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Diastereoselective Mukaiyama aldol reaction of (*N*-alkyl) isatins with 2-(trimethylsilyloxy)furan by using lanthanum triflate

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ABSTRACT

An efficient vinylogous Mukaiyama aldol reaction (VMAR) of 2-(trimethylsilyloxy)furan with various (*N*-alkyl)isatins is described in the presence of lanthanum(III) triflates (5 mol %). The reaction proceeds rapidly and affords the corresponding 3-hydroxy-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one in high yields with good diastereoselectivities (*threo*:*erythro* ratio up to ≤95:5).

1. Introduction

Chiral γ-butenolides and their derivatives are common structural subunits in natural products as well as in biologically active compounds.[1] 3-Substituted 3-hydroxyoxindoles are also common skeletons found both in naturally occurring alkaloids and in potent bioactive compounds.[2,3] There are numerous transformations for the synthesis of 3-hydroxy oxindole derivatives from isatin.[3e,c,4] However, recently these compounds are prepared by Mukaiyama aldol protocol in stereoselective fashion.[3a,5a] The vinylogous Mukaiyama aldol reaction rapidly provides 5-(hydroxy(aryl) methyl)furan-2(5*H*)-ones by the addition of dienolate on a carbonyl framework.[6] Over past few years, some elegant enantioselective versions of this reaction have been described using catalytic amounts of various chiral mediators.[7]

2. Results and discussions

Due to medicinal and synthetic importance, it is desirable to develop an efficient and practical method for the synthesis of 3-substituted of oxindole derivatives. One of the most straightforward approach to 3-substituted-3-hydroxyindolin-2-ones is obviously a nucleophilic addition appropriate nucleophiles to isatins. Recently, $La(OTf)_3$ have been used for various organic transformations,[8] because of its high catalytic activity and moisture and air tolerance. Herein, we wish to report the first diastereoselective protocol for catalytic Mukaiyama aldol reaction (MAR) of isatin with 2-(trimethylsilyloxy)furan by using $La(OTf)_3$ as a Lewis acid.

Thus, aldol reaction of 2-(trimethylsilyloxy) furan **2** with isatin **1** underwent smoothly in the presence of $La(OTf)_3$ at −78 °C and resulted into inseparable aldol adduct **3** in high yield with good diastereoselectivity (Table 1, entry 1). The diastereomeric ratio 93:7 (*threo*:*erythro*) was determined by ^{1}H NMR of crude product. The scope and limitations of this protocol was studied with respect to isatin, 2-(trimethylsilyloxy) furan and results are summarised in the Table 1.

We presume that the $La(OTf)_3$ chelates with carbonyls of isatin from above of the plane. Later, 2-(trimethylsilyloxy) furan attack from below of the plane and gave the anti isomer as a major product. Interestingly 5, 6 or 7 substituted isatins reacted with 2-(trimethylsilyloxy) furan in identical conditions and gave corresponding products in good yields without any substantial difference on the selectivity. However the 4-substituted isatins (Table 1, entries 18, 19) gave good yield with less selectivity. This may be attributed to the steric hindrance of substituent. It was observed that alkyl substitutes like methyl, ethyl, and benzyl on nitrogen have no remarkable influence on the dr ratio (Table 1, entries 7, 8, 10). However, the phenyl substitution on nitrogen greatly reduce the selectivity (Table 1, entries 9).

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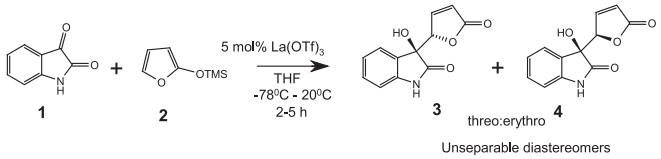
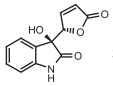
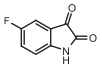
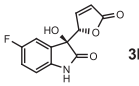
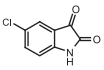
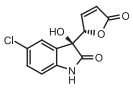
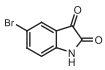
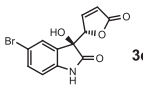
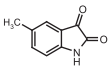
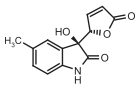
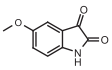
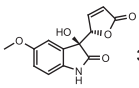
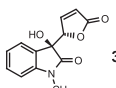
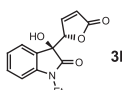
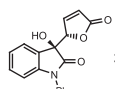
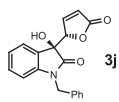
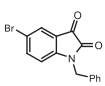
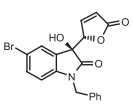
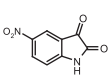
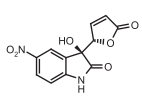
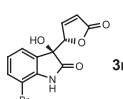
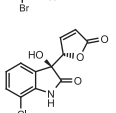
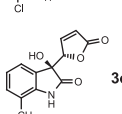
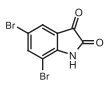
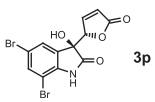
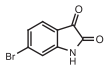
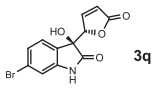
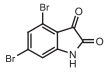
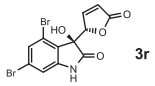
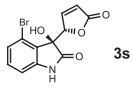
Table 1
Vinylogous La(OTf)3-catalyzed Mukaiyama Aldol Reactions Involving Various (*N*-alkyl) Isatins with 2-(Trimethylsilyloxy)furan[a]



Entry	Isatin (**1**)	Product(**3**) (major Isomer)[b]	Time (h)	Yield (%)[c]	*threo*:*erythro* ratio[d]
1		 **3a**	3	90	93:7
2		 **3b**	2.5	93	95:5
3		 **3c**	2.7	90	93:7
4		 **3d**	2.7	88	94:6
5		 **3e**	4	86	77:23
6		 **3f**	4.5	84	93:7
7		 **3g**	2.5	90	84:16
8		 **3h**	3	88	80:20
9		 **3i**	3.5	86	65:35
10		 **3j**	3.5	88	92:8
11		 **3k**	4	82	84:16
12		 **3l**	5	85	84:16
13		 **3m**	4	90	93:7
14		 **3n**	3.6	89	91:9
15		 **3o**	4.5	90	86:14

