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## An efficient one-pot synthesis of highly substituted furans catalyzed by *N*-bromosuccinimide

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**Abstract:** *N*-Bromosuccinimide was found to efficiently catalyze the synthesis of highly functionalized, tetra-substituted furan derivatives in the one-pot reactions of but-2-ene-1,4-diones and acetoacetate esters in the presence of *i*-PrOH as solvent under mild and neutral conditions at 80–90 °C for 3–7 h in high yields (87–94 %).

**Keywords:** highly substituted furans; *N*-bromosuccinimide; but-2-ene-1,4-diones; acetoacetate esters.

### INTRODUCTION

Highly substituted furans are a structural component of a vast number of biologically active natural and synthetic compounds.<sup>1–4</sup> These compounds are found as structural units in many natural products, such as kallolides,<sup>5</sup> combrenolides,<sup>6</sup> pheromones<sup>7</sup> and polyether antibiotics.<sup>8</sup> These heterocycles have found applications in many pharmaceuticals, fragrances and dyes.<sup>9</sup> Furan subunits have also been used as building blocks for a large number of heterocyclic compounds and as synthons in natural product synthesis.<sup>10</sup> As a consequence, the synthesis of furan derivatives has been a subject of research for over a century, and a variety of well-established classical methods are now available in the literature.<sup>11–14</sup> The development of newer approaches for heterocycle syntheses employing efficient and economic routes is a popular research area nowadays. The most common strategy involved in the synthesis of furans is the cyclization<sup>15</sup> of 1,4-dicarbonyl compounds. Of the other various methods, syntheses involving transition-metal salts have recently been described for the preparation of substituted furan derivatives.<sup>16,17</sup> Oh *et al.*<sup>18</sup> synthesized highly substituted furans *via* Pt-catalyzed hydroxyl- or alkoxy-assisted cyclization of 2-(1-alkynyl)-2-alkene-1-ones. More recently, Dey and coworkers reported a novel method to

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highly substituted furans by  $\text{InCl}_3$ -catalyzed nucleophilic addition followed by cyclization reaction, although it is limited to specific substrate classes.<sup>19</sup>

#### EXPERIMENTAL

The employed chemicals were obtained from either Merck or Fluka. The IR spectra were recorded using a Shimadzu 435-U-04 spectrophotometer (KBr pellets) and the NMR spectra were obtained in  $\text{CDCl}_3$  using a 90 MHz JEOL FT NMR spectrometer. All melting points were determined on a Büchi 530 melting point apparatus and are reported uncorrected.

##### *Typical procedure for the synthesis of tetra-substituted furans*

To a stirred solution of but-2-ene-1,4-dione, **1a** (0.24 g, 1.0 mmol), and methyl acetoacetate, **2a** (0.18 g, 1.0 mmol), in dry *i*-PrOH (7.0 ml) was added anhydrous *N*-bromosuccinimide, NBS (52 mg, 0.23 mmol). The reaction mixture was then stirred under reflux at 80–90 °C for 3.1 h. After complete disappearance of the starting materials (monitored by TLC using petroleum ether–chloroform (6:4)), the solvent was removed from the reaction mixture on a rotary evaporator. The residue was then diluted with water (15 ml) and extracted with  $\text{CHCl}_3$  (4×15 ml). The organic layer was separated, washed with brine and then dried over anhydrous  $\text{MgSO}_4$ . Removal of the solvent resulted in a solid which was chromatographed over silica gel using petroleum ether and an increasing proportion of ethyl acetate as eluent. Petroleum ether–ethyl acetate (96:4) eluent gave a solid which was recrystallized from chloroform–petroleum ether (2:8): **3a** (0.31 g, 93 %), white solid, m.p. 92 °C.

The products **3b–j** were obtained in a similar manner using the appropriate but-2-ene-1,4-dione and acetoacetate ester.

The products were characterized on the basis of their physical and spectral analysis (Table I) and by direct comparison with literature data.<sup>19</sup>

TABLE I. IR (KBr),  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectral data of the tetra-substituted furans **3a–j**

