HETEROCYCLES, Vol. 94, No. 9, 2017, pp. 1759 - 1765. © 2017 The Japan Institute of Heterocyclic Chemistry
Received, 31st May, 2017, Accepted, 20th July, 2017, Published online, 28th July, 2017
DOI: 10.3987/COM-17-13756

ONE-POT SYNTHESIS OF 2-SUBSTITUTED 3-THIOXO-2,3-DIHYDRO-1*H*-ISOINDOL-1-ONES BY THE REACTION OF *N*-SUBSTITUTED 2,*N*-DILITHIOBENZAMIDES WITH ISOTHIOCYANATES

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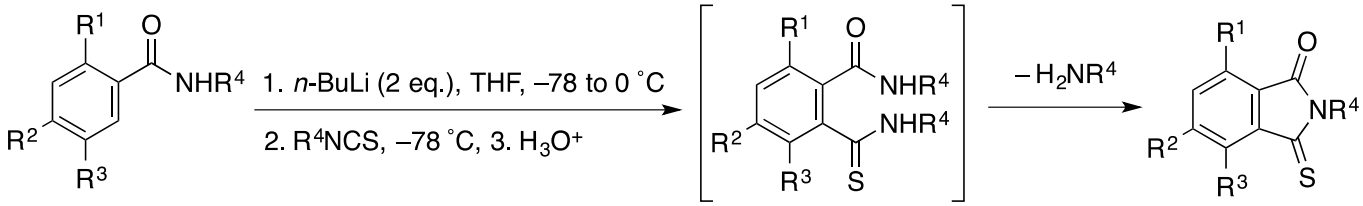
Abstract – This paper describes an efficient one-pot method for the preparation of 2-substituted 3-thioxo-1*H*-isoindol-1-ones (3-thioxoisoindolinones) by the reaction of *N*-substituted 2,*N*-dilithiobenzamides, generated by treating *N*-substituted benzamides with two equivalents of butyllithium, with the respective isothiocyanates followed by ring closure of the resulting *N*-alkyl(or aryl)-2-(alkyl(or aryl)thiocarbamoyl)benzamide derivatives with loss of alkyl(or aryl)amines.

Substituted 3-thioxo-2,3-dihydro-1*H*-isoindol-1-ones are an important class of heterocyclic compounds and have shown diverse and excellent biological effects,[1] such as fungicidal,[1a] sarcopenia preventive[1d] and antiangiogenic activities.[1h] While these derivatives have been ordinarily prepared by the reaction of phthalimide derivatives with P_2S_5[2] or Lawesson's reagent[3] under rather drastic conditions, there is a need for development of new and facile method to be conducted under mild conditions.

We recently found that *N*-substituted 2,*N*-dilithiobenzamides, generated by treating *N*-substituted benzamides with two equivalents of butyllithium, react with α-keto esters to give 4-substituted 4-hydroxyisoquinoline-1,3(2*H*,4*H*)-dione derivatives in one pot.[4] In our continuing study to extend the utility of these dilithium compounds,[5] it has been found that the reaction of *N*-substituted 2,*N*-dilithiobenzamides with alkyl(or aryl) (same to the *N*-substituent of the starting benzamides) isothiocyanates gave, after aqueous workup followed by spontaneous cyclization of the resulting 2-thiocarbamoylbenzamides with loss of alkyl(or aryl)amines, 2-substituted 3-thioxo-2,3-dihydro-1*H*-isoindol-1-ones in one pot. Herein, we wish to report the results of our investigation, which provide a novel and efficient method for the synthesis of this interesting class of isoindolinones. A series of

3-thioxoisoindolinone derivatives, which are hard to prepare by the previous methods, can be obtained feasibly by using the present method.