(*continued on next page*)

Table 1 (*continued*)

Entry	Isatin (**1**)	Product(**3**) (major Isomer)[b]	Time (h)	Yield (%)[c]	*threo*:*erythro* ratio[d]
16		 **3p**	5	87	84:16
17		 **3q**	3	88	84:16
18		 **3r**	3.5	82	70:30
19		 **3s**	3.7	80	65:35

[a] Reaction was performed with 1 mmol isatin, 1.5 mmol 2-(trimethylsiloxy)furan and 5 mol % $La(OTf)_3$.
[b] All the products were characterised by ^1H and ^{13}C NMR, IR, and mass spectroscopy.
[c] Yield refers to pure product after column chromatography.
[d] *threo*/*erythro* ratio was determined by ^1H NMR spectra of crude product.

The diastereomeric ratio of product was determined by ^1H NMR of crude product. The structure of major isomer with anti configuration was confirmed by single X-ray crystallography of the fluoro derivative (Table 1, entry 2) (Fig. 1).

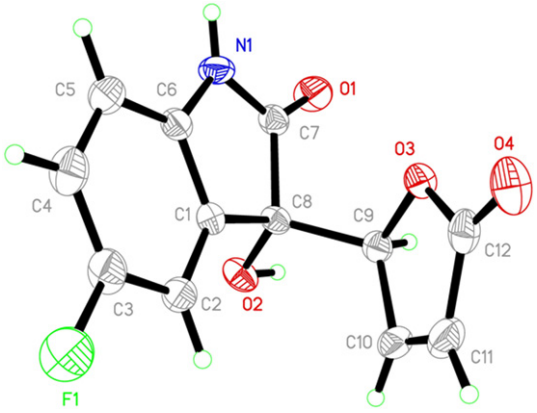


Fig. 1. ORTEP diagram of product **3b**.

3. Conclusion

In summary, we have demonstrated efficient vinylogous Mukaiyama aldol reaction (VMAR) of isatin with 2-(trimethylsilyloxy) furan in the presence of $La(OTf)_3$. The present method offers several advantages like high yields, good diastereoselectivity, highly catalytic (5 mol %) process, without any formation of by-products. Moreover, this method is applicable for various substituted isatins. This protocol may find utility in the synthesis of biologically active compounds. Development of other $La(OTf)_3$-catalyzed Mukaiyama aldol reactions and related mechanistic studies will be reported in due course.

4. Experimental section

4.1. General remarks

Melting points were measured on a BUCHI Melting Point machine. All reactions were conducted under an atmosphere of nitrogen (IOLAR Grade I). Progress of the reactions was monitored by TLC on Merck Silica Gel 60 F-254 precoated. Evaporation of solvents was performed at reduced pressure on a Buchi rotary evaporator. Column chromatography was carried out with silica gel grade 60–120 and 100–200 mesh. ^1H NMR spectra were recorded at 300 MHz and ^{13}C NMR spectra at 75 MHz in $CDCl_3$ or $CDCl_3$+DMSO-d_6. *J* values were recorded in hertz and abbreviations used were s—singlet, d—doublet, m—multiplet, and br—broad. Chemical shifts (δ) are reported relative to TMS (δ=0.0) as an internal standard. IR spectra were recorded on Thermo Nicolet FT/IR-5700. Mass spectral data were obtained using MS (ESI), HRMS data obtained using quadrupole time-of-flight (QTOF) mass spectrometer (QSTAR XL, Applied Biosystems MDS Sciex, Foster City, USA). One or more of the following methods were used for visualization: UV absorption by fluorescence quenching; iodine staining; anisaldehyde stain (ethanol (135 mL)/H_2SO_4 (5 mL)/AcOH (1.5 mL)/*p*-anisaldehyde 3.7 mL). Ethyl acetate and hexane were the common eluents used unless specified. Elemental analyses (C, H, N) were performed with a VARIO EL III elemental analyzers.

4.2. Typical procedure for the lanthanum triflate-catalyzed Mukaiyama aldol reaction

To a solution of isatin **1** (1 mmol) in THF (2 mL) was added La $(OTf)_3$ (5 mol %). The mixture was cooled to −78 °C and stirred at this temperature for 15 min. Then the solution of 2-(trimethylsilyloxy)furan **2** (1.5 mmol) in THF (1 mL) was added dropwise. The mixture was stirred at −78 °C for 2 h. Later the mixture was brought to 20 °C and stirred for additional 0.5–1.5 h (Table 1). The reaction was monitored by TLC. After the completion of the reaction it was diluted with tetrahydrofuran (2.0 mL) and quenched with 10% aqueous HCl (1.0 mL). The mixture was stirred for 0.25 h at room temperature, neutralized by the addition of a saturated aqueous $NaHCO_3$ solution, and extracted with ethyl acetate (3×5 mL). The combined organic layer washed with brine solution, dried over Na_2SO_4, and concentrated under reduced pressure (rotary evaporator). The crude products were purified by silica gel column chromatography (ethyl acetate/hexanes). The inseparable diastereomers *threo*/*erythro* ratio of the product was determined by ^1H NMR analysis of the crude reaction mixture.

