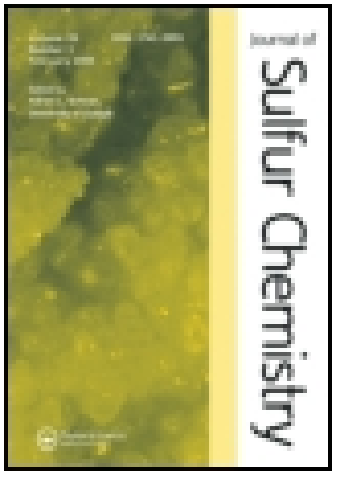

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Catalytic application of *N*,2-dibromo-6-chloro-3,4-dihydro-2*H*-benzo[e][1,2,4]thiadiazine-7-sulfonamide 1,1-dioxide on the synthesis of 1-carbamato-alkyl-2-naphthols and 1-thioamido-alkyl-2-naphthols

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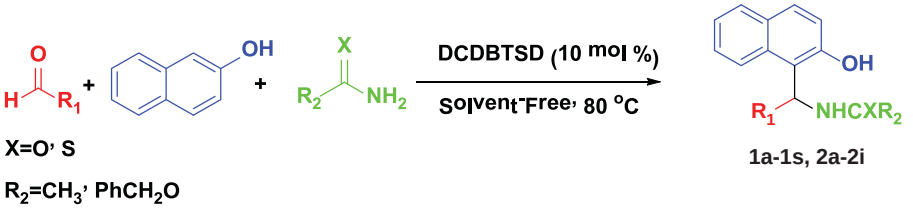
A novel *N*-bromo sulfonamide reagent, namely *N*, 2-dibromo-6-chloro-3,4-dihydro-2*H*-benzo[e][1,2,4]thiadiazine-7-sulfonamide 1,1-dioxide, is prepared and employed as a new and highly efficient catalyst for the preparation of 1-carbamato-alkyl-2-naphthol and 1-thioamido-alkyl-2-naphthol derivatives.



Keywords: *N*-halo reagent; benzyl carbamate; thioacetamide; aldehyde; 1-carbamato-alkyl-2-naphthol; 1-thioamido-alkyl-2-naphthol

1. Introduction

Multicomponent reactions (MCRs) are generally defined as reactions where more than two starting materials react to give a product, unifying essentially all of the atoms of the educts.[1,2] MCRs play a significant role in combinatorial chemistry due to the ability to synthesize target compounds with greater efficiency and atom economy by forming structural complexity in a single step from three or more reactants. Also, MCRs offer some advantages of simplicity and synthetic efficiency over conventional chemical reactions and lead to the preparation of important heterocyclic rings.[2–4]

1-Amido-alkyl-2-naphthols and 1-thiomido-alkyl-2-naphthols are important compounds which can be converted to biologically useful 1-aminoalkyl-2-naphthols *via* hydrolysis of amidic groups. [5] Based on their wide biological activities, bradycardiac and hypotensive properties of 1-aminoalkyl-2-naphthols have been examined.[5,6] 1-Amido-alkyl-2-naphthols can be also

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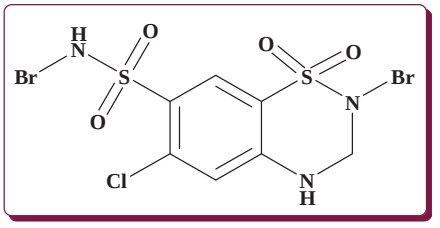
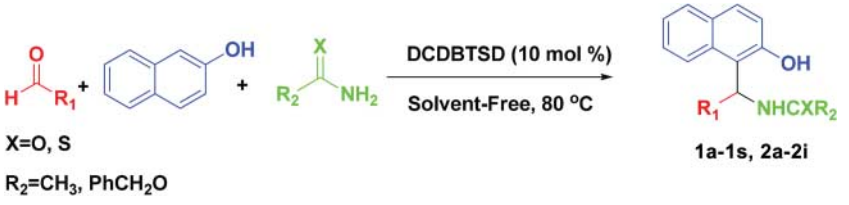


Scheme 1. The structure of DCDBTSD.



