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One-pot synthesis of new derivatives of pyran using *N*-halosulfonamide[†]

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In this study *N*,*N*,*N*',*N*'-tetrabromobenzene-1,3-disulfonamide was used as a reagent for the threecomponent one-pot synthesis of some novel 9-aryl-9*H*-2,4,5,7-tetramethyl-diuracilopyrans, which has not been published before, as well as 9-aryl-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)-diones in excellent yields. This is obtained from the condensation of aromatic and heterocyclic aldehydes and *N*,*N*dimethylbarbituric acid or cyclohexane-1,3-diones in ethanol at room temperature.

Introduction

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An important challenge in organic synthesis is development of powerful methods for the synthesis of complex heterocyclic molecules from readily available reagents.¹ Multicomponent condensation reactions (MCRs) have recently been discovered to be a powerful method for the synthesis of organic compounds. High selectivity, high atom-economy, simplicity and relatively high speed are central issues in multicomponent reactions.^{2–4}

In recent years, 4*H*-pyrans and their derivatives have attracted intense interest due to their useful biological and pharmacological properties, such as being anti-inflammatory,⁵ antimicrobial,⁶ anticonvulsant activities,⁷ anticoagulant, spasmolytic, anticancer and anti-anaphylactic agents.⁸ There are widely applied in photodynamic therapy,⁹ and used as fluorescent dye materials¹⁰ for visualization of biomolecules,^{11,12} and in laser technology¹³ (Fig. 1). Furthermore, substituted 4*H*-pyrans constitute a structural unit of a series of natural products.¹⁴ Especially pyran diones, that belong to the naturally-occurring compounds, are used as reactive intermediates for the





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synthesis of various important compounds which have recently drawn great attention.

Results and discussion

To pursue our interest in the application of N,N,N',N' tetrabromobenzene-1,3-disulfonamide [TBBDA],¹⁵ (Fig. 2) in organic synthesis,^{15–20} herein we introduce a facile, threecomponent one-pot synthesis of new 9-aryl-9*H*-2,4,5,7tetramethyl-diuracilopyran (route A), which has not been published before, as well as 9-aryl-3,4,6,7-tetrahydro-2*H*-xanthene-1,8(5*H*,9*H*)-dione (route B). This is obtained from the condensation of various aromatic and heteroaromatic aldehydes and *N*,*N*-dimethylbarbituric acid or cyclohexane-1,3-dione in the presence of TBBDA as reagent in ethanol at room temperature (Scheme 1).

It is easy to prepare TBBDA and it is stable under atmospheric conditions for two months. Furthermore, TBBDA is a non-volatile, inexpensive, and safe reagent. After completion of the reaction, the sulphonamide was recovered, brominated and used for several times.

Initial efforts were focused on optimizing conditions for the formation of 9-(2-chlorophenyl)-9*H*-2,4,5,7-tetramethyl-diuracilopyran using *N*,*N*-dimethylbarbituric acid and 2-chlorobenzaldehyde under various conditions (Table 1).

In this context, ethanol (EtOH) selected as best solvent for these reactions. Reactions in EtOH are generally considered environmentally safe, devoid of any carcinogenic effects, simple



TBBDA

Fig. 2 The structure of TBBDA.

[†] Electronic supplementary information (ESI) available. See DOI: 10.1039/c4ra04929b

Scheme 1 Synthesis of new derivatives of pyran using TBBDA.

Table 1Optimization of reaction conditions for the synthesis of 9-(2-
chlorophenyl)-9H-2,4,5,7-tetramethyl-diuracilopyran^a

Entry	Solvent	Amount of TBBDA (g)	Time (min)	Yield (%)
1	Ethanol	0	60	23
2	Ethanol	0.05	60	62
3	Ethanol	0.1	60	91
4	Ethanol	0.15	60	85
5	No solvent	0.1	60	41
6	Acetone	0.1	30	33
7	H_2O	0.1	30	42

^{*a*} Experimental conditions: 2-chlorobenzaldehyde (1 mmol) and *N*,*N*-dimethylbarbituric acid (2 mmol).

to handle, cheaper to operate and especially important in industry.

We found that the reaction was rapid when using N,N,N',N' tetrabromobenzene-1,3-disulfonamide [TBBDA] (0.1 g, 60 min, entry 3). We then examined a wide variety of aromatic aldehyde and we observed that they successfully react with N,N-dimethylbarbituric acid or cyclohexane-1,3-dione. This clearly establishes the scope and generality of this method. As it is shown in Table 2, the electron withdrawing or donating groups on the phenyl rings did not have an influence on the reaction.

The probable mechanism of this reagent is that it releases Br⁺ *in situ*, which can act as an electrophilic species.^{15–20} Therefore, the mechanism shown in Scheme 2 may be suggested for the conversion of the various aldehyde, *N*,*N*-dimethylbarbituric acid, cyclohexane-1,3-dione to pyran derivatives.

Conclusions

In this study, we have developed a green one-pot synthesis of pyrans derivatives using [TBBDA] as new reagent. The conditions are mild and a wide range of functional groups can be tolerated. This method offers several advantages such as inexpensive reagent, short reaction times, easy synthetic procedure, good to high yields, simple work-up procedure and easy isolation.