*4.2.1. 3-Hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3a**; Table 1, entry 1).* R_f(40% EtOAc/hexanes) 0.33; White solid; mp: 192–194 °C; *threo*:*erythro* ratio: 93:7; ^1H NMR (300 MHz,

$CDCl_3$+DMSO-d_6): δ=5.27 (t, J=1.7 Hz, 1H), 6.18 (dd, J=3.7 Hz, J=1.8 Hz, 1H), 6.48 (s, 1H), 6.86 (m, 2H), 7.05 (d, J=7.3 Hz, 1H), 7.20 (td, J=6.6 Hz, J=1.1 Hz, 1H), 7.90 (dd, J=5.6 Hz, J=1.5 Hz, 1H), 10.27 (s, 1H) (*threo*); 5.33 (t, J=1.7 Hz, 1H), 6.00 (dd, J=3.7 Hz, J=1.8 Hz, 1H), 6.60 (s, 1H), 6.90 (m, 2H), 7.32 (d, J=7.3 Hz, 1H), 7.50 (m, 1H), 7.58 (m, 1H), 10.20 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=83.0, 84.3, 109.1, 120.2, 121.8, 124.3, 128.6, 128.8, 141.2, 152.5, 170.6, 175.5 (*threo*); 83.29, 85.07, 120.9, 121.2, 123.3, 124.9, 126.5, 129.7, 140.6, 152.0, 171.2, 174.3 (*erythro*) ppm. IR (KBr): ν_{max}=3482, 3201, 3097, 1764, 1720, 1618, 1466, 1199, 1168, 1064, 823, 756 cm^{-1}. MS(ESI): m/z=254 $[M+Na]^+$. HRMS: calculated for $C_{12}H_9NO_4Na$ 254.0429; found 254.0421. Anal. Calcd for $C_{12}H_9NO_4$: C, 62.33; H 3.92; N, 6.05. Found: C, 62.40; H, 3.93; N, 6.86.

*4.2.2. 5-Fluoro-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3b**; Table 1, entry 2).* R_f(40% EtOAc/hexanes) 0.37; White solid; mp: 184–186 °C; *threo*:*erythro* ratio: 95:5; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.22 (t, J=1.5 Hz, 1H), 6.24 (dd, J=3.9 Hz, J=1.7 Hz, 1H), 6.70 (d, J=1.5 Hz, 1H), 6.82 (td, J=4.3 Hz, J=2.4 Hz, 2H), 6.92 (dd, J=6.6 Hz, J=2.4 Hz, 1H), 7.92 (dd, J=4.7 Hz, J=1.1 Hz, 1H), 10.5 (s, 1H) (*threo*); 5.35 (m, 1H), 6.04 (dd, J=3.9 Hz, J=1.7 Hz, 1H), 6.77 (m, 1H), 7.10 (m, 1H), 7.60 (m, 1H), 7.84 (m, 2H), 10.45 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.8, 84.0, 111.8, 112.2, 114.8, 115.1, 121.9, 137.2, 152.2, 158.0, 170.5, 175.3 (*threo*); 83.0, 83.9, 111.0, 111.3, 114.7, 115.0, 121.3, 136.6, 151.8, 158.6, 171.0, 174.1 (*erythro*) ppm. IR (KBr): ν_{max}=3460, 3367, 3068, 2920, 1751, 1723, 1674, 1584, 1491, 1345, 1236, 1101, 848, 752, 612 cm^{-1}. MS(ESI): m/z=272 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8FNO_4Na$ 272.0335; found 272.0327. Anal. Calcd for $C_{12}H_8FNO_4$: C, 57.83; H 3.23; N, 5.62. Found: C, 57.84; H, 3.21; N, 5.65.

*4.2.3. 5-Chloro-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3c**, Table 1, entry 3).* R_f(40% EtOAc/hexanes) 0.36; Orange red solid; mp: 178–180 °C; *threo*:*erythro* ratio: 93:7; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ 5.22 (t, J=1.5 Hz, 1H), 6.25 (dt, J=4.1 Hz, J=1.7 Hz, 1H), 6.72 (d, J=4.7 Hz, 1H), 6.84 (dd, J=6.7 Hz, J=1.5 Hz, 1H), 7.05 (d, J=1.8 Hz, 1H), 7.19 (dt, J=4.7 Hz, J=1.5 Hz, 1H), 7.92 (dt, J=3.0 Hz, J=1.7 Hz, 1H), 10.52 (s, 1H) (*threo*); 5.34 (t, J=1.5 Hz, 1H), 6.07 (dt, J=4.1 Hz, J=1.7 Hz, 1H), 6.80 (m, 1H), 7.31 (d, J=1.8 Hz, 1H), 7.46 (m, 1H), 7.64 (dt, J=4.7 Hz, J=1.5 Hz, 1H), 7.80 (m, 1H), 10.42 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.6, 83.8, 110.3, 121.8, 124.3, 124.8, 126.8, 128.6, 139.8, 152.4, 170.5, 175.1 (*threo*); 82.4, 84.0, 110.7, 121.1, 123.4, 125.4, 126.5, 128.4, 139.3, 152.1, 171.0, 173.8 (*erythro*) ppm. IR (KBr): ν_{max}=3309, 3240, 2924, 1789, 1732, 1618, 1477, 1313, 1187, 1088, 826, 637, 577 cm^{-1}. MS(ESI) m/z=288 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8ClNO_4Na$ 288.0039; found: 288.0040. Anal. Calcd for $C_{12}H_8ClNO_4$: C, 54.25; H 3.03; N, 5.27. Found: C, 54.34; H, 3.01; N, 5.25.