Scheme 2. The synthesis of 1-carbamato-alkyl-2-naphthols and 1-thioacetamido-alkyl-2-naphthols.

converted to 1,3-oxazine derivatives.[7] 1,3-Oxazines exhibit several pharmaceutical properties including antibiotic,[8] antitumor [9] and analgesic activities.[10]

Recently, several synthetic protocols have been reported for the synthesis of 1-amido-alkyl-2-naphthols and 1-thioamido-alkyl-2-naphtols by multicomponent condensation reactions using [Dsim]HSO_4,[11] TCCA and DCDMH,[12] Trityl chloride,[13] *p*-TSA,[14] H_2SO_4/SiO_2 [15] and $Fe(HSO_4)_3$.[16] Because of the importance of these compounds, the introduction of a milder, faster and more eco-friendly method accompanied with higher yields is still needed.

An enormous group of compounds commonly called *N*-halo reagents are utillized as potentially reactive intermediates. These compounds are widely used in organic transformations. Some specific features of *N*-halo reagents including the high activity of the *N*−X bond and the various modes of splitting of this bond cause their wide application in organic synthesis.[17] Due to significant features of *N*-halo reagents, a novel and effective *N*-halo reagent was synthesized using procedures from prior investigation of this compound class,[18–21] namely *N*,2-dibromo-6-chloro-3,4-dihydro-2*H*-benzo[e][1,2,4]thiadiazine-7-sulfonamide 1,1-dioxide (DCDBTSD) (Scheme 1).[22] DCDBTSD has been used as an applicable and suitable catalyst for the synthesis of 1-amido-alkyl (carbamato/thioacetamido)-2-naphthol *via* one-pot three-component condensation reaction of arylaldehydes, 2-naphthol and benzyl carbamate/thioacetamide at 80°C under solvent-free conditions (Scheme 2).

2. Results and discussion

The structure of DCDBTSD was identified by IR, ^{1}H and ^{13}C NMR, X-ray diffraction as well as mass spectra. The IR spectrum of the catalyst and hydrochlorothiazide is displayed in Figure S1. Comparison of IR spectra of hydrochloroyhiazide and DCDBTSD demonstrated that two sharp peaks for the *N*-H groups of the hydrochlorothiazide at 3363 and 3169 cm^{-1} were eliminated in DCDBTSD. These differences provide evidence that the two hydrogens of the *N*-H groups in hydrochlorothizide were replaced with two *N*-Br groups in DCDBTSD (Figure S1) (see supplementary data, http://dx.doi.org/ doi:10.1080/17415993.2015.1028938).

In another study, thermal gravimetric (TG) and derivative thermal gravimetric (DTG) analysis of DCDBTSD were investigated in the range of 30–618°C, with a temperature rate increase of

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10°C min^{-1} in a nitrogen atmosphere. The corresponding diagrams are shown in Figure S2. In the TG pattern of DCDBTSD multistage decomposition is observed. Therefore, DCDBTSD can be used as a catalyst below 200°C, but decomposes above 255°C (Figure S2) (see supplementary data, http://dx.doi.org/ doi:10.1080/17415993.2015.1028938).

After full characterization of DCDBTSD, its catalytic activity for the preparation of 1-carbamato-alkyl-2-naphthol and 1-thioacetamido-alkyl-2-naphthol was examined.

Initially, to optimize the reaction conditions (amount of catalyst and temperature), as a model reaction, the condensation of 2-naphthol (1 mmol), benzaldehyde (1 mmol) and benzyl carbamate (1.1 mmol) was studied in the presence of different amounts of DCDBTSD, over a range of 70–90°C under solvent-free conditions. The results are summarized in Table 1.

Table 1. Optimization of the reaction conditions for the synthesis of 1-carbamato-alkyl-2-naphthol.