Product	IR, $\tilde{\nu} / \text{cm}^{-1}$	$^1\text{H-NMR}$ , $\delta / \text{ppm}$	$^{13}\text{C-NMR}$ , $\delta / \text{ppm}$
<b>3a</b>	3068, 1712, 1610, 1452, 1058, 772	2.35 (3H, <i>s</i> , $-\text{CH}_3$ ), 3.28 (3H, <i>s</i> , $-\text{OCH}_3$ ), 4.45 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.36–7.55 (10H, <i>m</i> , Ar)	14.40 ( $-\text{CH}_3$ ), 35.63 ( $-\text{CH}_2-$ ), 51.12 ( $-\text{OCH}_3$ ), 107.69 ( $\text{C}_3$ ), 122.79 ( $\text{C}_4$ ), 126.01–134.57 (Ph), 149.02 ( $\text{C}_5$ ), 155.15 ( $\text{C}_2$ ), 166.78 ( $-\text{CO}_2\text{Me}$ ), 196.69 (CO)
<b>3b</b>	3063, 1716, 1625, 1460, 1050, 796	1.24 (3H, <i>t</i> , $-\text{CH}_2-\text{CH}_3$ ), 2.34 (3H, <i>s</i> , $-\text{CH}_3$ ), 4.12 (2H, <i>q</i> , $-\text{CH}_2-\text{CH}_3$ ), 4.42 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.30–7.57 (10H, <i>m</i> , Ar)	14.01 ( $-\text{CH}_2-\text{CH}_3$ ), 16.49 ( $-\text{CH}_3$ ), 37.57 ( $-\text{CH}_2-$ ), 59.74 ( $-\text{OCH}_2-$ ), 111.32 ( $\text{C}_3$ ), 125.65 ( $\text{C}_4$ ), 127.97–135.12 (Ph), 149.02 ( $\text{C}_5$ ), 155.74 ( $\text{C}_2$ ), 166.49 ( $-\text{CO}_2\text{Et}$ ), 196.62 (CO)
<b>3c</b>	3058, 2933, 1783, 1678, 1509, 1316, 1050, 827	2.31 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.35 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.44 (3H, <i>s</i> , $-\text{CH}_3$ ), 3.62 (3H, <i>s</i> , $-\text{OCH}_3$ ), 4.43 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.18–7.33 (4H, <i>dd</i> , Ar), 7.35 (2H, <i>d</i> , Ar), 7.96 (2H, <i>d</i> , Ar)	14.85 ( $-\text{CH}_3$ ), 21.08 ( $-\text{CH}_3$ ), 21.67 ( $-\text{CH}_3$ ), 35.78 ( $-\text{CH}_2-$ ), 51.38 ( $-\text{OCH}_3$ ), 114.36 ( $\text{C}_3$ ), 114.85 ( $\text{C}_4$ ), 127.03–144.15 (Ph), 150.79 ( $\text{C}_5$ ), 159.27 ( $\text{C}_2$ ), 165.08 ( $-\text{CO}_2\text{Me}$ ), 197.33 (CO)