*4.2.4. 5-Bromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3d**, Table 1, entry 4).* R_f(40% EtOAc/hexanes) 0.36; Yellow solid; mp: 198–200 °C; *threo*:*erythro* ratio: 94:6; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.25 (s, 1H), 6.25 (dd, J=4.7 Hz, J=1.1 Hz, 1H), 6.70 (s, 1H), 6.79 (d, J=8.3 Hz, 1H), 7.18 (d, J=1.1 Hz, 1H), 7.35 (dd, J=7.1 Hz, J=1.1 Hz, 1H), 7.94 (d, J=5.8 Hz, 1H), 10.52 (s, 1H) (*threo*); 5.35 (s, 1H), 6.07 (dd, J=4.7 Hz, J=1.1 Hz, 1H), 6.82 (s, 1H), 6.90 (m, 1H), 7.45 (m, 1H), 7.65 (d, J=7.1 Hz, 1H), 7.80 (m, 1H), 10.42 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.6, 82.8, 108.8, 110.6, 121.6, 121.8, 128.6, 131.3, 140.3, 152.1, 170.3, 174.8 (*threo*); 83.7, 84.1, 111.3, 112.1, 120.6, 121.1, 128.5, 131.2, 140.5, 151.9, 170.4, 175.3 (*erythro*) ppm. IR (KBr): ν_{max}=3320, 3216, 2925, 1745, 1719, 1615, 1475, 1314, 1191, 1168, 1089, 816, 632, 570 cm^{-1}. MS(ESI) m/z=332 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8BrNO_4Na$ 331.9534; found 331.9525. Anal. Calcd for $C_{12}H_8BrNO_4$: C, 46.47; H 2.60; N, 4.51. Found: C, 46.40; H, 2.63; N, 4.56.

*4.2.5. 3-Hydroxy-5-methyl-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3e**, Table 1, entry 5).* R_f(40% EtOAc/hexanes) 0.34; Light orange solid; mp: 201–203 °C; *threo*:*erythro* ratio: 77:23; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=2.28 (s, 3H), 5.24 (m, 1H), 6.20 (d, J=5.2 Hz, 1H), 6.5 (s, 1H), 6.72 (d, J=8.3 Hz, 1H), 6.89 (s, 1H), 7.00 (d, J=7.3 Hz, 1H), 7.9 (d, J=5.2 Hz, 1H), 10.22 (s, 1H) (*threo*); 2.3 (s, 3H), 5.3 (m, 1H), 6.01 (d, J=4.1 Hz, 1H), 6.6 (s, 1H), 6.7 (m, 1H), 7.12 (s, 1H), 7.60 (d, J=5.2 Hz, 1H), 7.88 (m, 1H), 10.18 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=19.6, 82.8, 84.0, 108.5, 121.4, 124.8, 125.2, 128.7, 128.8, 138.7, 152.2, 170.1, 175.1 (*threo*); 20.2, 83.9, 84.2, 108.8, 120.9, 123.9, 126.2, 128.5, 129.5, 138.9, 151.8, 169.9, 174.0 (*erythro*) ppm. IR (KBr): ν_{max}=3310, 3299, 2922, 1755, 1720, 1610, 1199, 1090, 636 cm^{-1}. MS(ESI) m/z=268 $[M+Na]^+$. HRMS: calculated for $C_{13}H_{11}NO_4Na$ 268.0574; found 268.0585. Anal. Calcd for $C_{13}H_{11}NO_4$: C, 63.67; H 4.52; N, 5.71. Found: C, 63.60; H, 4.53; N, 5.76.

*4.2.6. 3-Hydroxy-5-methoxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3f**, Table 1, entry 6).* R_f(40% EtOAc/hexanes) 0.33; Ash color solid; mp: 204–206 °C; *threo*:*erythro* ratio: 93:7; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=3.7 (s, 3H), 5.22 (s, 1H), 6.20 (d, J=4.3 Hz, 1H), 6.59 (s, 1H), 6.64 (s, 1H), 6.74 (m, 2H), 7.90 (d, J=5.1 Hz, 1H), 10.20 (s, 1H) (*threo*); 3.72 (s, 3H), 5.32 (s, 1H), 6.00 (d, J=4.3 Hz, 1H), 6.67 (s, 1H), 6.79 (m, 2H), 6.91 (s, 1H), 7.52 (d, J=5.1 Hz, 1H), 10.12 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=53.7, 82.1, 83.6, 108.6, 112.5, 121.2, 125.9, 133.9, 134.8, 152.1, 152.7, 169.7, 174.6 (*threo*); 53.8, 82.7, 84.7, 108.7, 112.8, 120.6, 127.3, 133.8, 135.3, 152.0, 153.3, 169.6, 174.7 (*erythro*) ppm. IR (KBr): ν_{max}=3410, 3312, 3082, 2926, 1794, 1753, 1605, 1488, 1205, 1165, 1092, 1029, 833, 636 cm^{-1}. MS(ESI) m/z=284 $[M+Na]^+$. HRMS: calculated for $C_{13}H_{11}NO_5Na$ 284.0534; found 284.0532. Anal. Calcd for $C_{13}H_{11}NO_5$: C, 59.77; H 4.24; N, 5.36. Found: C, 59.70; H, 4.23; N, 5.38.

*4.2.7. 3-Hydroxy-1-methyl-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3g**, Table 1, entry 7).* R_f(40% EtOAc/hexanes) 0.42; Orange solid; mp: 138–140 °C; *threo*:*erythro* ratio: 84:16; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=3.20 (s, 3H), 5.30 (t, J=1.7 Hz, 1H), 6.22 (dd, J=3.9 Hz, J=1.8 Hz, 1H), 6.69 (s, 1H), 6.90 (d, J=7.7 Hz, 1H), 6.97 (td, J=7.7 Hz, J=0.5 Hz, 1H), 7.12 (d, J=7.5 Hz, 1H), 7.32 (td, J=6.6 Hz, J=1.1 Hz, 1H), 7.94 (dd, J=4.3 Hz, J=1.1 Hz, 1H) (*threo*); 3.25 (s, 3H), 5.37 (t, J=1.7 Hz, 1H), 6.02 (dd, J=3.9 Hz, J=1.8 Hz, 1H), 6.77 (s, 1H), 6.85 (m, 1H), 7.55 (dd, J=4.3 Hz, J=1.1 Hz, 1H), 7.85 (m, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=24.8, 82.7, 84.0, 107.2, 120.8, 121.7, 123.9, 124.3, 128.9, 142.5, 152.4, 170.4, 173.6 (*threo*); 24.7, 82.9, 84.1, 121.1, 121.4, 123.0, 124.1, 125.8, 128.7, 142.4, 151.8, 170.9, 174.2 (*erythro*) ppm. IR (KBr): ν_{max}=3362, 3099, 2930, 1789, 1755, 1708, 1612, 1467, 1377, 1116, 1048, 755 cm^{-1}. MS(ESI) m/z=268 $[M+Na]^+$. HRMS: calculated for $C_{13}H_{11}NO_4Na$ 268.0574; found 268.0585. Anal. Calcd for $C_{13}H_{11}NO_4$: C, 63.67; H 4.52; N, 5.71. Found: C, 63.66; H, 4.50; N, 5.66.