Entry	Catalyst (mol%)	Temperature (°C)	Time(min)	Yield[a] (%)
1	No catalyst	80	180	–
2	Hydrochlorothiazide	80	180	–
3	5	80	4	77
4	10	80	2	86
5	15	80	2	86
6	10	70	5	80
7	10	90	2	85

[a]Isolated yield.

Table 2. Preparation of 1-carbamato-alkyl-2-naphthols and 1-thioacetamido-alkyl-2-naphthols using DCDBTSD at 80°C.

				Mp. °C	
Entry	R^1/R^2	X	Time (min)/yield[a] (%)	Found	Ref.
1a	C_6H_5/ $PhCH_2O$	O	10/86	185–187	[12]
1b	2-$NO_2C_6H_4/PhCH_2O$	O	4/85	200–202	[12]
1c	3-$NO_2C_6H_4/PhCH_2O$	O	3/84	201–202	[12]
1d	4-$NO_2C_6H_4/PhCH_2O$	O	2/88	203–205	[12]
1e	2-$ClC_6H_4/PhCH_2O$	O	2/83	210–211	[12]
1f	3-$ClC_6H_4/PhCH_2O$	O	3/85	185–187	[12]
1g	4-$ClC_6H_4/PhCH_2O$	O	4/86	180–181	[12]
1h	2,3-di$ClC_6H_3/PhCH_2O$	O	2/93	218–220	–
1i	2,4-di$ClC_6H_3/PhCH_2O$	O	2/92	208–210	[23]
1j	2-$FC_6H_4/PhCH_2O$	O	4/87	188–189	–
1k	3-$FC_6H_4/PhCH_2O$	O	6/80	208–210	–
1l	4-$FC_6H_4/PhCH_2O$	O	3/80	209–210	[12]
1m	2-$BrC_6H_4/PhCH_2O$	O	2/83	220–221	–
1n	3-$BrC_6H_4/PhCH_2O$	O	3/90	183–185	–
1o	4-$BrC_6H_4/PhCH_2O$	O	3/77	180–181	[12]
1p	1-$C_{10}H_7/PhCH_2O$	O	6/79	214–216	[12]
1q	2-$C_{10}H_7/PhCH_2O$	O	4/80	210–211	–
1r	4-$CH_3C_6H_4/PhCH_2O$	O	11/65	154–155	[12]
1s	2-$OCH_3C_6H_4/PhCH_2O$	O	10/79	202–203	[12]
2a	C_6H_5/Me	S	25/80	185–187	[13]
2b	3-$NO_2C_6H_4$/Me	S	30/78	190–191	–
2c	3-ClC_6H_4/Me	S	20/83	256–258	–
2d	4-ClC_6H_4/Me	S	20/84	243–245	[16]
2e	2,4-diClC_6H_3/Me	S	20/88	219–221	–
2f	4-FC_6H_4/Me	S	30/83	203–205	–
2g	4-BrC_6H_4/Me	S	40/77	195–196	–
2h	4-$CH_3C_6H_4$/Me	S	35/75	185–187	–
2i	4-$OCH_3C_6H_4$/Me	S	45/60	181–182	[16]