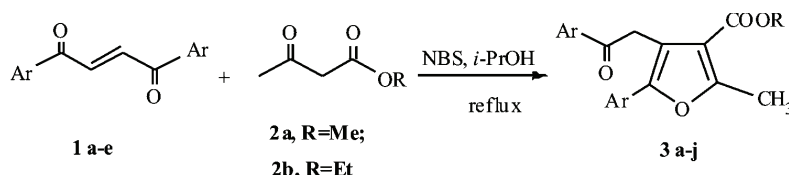
TABLE I. Continued

Product	IR, $\tilde{\nu}$ / $\text{cm}^{-1}$	$^1\text{H-NMR}$ , $\delta$ / ppm	$^{13}\text{C-NMR}$ , $\delta$ / ppm
<b>3d</b>	3070, 1710, 1609, 1448, 1250, 1108, 770	1.22 (3H, <i>t</i> , $-\text{CH}_2-\text{CH}_3$ ), 2.32 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.34 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.37 (3H, <i>s</i> , $-\text{CH}_3$ ), 4.10 (2H, <i>q</i> , $-\text{CH}_2-$ , $\text{CH}_3$ ), 4.45 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.03–7.19 (4H, <i>dd</i> , Ar), 7.76 (2H, <i>d</i> , Ar), 8.02 (2H, <i>d</i> , Ar)	14.15 ( $-\text{CH}_2-\text{CH}_3$ ), 16.58 ( $-\text{CH}_3$ ), 21.10 ( $-\text{CH}_3$ ), 21.82 ( $-\text{CH}_3$ ), 35.79 ( $-\text{CH}_2-$ ), 58.45 ( $-\text{OCH}_2-$ ), 115.01 ( $\text{C}_3$ ), 114.97 ( $\text{C}_4$ ) 127.03–143.47 (Ph), 157.98 ( $\text{C}_5$ ), 164.35 ( $\text{C}_2$ ), 166.08 ( $-\text{CO}_2\text{Et}$ ), 197.97 (CO)
<b>3e</b>	3080, 1704, 1605, 1442, 1238, 1025, 785	2.36 (3H, <i>s</i> , $-\text{CH}_3$ ), 3.30 (3H, <i>s</i> , $-\text{OCH}_3$ ), 4.47 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.48–7.56 (4H, <i>dd</i> , Ar), 7.70–7.81 (4H, <i>dd</i> , Ar)	15.32 ( $-\text{CH}_3$ ), 31.29 ( $-\text{CH}_2-$ ), 50.73 ( $-\text{OCH}_3$ ), 118.49 ( $\text{C}_3$ ), 123.84 ( $\text{C}_4$ ), 126.03–141.49 (Ph), 149.02 ( $\text{C}_5$ ), 155.17 ( $\text{C}_2$ ), 165.23 ( $-\text{CO}_2\text{Me}$ ), 196.70 (CO)
<b>3f</b>	3085, 1709, 1611, 1437, 1245, 1062, 788	1.24 (3H, <i>t</i> , $\text{CH}_2-\text{CH}_3$ ), 2.41 (3H, <i>s</i> , $-\text{CH}_3$ ), 4.13 (2H, <i>q</i> , $-\text{CH}_2-\text{CH}_3$ ), 4.48 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.41–7.50 (4H <i>dd</i> , Ar), 7.68–7.76 (4H, <i>dd</i> , Ar)	16.01 ( $\text{CH}_2-\text{CH}_3$ ), 17.07 ( $-\text{CH}_3$ ), 36.11 ( $-\text{CH}_2-$ ), 59.82 ( $-\text{OCH}_2-$ ), 116.12 ( $\text{C}_3$ ), 116.97 ( $\text{C}_4$ ), 131.17–147.53 (Ph), 158.38 ( $\text{C}_5$ ), 165.25 ( $\text{C}_2$ ), 165.98 ( $-\text{CO}_2\text{Et}$ ), 198.17 (CO)
<b>3g</b>	3100, 1705, 1610, 1450, 1248, 1055, 792	2.35 (3H, <i>s</i> , $-\text{CH}_3$ ), 3.28 (3H, <i>s</i> , $-\text{OCH}_3$ ), 4.45 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.39 (2H, <i>d</i> , Ar), 7.50–7.58 (4H, <i>dd</i> , Ar), 7.85 (2H, <i>d</i> , Ar)	14.68 ( $-\text{CH}_3$ ), 31.57 ( $-\text{CH}_2-$ ), 51.38 ( $-\text{OCH}_3$ ), 116.33 ( $\text{C}_3$ ), 127.81 ( $\text{C}_4$ ), 129.10–142.40 (Ph), 151.03 ( $\text{C}_5$ ), 156.37 ( $\text{C}_2$ ), 167.03 ( $-\text{CO}_2\text{Me}$ ), 196.19 (CO)
<b>3h</b>	3090, 1706, 1613, 1458, 1090, 838	1.20 (3H, <i>t</i> , $-\text{CH}_2-\text{CH}_3$ ), 2.32 (3H, <i>s</i> , $-\text{CH}_3$ ), 4.08 (2H, <i>q</i> , $-\text{CH}_2-$ ), $\text{CH}_2-\text{CH}_3$ ), 4.51 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.31 (2H, <i>d</i> , Ar), 7.45–7.51 (4H, <i>dd</i> , Ar), 7.73 (2H, <i>d</i> , Ar)	15.01 ( $-\text{CH}_2-\text{CH}_3$ ), 17.19 ( $-\text{CH}_3$ ), 34.24 ( $-\text{CH}_2-$ ), 59.01 ( $-\text{OCH}_2-$ ), 116.57 ( $\text{C}_3$ ), 115.87 ( $\text{C}_4$ ), 132.54–147.78 (Ph), 157.44 ( $\text{C}_5$ ), 166.05 ( $\text{C}_2$ ), 167.01 ( $-\text{CO}_2\text{Et}$ ), 197.18 (CO)
<b>3i</b>	3110, 1703, 1606, 1451, 1100, 840	2.18 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.21 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.29 (3H, <i>s</i> , $-\text{CH}_3$ ), 3.32 (3H, <i>s</i> , $-\text{OCH}_3$ ), 4.56 (2H, <i>s</i> , $-\text{CH}_2-$ ) 7.24 (3H, <i>m</i> , Ar), 7.51 (3H, <i>m</i> , Ar)	14.11 ( $-\text{CH}_3$ ), 25.98 ( $-\text{CH}_3$ ), 26.29 ( $-\text{CH}_3$ ), 37.12 ( $-\text{CH}_2-$ ), 51.74 ( $-\text{OCH}_3$ ) 116.30 ( $\text{C}_3$ ), 116.84 ( $\text{C}_4$ ), 129.14–145.35 (Ph), 159.70 ( $\text{C}_5$ ), 169.47 ( $\text{C}_2$ ), 170.22 ( $-\text{CO}_2\text{Me}$ ), 198.35 (CO)
<b>3j</b>	3105, 1707, 1611, 1452, 1235, 1039, 770	1.23 (3H, <i>t</i> , $\text{CH}_2-\text{CH}_3$ ), 2.17 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.28 (3H, <i>s</i> , $-\text{CH}_3$ ), 2.31 (3H <i>s</i> , $-\text{CH}_3$ ), 4.10 (2H <i>q</i> , $-\text{CH}_2-\text{CH}_3$ ), 4.53 (2H, <i>s</i> , $-\text{CH}_2-$ ), 7.12 (3H, <i>m</i> , Ar), 7.49 (3H, <i>m</i> , Ar)	14.15 ( $-\text{CH}_2-\text{CH}_3$ ), 16.58 ( $-\text{CH}_3$ ) 26.78 ( $-\text{CH}_3$ ), 27.27 ( $-\text{CH}_3$ ), 37.48 ( $-\text{CH}_2-$ ), 59.37 ( $-\text{OCH}_2-$ ), 115.01 ( $\text{C}_3$ ), 116.13 ( $\text{C}_4$ ) 130.12–145.25 (Ph), 160.15 ( $\text{C}_5$ ), 170.31 ( $\text{C}_2$ ), 172.15 ( $-\text{CO}_2\text{Et}$ ), 198.54 (CO)