Our one-pot synthesis of 3-thioxo-2,3-dihydro-1*H*-isoindol-1-one derivatives (**3**) from *N*-substituted benzamides (**1**) was conducted according to the procedure illustrated in Scheme 1. The starting compounds (**1**) were readily prepared from commercially available benzoyl chlorides and primary amines using literature procedures.[4-9] First, *N*-methylbenzamide (**1a**) was treated with two equivalents of butyllithium in THF at –78 ˚C and temperature was then raised to 0 ˚C. The resulting mixture was cooled again to –78 ˚C and methyl isothiocyanate was added. The resulting reaction mixture was quenched with saturated aqueous NH_4Cl, followed by extraction with ethyl acetate and concentration. After subsequent purification of the crude products on column chromatography on silica gel, 2-methyl-3-thioxo-2,3-isoindol-1-one (**3a**) was obtained in 56% yield (Table 1, Entry 1). Before aqueous workup, TLC analyses on silica gel of the reaction mixture scarcely revealed the presence of **3a**, though a new large spot somewhat less mobile than the starting material **1a** could be observed. After aqueous workup, this large spot had disappeared and the spot due to **3a** could be observed obviously. It is thought that the initially formed *N*-methyl-2-(methylthiocarbamoyl)benzamide (**2a**) spontaneously cyclized with loss of methylamine during workup to afford **3a**. The spectral (IR, ^{1}H and ^{13}C NMR) data of this product were identical to those of previously synthesized compound.[3] Then, the reactions of the corresponding 2,*N*-dilithiobenzamides, generated from the other nine *N*-substituted benzamides (**1b-j**) on the treatment with butyllithium, with the respective isothiocyanates, were similarly carried out, and the reaction mixtures were worked up and subsequently purified in a manner similar to that described above for the preparation of **3a**. The initially formed 2-thiocarbamoylbenzamides (**2b-j**) lost the corresponding primary amines to yield the desired products (**3b-j**).



1 **2** **3**

Scheme 1

The yields of these products are summarized in Table 1 as well. Although the production of two regioisomeric products was possible in each reaction using **1e**, **1h**, **1i** and **1j**, the corresponding products (**3e**), (**3h**), (**3i**) and (**3j**), respectively, were obtained as sole isolated products (Entries 5, 8, 9, and 10). This indicates that the 3-chloro and 3-methoxy substituents could completely control the regioselective lithiation at the 2-position as orientation groups. However, the yield of **3e** was somewhat lower than those

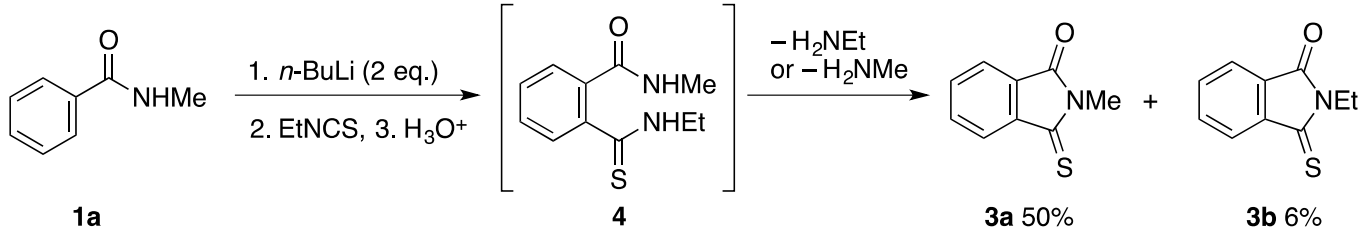
of the others. This may be ascribed to the lower stability of the 2,*N*-dilithio product from **1e** due to the lability to the formation of the corresponding benzyne derivative. *N*-Arylbenzamides (**1k**) and (**1l**) did not appreciably perform well. The reactions of these amides with two equivalents of butyllithium and then the respective aryl isothiocyanates under similar conditions described above to give, after similar aqueous workup and purification, only lower yields of the corresponding 2-aryl-3-thioxo-2,3-dihydroisoindol-1-ones (**3k**) and (**3l**) along with considerable amounts of recovered starting amides (Entries 11 and 12). This implies that the corresponding 2,*N*-dilithio products were generated in low yields under the conditions probably due to the lower coordinative ability of the arylamino groups compared to the alkylamino groups.

Table 1. Preparation of *N*-substituted 3-thioxo-2,3-dihydroisoindol-1-ones (**3**)

Entry	**1**	R^1	R^2	R^3	R^4	**3**	Yield/%[a]
1	**1a**	H	H	H	Me	**3a**	56
2	**1b**	H	H	H	Et	**3b**	54
3	**1c**	Cl	H	H	Me	**3c**	47
4	**1d**	H	Cl	H	Me	**3d**	52
5	**1e**	H	H	Cl	Me	**3e**	35
6	**1f**	H	OMe	H	Me	**3f**	45
7	**1g**	H	OMe	H	Et	**3g**	41
8	**1h**	H	H	OMe	Me	**3h**	56
9	**1i**	H	H	OMe	Et	**3i**	54
10	**1j**	H	H	OMe	*n*-Bu	**3j**	43
11	**1k**	H	H	H	Ph	**3k**	11
12	**1l**	H	Cl	H	4-$MeOC_6H_4$	**3l**	10

[a] Yields of isolated products.