*4.2.8. 1-Ethyl-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3h**, Table 1, entry 8).* R_f(40% EtOAc/hexanes) 0.40; Light orange solid; mp: 173–175 °C; *threo*:*erythro* ratio: 80:20; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=1.26 (t, J=6.8 Hz, 3H), 3.74 (q, J=6.8 Hz, 2H), 5.26 (s, 1H), 6.20 (m, 1H), 6.63 (d, J=4.8 Hz, 1H), 6.90 (m, 1H), 6.96 (t, J=7.8 Hz, 1H), 7.12 (d, J=7.8 Hz, 1H), 7.30 (t, J=7.8 Hz, 1H), 7.90 (d, J=4.8 Hz, 1H) (*threo*); 1.22 (t, J=6.8 Hz, 3H), 3.65 (q, J=6.8 Hz, 2H), 5.35 (s, 1H), 6.02 (m, 1H), 6.72 (d, J=4.8 Hz, 1H), 6.85 (m, 1H), 7.04 (t, J=7.8 Hz, 1H), 7.37 (d, J=7.8 Hz, 1H), 7.55 (m, 1H), 7.87 (d, J=4.8 Hz, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=11.3, 33.4, 83.0, 84.3, 120.7, 122.0, 121.4, 124.4, 128.8, 129.0, 141.9, 152.2, 170.3, 173.4 (*threo*); 11.3, 33.2, 82.8, 84.3, 120.5, 121.3, 123.5, 125.0, 128.6, 129.2, 141.7, 151.5, 170.4, 173.6

(*erythro*) ppm. IR (KBr): ν_{max}=3352, 3090, 2938, 1799, 1735, 1622, 1467, 1307, 1112, 1049, 756 cm^{-1}. MS(ESI) *m*/*z*=282 $[M+Na]^+$. HRMS: calculated for $C_{14}H_{13}NO_4Na$ 282.0742; found 282.0744. Anal. Calcd for $C_{14}H_{13}NO_4$: C, 64.85; H 5.05; N, 5.40. Found: C, 64.84; H, 5.03; N, 5.36.

*4.2.9. 3-Hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)-1-phenylindolin-2-one (**3i**, Table 1, entry 9).* R_f(40% EtOAc/hexanes) 0.43; Orange red color solid; mp: 168–170 °C; *threo*:*erythro* ratio: 65:35; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.4 (m, 1H), 6.28 (dd, *J*=3.9 Hz, *J*=1.8 Hz, 1H), 6.85 (t, *J*=7.7 Hz, 1H), 7.1 (t, *J*=7.5 Hz, 1H), 7.28 (t, *J*=7.7 Hz, 2H), 7.42–7.60 (m, 6H), 7.9 (dd, *J*=4.3 Hz, *J*=1.3 Hz, 1H) (*threo*); 5.45 (m, 1H), 6.10 (dd, *J*=3.9 Hz, *J*=1.8 Hz, 1H), 6.90 (m, 1H), 6.98 (m, 1H), 7,10 (m, 1H), 7.20 (m, 1H), 7.35 (m, 2H), 7.44-7.64 (m, 4H), 7.72 (dd, *J*=4.3 Hz, *J*=1.3, Hz, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.5, 83.7, 107.6, 120.9, 121.5, 124.1, 124.8, 126.5, 127.9, 128.3, 128.4, 132.3, 142.2, 152.0, 169.8, 172.8 (*threo*); 82.4, 83.5, 107.8, 123.1, 125.4, 125.8, 126.0, 126.9, 127.0, 127.6, 127.7, 132.1, 141.7, 151.8, 170.4, 171.4 (*erythro*) ppm. IR (KBr): ν_{max}=3410, 3078, 2923, 2853, 1759, 1725, 1608, 1500, 1461, 1383, 1163, 1088, 1047, 827, 757, 699 cm^{-1}. MS(ESI) *m*/*z*=330 $[M+Na]^+$. HRMS: calculated for $C_{18}H_{13}NO_4Na$ 330.0742; found 330.0736. Anal. Calcd for $C_{18}H_{13}NO_4$: C, 70.35; H 4.26; N, 4.55. Found: C, 70.40; H, 4.23; N, 4.56.