## RESULTS AND DISCUSSION

In continuation of on-going research on various transformations by halogenating agents and s ydnones,<sup>20–26</sup> and also in order to avoid the drawbacks generally resulting from the use of strong acidic media in nitrosation reactions, herein is reported the use of NBS as a more robust and efficient catalyst in the

one-pot synthesis of the highly functionalized tetra-substituted furan derivatives **3a–j** by reaction of but-2-ene-1,4-diones **1a–e** and acetoacetate esters **2a** or **2b** in *i*-PrOH in satisfactory yields (87–94 %) under neutral conditions (Scheme 1, Table II). As shown in Table II, the reactions occurred satisfactorily within 3.1–6.1 h under reflux conditions. The experimental results indicate that the most effective conversion occurred when a 1:0.23 substrate:NBS mole ratio was used. Longer reaction times were required when lower amounts of NBS were employed. It is important to note that no furan derivatives were afforded when the reactions were performed in the absence of NBS in the reaction mixture.



Scheme 1. Proposed mechanism for the synthesis of highly substituted furans.<sup>26</sup>

TABLE II. NBS-catalyzed synthesis of furans **3a–j**

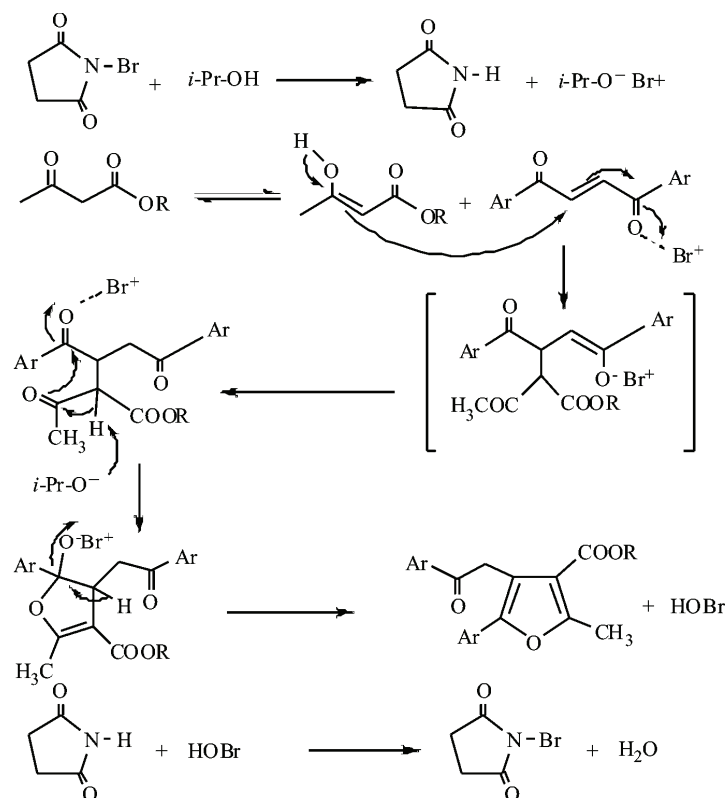
Entry	Product <sup>a</sup>	Ar	R	Time, h	Yield, % <sup>b</sup>	M.p., °C
1	<b>3a</b>	C <sub>6</sub> H <sub>5</sub> Me		3.1	93	92
2	<b>3b</b>	C <sub>6</sub> H <sub>5</sub> Et		3.9	91	89
3	<b>3c</b>	4-Me-C <sub>6</sub> H <sub>4</sub> Me		4.0	89	83
4	<b>3d</b>	4-Me-C <sub>6</sub> H <sub>4</sub> Et		3.8	87	80
5	<b>3e</b>	4-Br-C <sub>6</sub> H <sub>4</sub> Me		3.3	89	87
6	<b>3f</b>	4-Br-C <sub>6</sub> H <sub>4</sub> Et		5.2	94	85
7	<b>3g</b>	4-Cl-C <sub>6</sub> H <sub>4</sub> Me		4.7	90	96
8	<b>3h</b>	4-Cl-C <sub>6</sub> H <sub>4</sub> Et		5.5	91	94
9	<b>3i</b>	3-Cl,4-Me-C <sub>6</sub> H <sub>3</sub> Me		6.0	93	79
10	<b>3j</b>	3-Cl,4-Me-C <sub>6</sub> H <sub>3</sub> Et		6.1	94	77