We attempted the reaction of 2,*N*-dilithio products from *N*-methylbenzamide (**1a**) with ethyl isothiocyanate in order to clarify the mode of the cyclization of intermediary 2-(ethylthiocarbamoyl)-*N*-methylbenzamide (**4**). As illustrated in Scheme 2, 2-methyl-3-thioxo-2,3-isoindol-1-one (**3a**) was predominantly obtained (50%) along with a small quantity (6%) of 2-ethyl-3-thioxo-2,3-isoindol-1-one (**3b**). These products were isolated using column chromatography on silica gel (AcOEt/hexane 1:5) as an inseparable mixture, and the ratio was determined by its ^{1}H NMR spectroscopy. This indicates that the cyclization occurred mainly by the attack of amide nitrogen on the thiocarbonyl of the thioamide unit.



1a **4** **3a** 50% **3b** 6%

Scheme 2

In conclusion, we have demonstrated that the reaction of *N*-substituted 2,*N*-dilithiobenzamides with the respective isothiocyanates gives 2-substituted 2,3-dihydro-1*H*-isoindole-1-thiones of medicinally potent importance. The method described in this paper may have some potential utility for organic molecular transformations. Further studies on the synthesis of heterocyclic compounds utilizing similar 2,*N*-dilithiobenzamides are ongoing in our laboratory and will be reported in due course.

EXPERIMENTAL

All melting points were obtained on a Laboratory Devices MEL-TEMP II melting apparatus and are uncorrected. IR spectra were recorded with a Perkin–Elmer Spectrum 65 FTIR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ using TMS as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 500 and 125 MHz, respectively. High-resolution MS spectra were measured by a JEOL JMS-T100GCV (EI or FI, TOF; 70 eV or 2100 V, respectively) spectrometer. Elemental analyses were performed with an Elementar Vario EL II instrument. TLC was carried out on Merck Kieselgel 60 PF_{254}. Column chromatography was performed using WAKO GEL C-200E. All of the organic solvents used in this study were dried over appropriate drying agents and distilled prior to use.

Starting Materials. For preparation of *N*-alkylbenzamides (**1a**), (**1b**), (**1c**), (**1d**), (**1e**), (**1f**), (**1h**), and (**1i**) see ref. 4. Compounds (**1g**),[6] (**1j**),[9] (**1k**),[7] and (**1l**)[8] were prepared according to the appropriate reported procedures. Butyllithium was supplied by Asia Lithium Corporation. All other chemicals used in this study were commercially available.

Typical Procedure for the Preparation of 3-Thioxo-2,3-dihydro-1*H*-isoindol-1-ones. 2-Methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3a).[10] To a stirred solution of **1a** (0.20 g, 1.5 mmol) in THF (10 mL) at –78 ˚C was added *n*-BuLi (1.6 M in hexane; 3.0 mmol) dropwise and the temperature was gradually raised to 0 ˚C. After stirring for 1 h at the same temperature, the mixture was cooled to –78 ˚C and MeNCS (0.11 g, 1.5 mmol) was added dropwise. After 5 min, saturated aqueous NH_4Cl (25 mL) was added and the mixture was extracted with AcOEt (3 × 15 mL). The combined extracts were washed with brine (25 mL), dried (Na_2SO_4), and stood overnight at rt. Concentration of the solution by evaporation gave a residue, which was purified by column chromatography on SiO_2 (AcOEt/hexane 1:5) to give the product (**3a**) (0.14 g, 54%); a red solid; mp 95–96 ˚C (hexane/CH_2Cl_2) (lit.,[11] 97–98 ˚C). Spectral (IR, ^{1}H and ^{13}C NMR) data for this compound were identical to those reported previously.[3]

2-Ethyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3b):[12] a red oil; R_f 0.56 (AcOEt/hexane 1:10); IR (neat) 1740, 1074 cm^{-1}; ^{1}H NMR δ 1.29 (t, J = 7.4 Hz, 3H), 4.11 (q, J = 7.4 Hz, 2H), 7.68–7.69 (m, 2H), 7.76 (dd, J = 8.6, 3.4 Hz, 1H), 7.96 (dd, J = 8.0, 2.9 Hz, 1H); ^{13}C NMR δ 13.2, 35.9, 122.6, 123.7, 127.4, 133.0, 133.9, 137.2, 169.6, 196.7.

