26.57, 28.39, 31.28, 47.43, 115.42, 117.54, 124.95, 128.82, 135.63, 138.12, 161.32, 177.24. EIMS: *m/z* 262.3 [M]$^{+\cdot}$.

6h: Mp 165.3~167.5ºC. IR (KBr): υ=3196, 1690cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.89 (t, J_1=J_2=7.2Hz, 3H, CH_3), 1.29~1.83 (m, 10H, $(CH_2)_5$), 4.51 (t, J_1=J_2=7.8Hz, 2H, NCH_2), 7.21~8.14 (m, 4H, ArH), 10.78(s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): 14.57, 23.06, 26.03, 27.20, 29.39, 32.22, 47.59, 115.03, 116.66, 125.47, 128.95, 134.81, 138.86, 160.01, 176.12. EIMS: *m/z* 276.2 [M]$^{+\cdot}$.

6i: Mp 88.6~89.0 ºC. IR (KBr): υ=3194, 1697 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.88 (t, J_1=J_2=6.0Hz, 3H, CH_3), 1.25~ 1.80 (m, 20H, CH_2), 4.50 (t, J_1=J_2=7.8Hz, 2H, NCH_2), 7.15~8.14 (m, 4H, ArH), 10.42 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=14.11, 22.58, 26.10, 26.54, 29.12, 29.25, 29.40, 29.56, 29.78, 29.80, 31.90, 45.72, 114.79, 116.02, 125.01, 128.60, 135.42, 137.93, 159.51, 175.57. EIMS: *m/z* 346.5 [M]$^{+\cdot}$.

6j: Mp 138.4~140.0 ºC. IR (KBr): υ=3210, 1695 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.88 (t, J_1=J_2=7.2Hz, 3H, CH_3), 1.25~1.82 (m, 28H, CH_2), 4.50 (t, J_1=J_2= 7.8Hz, 2H, NCH_2), 7.16~8.14 (m, 4H, ArH), 10.50 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=14.10, 22.66, 26.10, 26.48, 26.70, 26.91, 29.26, 29.33, 29.54, 29.58, 29.63, 29.66, 29.68, 29.93, 31.90, 47.13, 114.49, 116.17, 124.94, 128.50, 135.32, 138.35, 159.51, 175.70. EIMS: *m/z* 402.5 [M]$^{+\cdot}$.

6k: Mp 139.7~141.5 ºC. IR (KBr): υ=3212, 1690 cm^{-1}, ^{1}H NMR (600 MHz, $CDCl_3$): δ=0.87 (t, J_1=J_2=7.2Hz, 3H, CH_3), 1.25~1.80 (m, 30H, CH_2), 4.49 (t, J_1=J_2=7.8Hz, 2H, NCH_2), 7.10~8.14 (m, 4H, ArH), 10.10(s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=14.10, 22.67, 26.30, 26.41,26.55, 26.61, 26.70, 26.92, 29.26, 29.31, 29.54, 29.64,29.70, 29.75, 29.81, 29.84, 31.85, 46.07, 114.30, 116.09,124.83, 128.30, 134.98, 138.50, 159.43, 175.80. EIMS: *m/z* 430.5 [M]$^{+\cdot}$.

6l: Mp 266.6~268.4ºC(lit.[7]307ºC), IR (KBr): υ=3206, 1673 cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=7.28~7.95 (m, 9H, ArH), 13.04 (s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ= 114.82, 115.93, 123.08 (2C), 124.01, 126.91 (2C), 129.04, 132.96, 135.78, 138.99, 139.05, 159.40, 175.94. EIMS: *m/z* 254.1 [M]$^{+\cdot}$.

6m: Mp 270.1~271ºC. IR (KBr): υ=3206, 1673 cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=1.91(s, 3H, CH_3), 2.30 (s, 3H, CH_3), 7.01~ 7.96 (m, 7H, ArH), 13.07 (s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ=10.30, 15.87, 115.60, 116.81, 124.67, 125.90, 127.32, 128.01, 130.91, 132.54, 135.79, 136.01, 138.88, 139.20, 159.72, 175.96. EIMS: *m/z* 282.1[M]$^{+\cdot}$.

6n: Mp 247.3~248ºC. IR (KBr): υ=3206, 1673cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=2.15(s, 3H, CH_3), 2.40 (s, 3H, CH_3), 7.05~ 8.18 (m, 7H, ArH), 11.18 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=17.38, 21.35, 114.96, 116.30, 125.09, 127.82, 128.10, 128.81, 132.04, 135.01, 135.07, 135.68, 138.93, 139.30, 159.67, 176.19. EIMS: *m/z* 282.1 [M]$^{+\cdot}$.