<sup>a</sup>All the isolated products were characterized by their physical properties, by <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and IR spectra and by direct comparison with literature data; <sup>b</sup>isolated yields

The mechanism shown in Scheme 2 is proposed for these reactions.<sup>26</sup> Thus, the 1,4-diarylbut-2-ene-1,4-diones act as Michael acceptors and the acetoacetates as nucleophiles resulting in a Michael adduct that under the influence of NBS forms a hemiketal, which undergoes spontaneous dehydration to afford the furans. It is important to note that no furan derivatives were formed when the reactions were performed in the presence of HBr as catalyst. Furthermore, no reaction was seen when the 1,4-diarylbut-2-ene-1,4-diones and acetoacetates were used separately as substrates with NBS as the catalyst in the presence of *i*-PrOH under reflux.

The advantages or the characteristic aspects of the method described in this paper in comparison with other previously reported ones are the following: the yields of products were better than the previous reported yields and in addition, the catalyst NBS in comparison with 1,3-dibromo-5,5-dimethylhydantoin (DBH)

and  $\text{InCl}_3$  is inexpensive, has no moisture sensitivity, and no special measures are required for the reaction.



The role of the solvent was also investigated. Among the various solvents tested, *i*-PrOH afforded the maximum yield of the furan derivative **3a** (Table III). It is well known that reactions of this type are more efficient in polar solvents, which was corroborated in this study (Table III). It was also observed that the inclusion of water had very little or no effect on this reaction.

TABLE III. Role of the solvent in the synthesis of furan **3a**

Solvent	Ti	me, <sup>a</sup> h	Isolated yield of <b>3a</b> , %
<i>i</i> -PrOH	3.0		93
<i>i</i> -PrOH-H <sub>2</sub> O	(6:4)	11	60
MeOH	9.0		52
CH <sub>3</sub> CN	18		44
CH <sub>2</sub> Cl <sub>2</sub>	15		38
THF	14		61

<sup>a</sup>Extension of the reaction did not improve the product yield

## CONCLUSIONS

The present methodology shows that *N*-bromosuccinimide (NBS) is an efficient catalyst in the one-pot synthesis of highly functionalized tetra-substituted furan derivatives. The main advantages of the presented protocol are mild, clean and environmentally benign reaction conditions, as well as the high yields. Furthermore, this method is also expected to find application in organic synthesis due to the low cost of the reagent. It is believed that this method will be a useful addition to modern synthetic methodologies.

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## ИЗВОД

ЕФИКАСНА СИНТЕЗА У ЈЕДНОМ СУДУ ВИСОКО СУПСТИТУИСАНИХ ФУРАНА  
КАТАЛИЗОВАНА *N*-БРОМСУКЦИНИМИДОМ

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Утврђено је да *N*-бромсукцинимид ефикасно катализује синтезу високо функционализованих, тетрасупституисаних деривата фурана у реакцији у једном суду бут-2-ен-1,4-диона и ацетоацетатних естера у *i*-PrOH као растварачу под благим и неутралним условима на 80–90 °C током 3–7 h уз високе приносе (87–94 %).

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