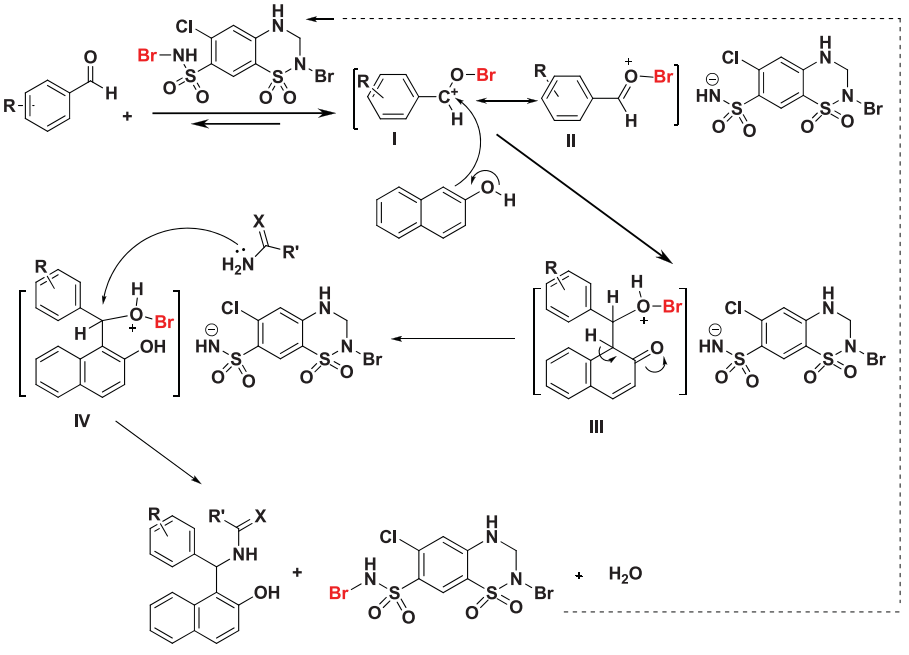
[a]Yield of the purified product.

As it is shown in Table 1, higher yield and shorter reaction time are achieved using 10 mol% of catalysts at 80°C under solvent-free conditions. No improvement in the reaction results was observed by increasing the quantities of the catalyst and the temperature. The solvent-free condensation was also examined at 80°C without catalyst and with hydrochlorothiazide and no reaction was observed even after long reaction time (3 h).

To evaluate the efficiency of the organic catalyst in the synthesis of 1-carbamato-alkyl-2-naphthols and 1-thioacetamido-alkyl-2-naphthols, the reaction of 2-naphthol and benzyl carbamate or thioacetamide with structurally different aromatic aldehydes in the presence of DCDBTSD as a catalyst was investigated under optimized conditions. The obtained results are described in Table 2. As it can be observed in Table 2, several aromatic aldehydes including electron-releasing- and electron-withdrawing substituents and halogens on the aromatic ring of aldehydes were successfully reacted and produced corresponding products in good to high yields and in relatively short reaction times.

Based on published reports [12,24,25] and the suggested mechanism (Scheme 3), the reaction is catalyzed by *in situ* generated Br^+ from the DCDBTSD. Therefore, in the catalytic process the aldehyde abstracts Br^+ from DCDBTSD to generate intermediates **I** and **II** as activated forms of the aldehyde. These complexes function as activated carbonyl compounds and react with 2-naphthol to give intermediate **III**, which is converted to **IV** after tautomerization. Finally, by the reaction of benzyl carbamate or thioacetamide with **IV** and the elimination of one molecule H_2O, the desired product is obtained and the catalyst is generated.

To demonstrate the formation of intermediates **I** and **II**, benzaldehyde was reacted with catalyst at 80°C, and then IR and UV spectra of the carbonyl functional group in the reaction mixture was compared with benzaldehyde. The observed decrease in the IR (nujol): ν (cm^{-1}) of C=O benzaldehyde from 1704 to 1692 in the reaction mixture is indicative of the decreased nature of the double bond and formation of an activated carbonyl (Figure S4). Also, the IR spectrum of this complex is revealed by two peaks sharp at 3380 and 3253 cm^{-1}. From these results, the



Scheme 3. The plausible mechanism for the synthesis of 1-amido-alkyl (carbamato-alkyl or thioamido-alkyl)-2-naphthols catalyzed by DCDBTSD.

proposed mechanism is confirmed and the catalyst is completely recovered during the reaction (Figure S3) (see supplementary data, http://dx.doi.org/ doi:10.1080/17415993.2015.1028938).