*4.2.10. 1-Benzyl-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3j**, Table 1, entry 10).* R_f(40% EtOAc/hexanes) 0.43; White solid; mp: 125–127 °C; *threo*:*erythro* ratio: 92:8; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=4.90 (d, *J*=15.7 Hz, 1H), 5.00 (d, *J*=15.7 Hz, 1H), 5.42 (s, 1H), 6.25 (d, *J*=5.2 Hz, 1H), 6.73 (d, *J*=8.3 Hz, 1H), 6.86 (s, 1H), 6.97 (t, *J*=7.3 Hz, 1H), 7.15 (d, *J*=7.3 Hz, 1H), 7.22 (t, *J*=7.3 Hz, 1H), 7.28 (m, 1H), 7.35 (m, 4H), 8.00 (d, *J*=5.2 Hz, 1H) (*threo*); 4.80 (d, *J*=15.7 Hz, 1H), 4.95 (m, 1H), 5.47 (s, 1H), 6.10 (d, *J*=5.2 Hz, 1H), 6.77 (m, 1H), 6.92 (s, 1H), 7.07 (d, *J*=7.3 Hz, 1H), 7.35 (m, 6H), 7.45 (m, 1H), 7.70 (d, *J*=5.2 Hz, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=41.9, 82.7, 83.9, 108.0, 120.7, 121.8, 124.1, 124.5, 125.7, 126.0, 127.2, 128.7, 134.0, 141.7, 152.2, 169.8, 173.8 (*threo*); 41.8, 83.6, 85.7, 107.8, 120.9, 121.4, 123.0. 127.3, 127.3, 128.5, 134.4, 141.2, 153.1, 170.5, 172.2, 173.4 (*erythro*) ppm. IR (KBr): ν_{max}=3357, 3081, 2928, 1759, 1702, 1610, 1463, 1377, 1264, 1165, 1105, 1047, 834, 746, 699 cm^{-1}. MS(ESI) *m*/*z*=344 $[M+Na]^+$. HRMS: calculated for $C_{19}H_{15}NO_4Na$ 344.0898; found 344.0886. Anal. Calcd for $C_{19}H_{15}NO_4$: C, 71.01; H 4.70; N, 4.35. Found: C, 71.00; H, 4.69; N, 4.36.

*4.2.11. 1-Benzyl-5-bromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl) indolin-2-one (**3k**, Table 1, entry 11).* R_f(40% EtOAc/hexanes) 0.44; Orange red solid; mp: 73–74 °C; *threo*:*erythro* ratio: 84:16; ^1H NMR (300 MHz, $CDCl_3$): δ=4.72 (d, *J*=15.6 Hz, 1H), 4.90 (d, *J*=15.6 Hz, 1H), 5.30 (s, 1H), 6.25 (d, *J*=4.8 Hz, 1H), 6.52 (d, *J*=8.7 Hz, 1H), 7.24 (d, *J*=7.8 Hz, 3H), 7.26–7.35 (m, 5H), 7.77 (d, *J*=5.8 Hz, 1H) (*threo*); 4.61 (d, *J*=15.6 Hz, 1H), 4.94 (d, *J*=15.6 Hz, 1H), 5.35 (s, 1H), 6.10 (d, *J*=4.8 Hz, 1H), 6.55 (d, *J*=8.7 Hz, 1H), 7.16 (d, *J*=7.8 Hz, 3H), 7.22 (m, 4H), 7.52 (d, *J*=5.8 Hz, 1H), 7.54 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ=43.9, 83.9, 84.9, 111.4, 115.6, 123.3, 124.1, 126.8, 127.8, 128.5, 128.8, 133.4, 134.1, 141.8, 151.9, 171.5, 174.8 (*threo*); 43.9, 84.1, 84.7, 111.3, 116.2, 123.6, 123.9, 127.0, 127.6, 127.9, 128.8, 133.2, 134.1, 141.4, 152.0, 171.9, 173.4 (*erythro*) ppm. IR (KBr): ν_{max}=3351, 3087, 2988, 1769, 1720, 1611, 1463, 1357, 1265, 1165, 1105, 1047, 834, 746, 699, 570 cm^{-1}. MS(ESI) *m*/*z*=422 $[M+Na]^+$. HRMS: calculated for $C_{19}H_{14}BrNO_4Na$ 422.0003; found 422.0006. Anal. Calcd for $C_{19}H_{14}BrNO_4$: C, 57.01; H 3.52; N, 3.49. Found: C, 57.00; H, 3.49; N, 3.48.

*4.2.12. 3-Hydroxy-5-nitro-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3l**, Table 1, entry 12).* R_f(40% EtOAc/hexanes) 0.30; Light yellow solid; mp: 234–236 °C; *threo*:*erythro* ratio: 84:16; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.30 (t, *J*=1.7 Hz, 1H), 6.28 (dd, *J*=3.7 Hz, *J*=1.8 Hz, 1H), 6.92 (s, 1H), 7.00 (m, 1H), 7.90 (d, *J*=2.0 Hz, 1H), 8.00 (dd, *J*=4.3 Hz, *J*=1.5 Hz, 1H), 8.17 (dd, *J*=6.4 Hz, *J*=2.2 Hz, 1H), 11.10 (s, 1H) (*threo*); 5.45 (t, *J*=1.7 Hz, 1H), 6.10 (dd, *J*=3.7 Hz, *J*=1.8 Hz, 1H), 6.97 (s, 1H), 7.00 (m, 1H), 7.72 (m, 1H), 8.20 (m, 1H), 8.25 (d, *J*=2.0 Hz, 1H), 11.00 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=83.2, 84.2, 109.7, 120.7, 122.9, 126.6, 141.6, 142.1, 148.7, 153.0, 170.7, 176.3 (*threo*); 83.2, 84.0, 109.5, 120.0, 121.9, 126.4, 141.5, 142.2, 148.4, 152.9, 170.2, 176.2 (*erythro*) ppm. IR (KBr): ν_{max}=3327, 3236, 3084, 2925, 2850, 1755, 1716, 1626, 1520, 1476, 1341, 1180, 1070, 835, 741, 683 cm^{-1}. MS(ESI) *m*/*z*=299 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8N_2O_6Na$ 299.0280; found 299.0294. Anal. Calcd for $C_{12}H_8N_2O_6$: C, 52.18; H 2.91; N, 10.14. Found: C, 52.14; H, 2.93; N, 10.16.