7-Chloro-2-methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3c): an orange solid; mp 150–152 ˚C (hexane/CH_2Cl_2); IR (KBr) 1754, 1306 cm^{-1}; ^1H NMR δ 3.47 (s, 3H), 7.58–7.63 (m, 2H), 7.88 (d, J = 6.9 Hz, 1H); ^{13}C NMR δ 27.8, 122.3, 123.5, 130.9, 134.7 (two overlapped Cs), 138.8, 167.4, 195.0. MR-MS (EI). Calcd for C_9H_6ClNOS (M): 210.9859. Found: *m/z* 210.9859. Anal. Calcd for C_9H_6ClNOS: C, 51.07; H, 2.86; N, 6.62. Found: C, 50.85; H, 2.96; N, 6.48.

5-Chloro-2-methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3d): an orange solid; mp 139–141 ˚C (hexane/CH_2Cl_2); IR (KBr) 1740, 1318 cm^{-1}; ^1H NMR δ 3.46 (s, 3H), 7.65 (dd, J = 8.0, 1.7 Hz, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.91 (d, J = 1.7 Hz, 1H); ^{13}C NMR δ 27.8, 123.99, 124.01, 125.6, 133.0, 138.3, 140.6, 168.8, 195.5. MR-MS (EI). Calcd for C_9H_6ClNOS (M): 210.9859. Found: *m/z* 210.9862. Anal. Calcd for C_9H_6ClNOS: C, 51.07; H, 2.86; N, 6.62. Found: C, 51.16; H, 2.96; N, 6.69.

4-Chloro-2-methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3e): a pink solid; mp 168–170 ˚C (hexane/CH_2Cl_2); IR (KBr) 1736, 1307 cm^{-1}; ^1H NMR δ 3.45 (s, 3H), 7.58 (dd, J = 8.0, 7.4 Hz, 1H), 7.68 (dd, J = 8.0, 1.1 Hz, 1H), 7.74 (dd, J = 7.4, 1.1 Hz, 1H); ^{13}C NMR δ 27.6, 121.7, 130.4, 130.5, 132.1, 133.1, 136.9, 168.7, 193.4. MR-MS (EI). Calcd for C_9H_6ClNOS (M): 210.9859. Found: *m/z* 210.9857. Anal. Calcd for C_9H_6ClNOS: C, 51.07; H, 2.86; N, 6.62. Found: C, 51.05; H, 2.99; N, 6.69.

5-Methoxy-2-methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3f): a yellow solid; mp 125–127 ˚C (hexane/CH_2Cl_2); IR (KBr) 1747, 1320 cm^{-1}; ^1H NMR δ 3.44 (s, 3H), 3.95 (s, 3H), 7.12 (dd, J = 8.6, 2.3 Hz, 1H), 7.42 (d, J = 2.3 Hz, 1H), 7.67 (d, J = 8.6 Hz, 1H); ^{13}C NMR δ 27.6, 56.0, 108.2, 119.2, 119.5, 124.5, 149.6, 164.7, 169.3, 197.0. MR-MS (EI). Calcd for $C_{10}H_9NO_2S$ (M): 207.0354. Found: *m/z* 207.0346. Anal. Calcd for $C_{10}H_9NO_2S$: C, 57.96; H, 4.38; N, 6.76. Found: C, 57.71; H, 4.40; N, 6.76.

2-Ethyl-5-methoxy-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3g): a yellow solid; mp 76–77 ˚C (hexane/CH_2Cl_2); IR (KBr) 1732, 1338 cm^{-1}; ^1H NMR δ 1.28 (t, J = 7.4 Hz, 3H), 3.95 (s, 3H), 4.07 (q, J = 7.4 Hz, 2H), 7.13 (dd, J = 8.0, 2.3 Hz, 1H), 7.42 (d, J = 2.3 Hz, 1H), 7.67 (d, J = 8.0 Hz, 1H); ^{13}C NMR δ 13.2, 36.0, 56.0, 108.2, 119.3, 119.5, 124.4, 139.8, 164.7, 169.1, 196.3. MR-MS (EI). Calcd for $C_{11}H_{11}NO_2S$ (M): 221.0510. Found: *m/z* 221.0504. Anal. Calcd for $C_{11}H_{11}NO_2S$: C, 59.71; H, 5.01; N, 6.33. Found: C, 59.35; H, 4.98; N, 6.39.