In another evidence, to demonstrate the formation of intermediates **I** and **II**, UV spectra of benzaldehyde, catalyst and the mixture of benzaldehyde and catalyst were studied. The maximum absorption of benzaldehyde and catalyst appeared at 289 and 333 nm, respectively. But λ_{max} of the complex of benzaldehyde with Br^+, which was formed by the reaction of DCDBTSD with benzaldehyde, was observed at 282 nm (Figure S4) (see supplementary data, http://dx.doi.org/ doi:10.1080/17415993.2015.1028938).

3. Conclusions

In conclusion, this paper offers a clean, efficient and simple procedure for preparation of 1-carbamato-alkyl-2-naphthol and 1-thioacetamido-alkyl-2-naphthol derivatives *via* one-pot, three-component condensation of arylaldehyde and 2-naphthol with amide (benzyl carbamate or thioacetamide) using DCDBTSD as new, effective and homogenous organic catalysts at 80°C under the solvent-free and neutral conditions.

4. Experimental

4.1. *General*

Chemicals were purchased from Merck and Aldrich Chemical Companies and used without further purification. All yields refer to isolated products after purification. The reactions were monitored by thin-layer chromatography (TLC) carried out on silica plates. The products were characterized by comparison of their physical data with those of known samples or by their spectral data. Infrared (IR) spectra were recorded on a Shimadzu IR 470 spectrophotometer. The ^{1}H NMR (400 or 90 MHz) and ^{13}C NMR (100 Hz or 22.5 MHz) were run on a Bruker AVANCE-DRX. Mass spectra were recorded on an Agilent technologies (HP) 5973 network mass-selective detector (MSD) operating at an ionization potential of 70 eV. Melting points were determined in open capillaries with a Stuart Scientific melting point apparatus. TLC was performed on Silica-gel polygram SILG/UV 254 plates. TG and DTG were analyzed by a Perkin Elmer (Model: Pyris 1) (30–618°C, temperature increase rate of 10°C min^{-1}, nitrogen atmosphere).

4.2. *General procedure for the synthesis of DCDBTSD*

A solution of sodium hydroxide (6 mol Lit^{-1}, 1 mL) was added dropwise to a stirring round-bottomed flask (50 mL) containing hydrochlorothizide (0.6 g, 2 mmol) in distillated water (2 mL) over a period of 10 min at room temperature. After the addition was completed, the reaction mixture was stirred for 20 min. Then, to the stirring solution of hydrochlorothiazide, bromine (0.16 mL, 3 mmol) was slowly added over a period of 15 min at 0°C. The insoluble brominated catalyst was removed by filtration and washed with H_2O (10 mL) to give DCDBTSD in 90% yield (0.82 g).[22]

4.2.1. *N,2-dibromo-6-chloro-3,4-dihydro-2H-benzo[e][1,2,4]thiadiazine-7-sulfonamide 1,1-dioxide*

Yield 90%; White solid; 255 °C;^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 4.70 (s, 2H), 6.85 (s, 1H), 7.20 (s, 1H), 7.88 (s, 1H), 8.07 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 55.08,

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107.26, 110.65, 122.57, 127.55, 136.34, 142.14; IR (KBr) ν/cm^{-1}3377, 3232, 1579, 1495, 1362, 1308, 1244, 1147, 1058, 1025, 794, 716, 675, 579, 545;MS *m/z* observed: 455 (M^+).

4.3. *General procedure for the synthesis of 1-carbamato-alkyl-2-naphtols using DCDBTSD (compound 1a–1s)*

A mixture of 2-naphthols (0.288g, 2 mmol), aldehydes (2 mmol), benzylcarbamate (0.320 g, 2.2 mmol) and DCDBTSD (0.091 g, 10 mol%) in a 10 mL round-bottomed flask connected to a reflux condenser, was stirred in oil bath (80°C) for the appropriate time (Table 2). After completion of the reaction, as monitored by TLC, the reaction mixture was cooled to room temperature and recrystallized from ethanol (95%).