*4.2.13. 7-Bromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3m**, Table 1, entry 13).* R_f(40% EtOAc/hexanes) 0.35; Orange color solid; mp: 180–182 °C; *threo*:*erythro* ratio: 93:7; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.25 (s, 1H), 6.20 (d, *J*=5.5 Hz, 1H), 6.66 (s, 1H), 6.84 (t, *J*=7.3 Hz, 1H), 7.05 (d, *J*=7.3 Hz, 1H), 7.35 (d, *J*=8.2 Hz, 1H), 7.90 (d, *J*=5.5 Hz, 1H), 10.44 (s, 1H) (*threo*); 5.31 (s, 1H), 6.05 (d, *J*=5.5 Hz, 1H), 6.77 (s, 1H), 6.90 (t, *J*=7.3 Hz, 1H), 7.30 (d, *J*=7.3 Hz, 1H), 7.60 (d, *J*=5.5 Hz, 1H), 7.64 (m, 1H), 10.37 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.7, 83.4, 100.9, 121.0, 121.4, 122.8, 126.8, 131.1, 140.3, 151.9, 169.6, 174.6 (*threo*); 82.0, 83.5, 100.7, 120.7, 121.7, 121.9, 128.4, 131.1, 139.8, 151.6, 170.1, 173.3 (*erythro*) ppm. IR (KBr): ν_{max}=3330, 3216, 2952, 1744, 1708, 1611, 1477, 1311, 1190, 1166, 1099, 810, 622, 571 cm^{-1}. MS(ESI) *m*/*z*=332 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8BrNO_4Na$ 331.9534; found 331.9522. Anal. Calcd for $C_{12}H_8BrNO_4$: C, 46.47; H 2.60; N, 4.51. Found: C, 46.36; H, 2.76; N, 4.59.

*4.2.14. 7-Chloro-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3n**, Table 1, entry 14).* R_f(40% EtOAc/hexanes) 0.36; Yellow solid; mp: 174–176 °C; *threo*:*erythro* ratio: 91:9; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.25 (s, 1H), 6.25 (dd, *J*=3.9 Hz, *J*=1.8 Hz, 1H), 6.75 (m, 1H), 6.90 (t, *J*=7.5 Hz, 1H), 7.0 (d, *J*=7.1 Hz, 1H), 7.20 (d, *J*=8.1 Hz, 1H), 7.95 (d, *J*=5.8 Hz, 1H), 10.7 (s, 1H) (*threo*); 5.35 (s, 1H), 6.05 (dd, *J*=3.9 Hz, *J*=1.8 Hz, 1H), 6.85 (m, 1H), 6.95 (m, 2H), 7.25 (d, *J*=8.1 Hz, 1H), 7.60 (m, 1H), 10.55 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=82.6, 83.4, 100.7, 121.1, 121.5, 122.7, 126.8, 131.0, 140.1, 151.8, 169.5, 174.7 (*threo*); 82.1, 83.4, 100.6, 120.7, 121.4, 121.8, 128.5, 131.0, 139.9, 151.7, 170.12 173.4 (*erythro*) ppm. IR (KBr): ν_{max}=3456, 3300, 2922, 1788, 1732, 1619, 1478, 1311, 1187, 1080, 826, 636, 579 cm^{-1}. MS(ESI) *m*/*z*=288 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8ClNO_4Na$ 288.0039; found 288.0043. Anal. Calcd for $C_{12}H_8ClNO_4$: C, 54.25; H 3.03; N, 5.27. Found: C, 54.24; H, 3.01; N, 5.26.

*4.2.15. 3-Hydroxy-7-methyl-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3o**, Table 1, entry 15).* R_f(40% EtOAc/hexanes) 0.33; Light orange color solid; mp: 199–201 °C; *threo*:*erythro* ratio: 86:14; ^1H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=2.25 (s, 3H), 5.27 (s, 1H), 6.21 (d, *J*=5.5 Hz, 1H), 6.53 (s, 1H), 6.82 (t, *J*=7.1 Hz, 1H), 7.02 (d, *J*=7.1 Hz, 1H), 7.15 (d, *J*=7.9 Hz, 1H), 7.93 (d, *J*=5.5 Hz, 1H), 10.46 (s, 1H) (*threo*); 2.23 (s, 3H), 5.34 (s, 1H), 6.05 (d, *J*=5.5 Hz, 1H), 6.63 (s, 1H), 6.90 (t, *J*=7.1 Hz, 1H), 7.08 (d, *J*=7.1 Hz, 1H), 7.16 (d, *J*=7.9 Hz, 1H), 7.60 (d, *J*=5.5 Hz, 1H), 10.40 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=15.2, 83.0, 84.3, 118.3, 120.2, 121.6, 121.7, 124.5, 130.2, 139.6, 152.7, 170.7, 175.9 (*threo*); 14.9, 82.7, 84.1, 118.4, 120.6, 120.8, 121.1, 123.4, 130.0, 139.1, 152.2, 171.3, 174.7 (*erythro*) ppm. IR (KBr): ν_{max}=3361, 3273, 3090, 2931, 1789, 1755, 1708, 1611, 1467, 1377, 1112, 1048, 755 cm^{-1}. MS(ESI) *m*/*z*=268 $[M+Na]^+$. HRMS: calculated for $C_{13}H_{11}NO_4Na$ 268.0574; found 268.0585. Anal. Calcd for $C_{13}H_{11}NO_4$: C, 63.67; H 4.52; N, 5.71. Found: C, 63.62; H, 4.50; N, 5.74.

*4.2.16. 5,7-Dibromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3p**, Table 1, entry 16).* R_f(40% EtOAc/hexanes) 0.39; Ash color solid; mp: 225–227 °C; *threo*:*erythro* ratio: 84:16; ^1H NMR

(300 MHz, $CDCl_3$+DMSO-d_6): δ=5.23 (s, 1H), 6.25 (d, J=5.5 Hz, 1H), 6.80 (s, 1H), 7.19 (s, 1H), 7.52 (s, 1H), 7.90 (d, J=5.5 Hz, 1H), 10.70 (s, 1H) (*threo*); 5.32 (s, 1H), 6.10 (d, J=5.5 Hz, 1H), 6.90 (s, 1H), 7.45 (s, 1H), 7.64 (s, 1H), 7.66 (d, J=5.5 Hz, 1H), 10.6 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=81.8, 82.7, 101.3, 111.6, 121.2, 125.6, 128.5, 132.7, 139.6, 151.6, 169.4, 173.9 (*threo*); 81.5, 82.4, 101.2, 112.3, 120.4, 124.8, 130.2, 132.5, 139.2, 151.7, 169.8, 172.5 (*erythro*). IR (KBr): ν_{max}=3391, 3294, 3092, 1749, 1726, 1608, 1573, 1429, 1326, 1170, 1107, 930, 818, 683, 590 cm^{-1}. MS(ESI) m/z=411 $[M+Na]^+$. HRMS: calculated for $C_{12}H_7Br_2NO_4Na$ 409.8639; found 409.8630. Anal. Calcd for $C_{12}H_7Br_2NO_4$: C, 37.05; H 1.81; N, 3.60. Found: C, 37.01; H, 1.83; N, 3.66.