4-Methoxy-2-methyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3h): a red solid; mp 165–166 ˚C (hexane/CH_2Cl_2); IR (KBr) 1728, 1610, 1048 cm^{-1}; ^1H NMR δ 3.41 (s, 3H), 4.02 (s, 3H), 7.24 (d, J = 7.4 Hz, 1H), 7.39 (d, J = 7.4 Hz, 1H), 7.62 (t, J = 7.4 Hz, 1H); ^{13}C NMR δ 27.1, 56.2, 115.0, 118.1, 122.4, 130.5, 134.7, 157.3, 170.0, 194.6. MR-MS (FI). Calcd for $C_{10}H_9NO_2S$ (M): 207.0354. Found: *m/z* 207.0359. Anal. Calcd for $C_{10}H_9NO_2S$: C, 57.96; H, 4.38; N, 6.76. Found: C, 57.82; H, 4.36; N, 6.72.

2-Ethyl-4-methoxy-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3i): a pale-red solid; mp 96–98 ˚C (hexane/CH_2Cl_2); IR (KBr) 1728, 1351 cm^{-1}; ^1H NMR δ 1.25 (t, J = 7.4 Hz, 3H), 4.02 (s, 3H), 4.07 (q, J =

7.4 Hz, 2H), 7.24 (d, *J* = 8.6 Hz, 1H), 7.40 (d, *J* = 7.4 Hz, 1H), 7.62 (dd, *J* = 8.0, 7.4 Hz, 1H); ^{13}C NMR δ 13.2, 35.2, 56.2, 115.0, 118.1, 122.5, 130.5, 134.7, 157.4, 169.4, 193.9. MR-MS (EI). Calcd for $C_{11}H_{11}NO_2S$ (M): 221.0510. Found: *m/z* 221.0501. Anal. Calcd for $C_{11}H_{11}NO_2S$: C, 59.71; H, 5.01; N, 6.33. Found: C, 59.65; H, 4.89; N, 6.37.

2-Butyl-4-methoxy-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3j): a red oil; R_f 0.56 (AcOEt/hexane 1:9); IR (neat) 1733, 1351 cm^{-1}; ^{1}H NMR δ 0.94 (t, *J* = 7.4 Hz, 3H), 1.36 (sext, *J* = 7.4 Hz, 2H), 1.66 (quint, *J* = 7.4 Hz, 2H), 4.01 (t, *J* = 7.4 Hz, 2H), 4.05 (s, 3H), 7.24 (d, *J* = 8.6 Hz, 1H), 7.39 (d, *J* = 7.4 Hz, 1H), 7.62 (dd, *J* = 8.6, 7.4 Hz, 1H); ^{13}C NMR δ 13.7, 20.1, 29.9, 40.1, 56.1, 115.0, 118.1, 122.4, 130.3, 134.6, 157.3, 169.7, 194.2. MR-MS (EI). Calcd for $C_{13}H_{15}NO_2S$ (M): 249.0823. Found: *m/z* 249.0812.

2-Phenyl-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3k):[13] an orange solid; mp 134–136 ˚C (hexane/CH_2Cl_2) (lit.,[14] mp 131–132 ˚C); IR (KBr) 1747, 1302 cm^{-1}; ^{1}H NMR δ 7.36 (dd, *J* = 8.0, 1.1 Hz, 2H), 7.46 (tt, *J* = 7.4, 1.1 Hz, 1H), 7.53 (dd, *J* = 8.0, 7.4 Hz, 2H), 7.76–7.78 (m, 2H), 7.86–7.88 (m, 1H), 8.05–8.06 (m, 1H).

5-Chloro-2-(4-methoxyphenyl)-3-thioxo-2,3-dihydro-1*H*-isoindol-1-one (3l): a orange solid; mp 164–166 ˚C (CH_2Cl_2); IR (KBr) 1750, 1297 cm^{-1}; ^{1}H NMR δ 3.88 (s, 3H), 7.04 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 8.02 (s, 1H); ^{13}C NMR δ 55.5, 114.5, 124.4, 124.6, 125.0, 125.8, 129.3, 133.7, 138.3, 140.9, 159.9, 168.6, 195.6. MR-MS (EI). Calcd for $C_{15}H_{10}ClNO_2S$ (M): 303.0121. Found: *m/z* 303.0133. Anal. Calcd for $C_{15}H_{10}ClNO_2S$: C, 59.31; H, 3.32; N, 4.61. Found: C, 58.92; H, 3.46; N, 4.60.

ACKNOWLEDGEMENTS

We thank Mrs. Miyuki Tanmatsu of this university for recording mass spectra and performing combustion analyses.

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