4.3.1. *Benzyl (2,3-dichlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**1h**)*

Yield 93%; white solid; mp 218–220°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.02 (2H), 6.84 (d, J = 8 Hz, 1H); 7.09 (d, J = 7.2 Hz, 2H), 7.26–7.31 (6H), 7.39 (d, J = 8 Hz, 1H), 7.47 (d, J = 8 Hz, 1H), 7.51 (d, J = 8 Hz, 1H), 7.72 (d, J = 8 Hz, 1H), 7.76 (d, J = 8 Hz, 1H), 7.96 (d, J = 8 Hz, 1H), 8.17 (d, J = 8 Hz, 1H), 9.92 (s, OH, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 52.04, 67.04, 117.74, 120.07, 124.03, 124.26, 128.12, 129.04, 129.33, 129.90, 130.28, 131.38, 134.20, 138.67, 143.91, 155.17, 157.71; IR (KBr) ν/cm^{-1}3414, 3295,1702, 1685, 1629, 1515, 1451, 1338, 1271, 1050, 781, 744, 720, 558; MS *m/z* observed:451 (M^+).

4.3.2. *Benzyl (2-fluorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**1j**)*

Yield 87%; white solid; mp 188–189°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.02 (s, 2H), 7.01–7.15 (b, 5H), 7.26–7.38 (b, 6H), 7.49 (2H), 7.71–7.77 (2H), 7.92 (s, 1H), 8.04 (s, 1H), 10.18 (s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 47.62, 67.23, 116.62, 116.83, 118.86, 119.98, 124.12, 125.41, 128.07, 129.24, 129.44, 129.94, 130.19, 130.34, 130.66, 131.16, 133.55, 138.49, 154.72, 157.24, 161.44; IR (KBr) ν/cm^{-1} 3427, 3279, 1689, 1630, 1525, 1435, 1342, 1271, 1043, 935, 86, 771, 747; MS *m/z* observed:401 (M^+).

4.3.3. *Benzyl (3-fluorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**1k**)*

Yield 80%, white solid; mp 208–210°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.06 (s, 2H), 6.88 (s, 1H), 7.06 (d, J = 8.4 Hz, 2H), 7.25–7.33 (b, 10H), 7.78 (d, J = 8.4 Hz, 2H), 7.90 (s, 2H), 10.23 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 51.64, 67.42, 116.33, 116.54, 120.04, 120.15, 124.24, 128.30, 129.39, 129.45, 129.52, 129.60, 130.26, 131.14, 133.56, 138.46, 140.00, 154.53, 157.70, 161.32; IR (KBr) ν/cm^{-1} 3425, 3250, 1677, 1506, 1439, 1327, 1218, 1065, 943, 704; MS *m/z* observed:401 (M^+).

4.3.4. *Benzyl (2-bromophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate (**1m**)*

Yield 83%, white solid; m p 220–221 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.00 (s, 2H), 6.79 (s, 1H), 7.12 (s, 1H), 7.28–7.43 (b, 10H), 7.52 (1H), 7.75 (2H), 8.00 (2H), 9.95 (s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 53.65, 66.89, 118.42, 120.09, 123.97, 124.46, 127.93, 128.68, 128.91, 129.24, 129.84, 130.20, 130.48, 131.15, 131.71, 134.27, 155.07, 157.11; IR (KBr) ν/cm^{-1} 3422, 3170, 1701, 1627, 1516, 1435, 1334, 1275, 1247, 1046, 753, 731, 527; MS *m/z* observed:461 (M^+).

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4.3.5. *Benzyl (2-hydroxynaphthalen-1-yl)(naphthalen-2-yl)methylcarbamate (**1q**)*

Yield 80%; white solid; mp 210–211°C ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.05 (d, J = 12.4 Hz, 1H), 5.12 (d, J = 12.8 Hz, 1H), 7.06 (d, J = 5.6 Hz, 1H), 7.23 (d, J = 8, 2H), 7.32–7.43 (m, 10H), 7.78 (b, 5H), 7.97 (b, 2H), 10.18 (s, OH, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 52.16, 67.38, 120.05, 120.21, 124.19, 124.71, 125.48, 128.99, 129.26, 129.40, 129.98, 130.23, 131.15, 133.43, 133.73, 134.30, 138.57, 141.56, 154.65, 157.76; IR (KBr) ν/cm^{-1} 3413, 3169, 1676, 1583, 1519, 1437, 1332, 1272, 1051, 938, 814, 732, 601, 473; MS *m/z* observed:433 (M^+).