*4.2.17. 6-Bromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3q**, Table 1, entry 17).* R_f(40% EtOAc/hexanes) 0.37; White solid; mp: 201–203 °C; *threo*:*erythro* ratio: 84:16; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=4.94 (s, 1H), 6.65 (s, 1H), 6.80 (d, J=7.5 Hz, 1H), 6.95–7.15 (m, 3H), 7.95 (s, 1H), 10.65 (s, 1H) (*threo*); 4.90 (s, 1H), 6.70 (s, 1H), 6.95–7.10 (m, 4H), 7.92 (s, 1H), 10.56 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=69.8, 74.3, 108.6, 118.5, 125.0, 126.3, 130.4, 144.0, 148.0, 150.4, 169.5, 174.6 (*threo*); 72.6, 75.3, 112.6, 121.2, 123.8, 124.9, 130.8, 144.1, 148.2, 150.3, 169.4, 175.3 (*erythro*) ppm. IR (KBr): ν_{max}=3333, 3229, 2950, 1740, 1706, 1611, 1457, 1310, 1199, 1166, 1100, 810, 639, 571 cm^{-1}. MS (ESI) m/z=332 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8BrNO_4Na$ 331.9534; found 331.9535. Anal. Calcd for $C_{12}H_8BrNO_4$: C, 46.47; H 2.60; N, 4.51. Found: C, 46.38; H, 2.71; N, 4.49.

*4.2.18. 4,6-Dibromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3r**, Table 1, entry 18).* R_f(40% EtOAc/hexanes) 0.38; White solid; mp: 198–200 °C; *threo*:*erythro* ratio: 70:30; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.40 (m, 1H), 6.10 (dd, J=3.7 Hz, J=2.0 Hz, 1H), 6.99 (d, J=3.3 Hz, 2H), 7.20 (d, J=1.3 Hz, 1H), 7.78 (dd, J=4.3 Hz, J=1.3 Hz, 1H), 10.73 (s, 1H) (*threo*); 5.80 (s, 1H), 6.14 (dd, J=3.7 Hz, J=2.0 Hz, 1H), 7.00 (d, J=3.3 Hz, 2H), 7.28 (d, J=1.3 Hz, 1H), 7.97 (dd, J=4.3 Hz, J=1.3 Hz, 1H), 10.63 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=80.8, 83.7, 112.1, 119.2, 121.3, 123.2, 123.1, 127.6, 144.8, 152.8, 170.8, 175.0 (*threo*); 80.9, 83.8, 111.9, 118.7, 120.4, 123.0, 125.3, 127.2, 145.0, 153.9, 171.1, 173.2 (*erythro*) ppm. IR (KBr): ν_{max}=3331, 3299, 3090, 1779, 1724, 1600, 1533, 1420, 1356, 1170, 1170, 939, 818, 686, 590 cm^{-1}. MS(ESI) m/z=411 $[M+Na]^+$. HRMS: calculated for $C_{12}H_7Br_2NO_4Na$ 409.8639; found 409.8638. Anal. Calcd for $C_{12}H_7Br_2NO_4$: C, 37.05; H 1.81; N, 3.60. Found: C, 37.04; H, 1.83; N, 3.58.

*4.2.19. 4-Bromo-3-hydroxy-3-(5-oxo-2,5-dihydrofuran-2-yl)indolin-2-one (**3s**, Table 1, entry 19).* R_f(40% EtOAc/hexanes) 0.36; White solid; mp: 171–173 °C; *threo*:*erythro* ratio: 65:35; ^{1}H NMR (300 MHz, $CDCl_3$+DMSO-d_6): δ=5.20 (s, 1H), 6.20 (m, 1H), 6.60 (s, 1H), 6.92–7.05 (m, 2H), 7.10 (d, J=7.3 Hz, 1H), 7.89 (s, 1H), 10.4 (s, 1H) (*threo*); 5.30 (s, 1H), 6.00 (m, 1H), 6.65 (s, 1H), 6.92–7.05 (m, 2H), 7.20 (d, J=7.3 Hz, 1H), 8.00 (m, 1H), 10.35 (s, 1H) (*erythro*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$+DMSO-d_6): δ=83.8, 85.1, 113.6, 123.1, 123.5, 124.3, 126.5, 131.2, 143.3, 152.6, 171.6, 176.5 (*threo*); 81.7, 85.9, 113.3, 122.7, 123.2, 124.5, 126.1, 131.3, 142.7, 152.0, 172.1, 175.3 (*erythro*) ppm. IR (KBr): ν_{max}=3330, 3298, 2955, 1769, 1732, 1621, 1447, 1391, 1199, 1156, 1099, 810, 622, 570 cm^{-1}. MS(ESI) m/z=332 $[M+Na]^+$. HRMS: calculated for $C_{12}H_8BrNO_4Na$ 331.9534; found 331.9521. Anal. Calcd for $C_{12}H_8BrNO_4$: C, 46.47; H 2.60; N, 4.51. Found: C, 46.48; H, 2.61; N, 4.49.

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Supplementary data

Supplementary data related to this article can be found online at doi:10.1016/j.tet.2011.02.033.

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