6o: Mp 144.0~145.7ºC. IR (KBr): υ=3206, 1673 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=2.16(s, 3H, CH_3), 2.41 (s, 3H, CH_3), 7.05~8.18(m, 7H, ArH), 11.09 (s, 1H, NH).^{1} ^{3}C NMR (150 MHz, $CDCl_3$): δ=16.30, 23.07, 114.89, 116.72, 125.21, 127.91, 128.30, 129.01, 132.00, 134.95, 135.10, 135.70, 138.87, 139.20, 159.09, 175.03. EIMS: *m/z* 282.1 [M]$^{+\cdot}$.

6p: Mp 239.2~240.8ºC. IR (KBr): υ=3105, 1682 cm^{-1}. ^{1}H NMR (600 MHz, $CDCl_3$): δ=2.06 (m, 6H, CH_3ArCH_3), 7.16~8.26 (m, 7H, ArH), 10.98 (s, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ=16.88 (2C), 115.02, 116.47, 124.59, 124.72, 126.80 (2C),

128.63, 135.40 (2C), 135.61, 138.82, 140.01, 159.70, 175.96. EIMS: *m/z* 282.1 $[M]^{+\cdot}$.

6q: Mp 238.1~239.6ºC. IR (KBr): υ＝3124, 1661cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=2.24 (s, 3H, CH_3), 2.28 (s, 3H, CH_3), 6.98~7.95 (m, 7H, ArH), 13.02 (s, 1H, NH).^{13}C NMR (150 MHz, DMSO): δ=19.15, 19.39, 115.68, 116.11, 124.32, 126.10, 127.40, 129.48, 129.91, 135.55, 136.19, 136.82, 136.85, 139.54, 159.83, 176.15. EIMS: *m/z* 282.1 $[M]^{+\cdot}$.

6r: Mp 315.4~316.2ºC (lit.[7]317ºC). IR (KBr): υ＝3206, 1676 cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=7.34~7.96(m, 8H, ArH), 13.09(s, 1H, NH). ^{13}C NMR (150MHz, DMSO): δ=115.72, 116.20, 124.41, 127.41, 129.00 (2C), 131.05 (2C), 132.71, 135.66, 138.24, 139.59, 159.78, 175.89. EIMS: *m/z* 288.6 $[M]^{+\cdot}$.

6s: Mp 283.8~285.2 ºC. IR (KBr): υ=3174, 1685 cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=7.35~7.96 (m, 7H, ArH), 13.12 (s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ=115.74, 116.18, 124.41, 127.35, 129.86, 130.80, 130.98, 131.12, 131.40, 135.67, 139.19, 139.56, 159.68, 175.65. EIMS: *m/z* 323.2 $[M]^{+\cdot}$.

6t: Mp 302~303.7ºC. IR (KBr): υ=3208, 1675 cm^{-1}.^{1}H NMR (600 MHz, DMSO): δ=7.27~ 7.97(m, 8H, ArH), 13.07(s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ=115.72, 116.19, 121.28, 124.41, 127.40, 131.39 (2C), 131.96 (2C), 135.66, 138.69, 139.58, 159.73, 175.82. EIMS: *m/z* 333.4 $[M]^{+\cdot}$.

6u: Mp 328.9~331.2ºC. IR (KBr): υ＝3243, 1668cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=7.35~7.97 (m, 8H, ArH), 13.13 (s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ =115.74, 116.30, 124.42, 124.98, 126.35, 127.37, 130.08 (2C), 133.59 (2C), 135.68, 139.62, 140.02, 159.87, 175.84. EIMS: *m/z* 336.5 $[M]^{+\cdot}$.

6v: Mp 277.8~280.0ºC. IR (KBr): υ=3246, 1660cm^{-1}. ^{1}H NMR (600 MHz, DMSO): δ=7.34~7.96 (m, 8H, ArH), 13.10(s, 1H, NH). ^{13}C NMR (150 MHz, DMSO): δ=114.08, 115.71, 116.19, 120.90, 121.46, 121.90, 124.39, 127.37, 131.21, 135.64, 138.27, 139.58, 147.79, 159.80, 175.89. EIMS: *m/z* 332.1$[M]^{+}$.

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