4.4. *General procedure for the synthesis of 1-thioamido-alkyl-2-naphtols using DCDBTSD (compound 2a–2i)*

A mixture of 2-naphthols (0.288g, 2 mmol), aldehydes (2 mmol), thioacetamide (0.180 g, 2.4 mmol) and DCDBTSD (0.091 g, 10 mol%) in a 10 mL round-bottomed flask connected to a reflux condenser, was stirred in oil bath (80°C) for the appropriate time (Table 2). After completion of the reaction, as monitored by TLC, the crude products were purified by plate chromatography on silica gel eluted with EtOAc/*n*-hexane.

4.4.1. *N-((3-Chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl) thioacetamide (**2c**)*

Yield 83%; white solid; mp 255–258°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 1.97 (s, 3H, CH_3), 7.04–7.10 (m, 2H), 7.17–7.29 (m, 6H), 7.37–7.41 (m, 1H), 7.76–7.82 (m, 2H), 8.51 (d, J = 8 Hz, 1H), 10.08 (s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ (ppm) 23.04, 47.95, 118.78, 118.91, 122.97, 123.37, 125.28, 126.13, 126.54, 127.03, 128.81, 129.09, 130.03, 130.39, 132.68, 133.25, 145.92, 153.73, 169.93; IR (KBr) ν/cm^{-1} 3410, 3168, 1647, 1597, 1574, 1516, 1439, 1335, 1281, 1192, 1066, 812, 744, 716; MS *m/z* observed: 341 (M^+).

4.4.2. *N-((2,4-Dichlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)thioacetamide (**2e**)*

Yield 84%; white solid; mp 218–220°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 1.92 (s, 3H, CH_3), 6.98 (s, J = 8 Hz, 1H), 7.10 (d, J = 8.8 Hz, 1H), 7.27 (d, J = 7.7 Hz, 1H), 7.36–7.46 (m, 3H), 7.59(d, J = 8 Hz, 1H), 7.73 (d, J = 8.8 Hz, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.95 (d, J = 8.4 Hz, 1H), 8.64 (s, 1H), 9.89(s, 1H, OH); ^{13}C NMR (DMSO-d_6): δ (ppm) 22.82, 47.78, 116.80, 119.11, 122.80, 122.99, 126.85, 126.95, 128.67, 128.90, 129.11, 130.10, 131.70, 132.09, 133.22, 133.31, 139.81, 154.28, 169.31; IR (KBr) ν/cm^{-1} 3405, 3118, 1650, 1581, 1517, 1438, 1369, 1321, 1279, 1163, 1088, 1063, 870, 814, 750; MS *m/z* observed: 376 (M^+).

4.4.3. *N-((4-Fluorophenyl)(2-hydroxynaphthalen-1-yl)methyl)thioacetamide (**2f**)*

Yield 83%; white solid; m p 202–203°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 1.95 (s, 3H, CH_3), 7.04–7.07 (b, 3H), 7.16–7.23 (m, 4H), 7.33–7.38 (b, 1H), 7.72–7.79 (m, 3H), 8.51 (m, 1H), 10.21 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 24.26, 56.51, 116.14, 116.35, 120.08, 120.26, 123.91, 124.62, 127.98, 129.49, 129.57, 129.94, 130.17, 130.90, 133.85, 140.39, 155.12, 161.11, 170.84; IR (KBr) ν/cm^{-1} 3396, 3077, 3055, 2975, 1627, 1581, 1510, 1439, 1335, 1276, 1160, 1064, 984, 820, 748; MS *m/z* observed: 325 (M^+).

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Disclosure statement

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Supplemental data

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