Materials and equipment

All commercially available chemicals were obtained from Merck and Fluka and used without further purification unless otherwise stated. ¹H and ¹³C-NMR spectra were recorded on Bruker Advance 400 FT NMR spectrometers (undertaken at University of Mazandaran, Iran). Infrared (IR) spectroscopy was performed on a Perkin Elmer GX FT-IR spectrometer. Mass spectra were recorded on a 5973 Network Mass Selective Detector Mass Spectrometer (undertaken at University of Tehran, Iran). Elemental analyses (C, H, N) were performed with a Elemental Combustion System 4010 (undertaken at University of Tehran, Iran).

Typical procedure for the preparation of 9-(2-chlorophenyl)-9*H*-2,4,5,7-tetramethyl-diuracilopyran (Table 2, entry 4)

A mixture of *N*,*N*-dimethylbarbituric acid (0.312 g, 2 mmol), 2chlorobenzaldehyde (0.140 g, 1 mmol), ethanol (2 mL) and TBBDA (0.1 g, 0.18 mmol) was stirred at room temperature for 60 min. The progress of the reaction was monitored by TLC [acetone/*n*-hexane (3 : 10)]. After completion of the reaction, the reaction mixture was filtered and washed with ethanol. The crude product was recrystallized from hot ethanol to afford the pure product as a white powder (91%). After evaporation of the ethanol, cold methylene dichloride (2 mL) was added, and reagent was recovered by filtration.

9-(2-Chlorobenzo[h]quinoline)-9*H*-2,4,5,7-tetramethyldiuracilopyran (Table 2, entry 1); white powder 88%; m.p. 273– 275 °C; elem. anal. found: C, 59.94; H, 3.35; N, 13.08 calc. for $C_{26}H_{20}ClN_5O_5$: C, 60.29; H, 3.89; N, 13.52%. IR (KBr, ν_{max}/cm^{-1}): 3450, 3053, 2956, 1724, 1650; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 2.44 (s, 3H, OMe), 3.14 (s, 3H, OMe), 3.27 (s, 3H, OMe), 3.47 (s, 3H, OMe), 5.45 (s, 1H, CH), 7.80–7.90 (m, 5H, ArH), 8.46 (s, 1H, ArH), 8.99–9.01 (q, 1H, ArH); ¹³C-NMR (100 MHz, DMSOd6) $\delta_{\rm C}$ (ppm): 28.2, 28.2, 29.3, 30.3, 52.8, 85.6, 89.2, 124.4, 125.2, 125.6, 127.2, 128.2, 128.7, 129.2, 129.5, 129.9, 134.2, 141.5, 145.6, 148.4, 150.6, 151.4, 158.5, 163.2, 163.4, 165.8; MS *m/z*: 517 (M⁺).

9-(2-Chloroquinolin-3-yl)-9H-2,4,5,7-tetramethyl-diuracilopyran (Table 2, entry 2); opalescent powder 85%; m.p. 265-267 °C; elem. anal. found: C, 56.43; H, 3.59; N, 14.78 calc. for $C_{22}H_{18}ClN_5O_5$: C, 56.48; H, 3.88; N, 14.97%. IR (KBr, ν_{max}/cm^{-1}): 3406, 3063, 2955, 1676, 1646; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 2.44 (s, 3H, OMe), 2.89 (s, 3H, OMe), 3.11 (s, 3H, OMe), 3.13 (s, 3H, OMe), 3.21 (s, 3H, OMe), 3.25 (s, 3H, OMe), 3.45 (s, 3H, OMe), 3.49 (s, 3H, OMe), 5.11 (s, 1H, CH), 5.39 (s, 1H, CH), 7.58-7.61 (t, J 7.2, 1H, ArH), 7.67-7.71 (t, J 7.6, 1H, ArH), 7.78-7.81 (t, J 7.6, 1H, ArH), 7.84-7.86 (d, J 7.6, 1H, ArH), 7.88-7.92 (t, J 7.2, 1H, ArH), 7.96-7.98 (d, J 8.4, 1H, ArH), 8.01-8.04 (t, J 7.6, 2H, ArH), 8.32 (s, 1H, ArH), 8.43 (s, 1H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 28.2, 28.2, 29.3, 29.6, 30.3, 35.1, 52.8, 55.6, 85.6, 86.1, 89.2, 126.3, 127.0, 127.1, 127.7, 128.0, 128.3, 128.5, 128.7, 131.5, 132.3, 139.2, 141.9, 147.0, 149.2, 150.6, 151.4, 158.5, 161.5, 163.1, 165.7; MS m/z: 467 (M⁺).

9-(4-Methoxyphenyl)-9*H*-2,4,5,7-tetramethyl-diuracilopyran (Table 2, entry 3); white powder 85%; m.p. 246–248 °C; elem. anal. found: C, 51.91; H, 4.09; N, 12.4 calc. for $C_{20}H_{20}N_4O_6$: C, 53.25; H, 4.89; N, 13.59%. IR (KBr, ν_{max}/cm^{-1}): 3451, 2964, 1715, 1674; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 2.46 (s, 3H, OMe), 3.11 (s, 3H, OMe), 3.21 (s, 3H, OMe), 3.39 (s, 3H, OMe),

Table 2	Synthesis of ne	w derivatives of	pyran using	TBBDA
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Entry	Aldehyde	Product	Time (min)	Yield ^a (%)	M.P. (°C)
1	N CHO	$Me_{N} \rightarrow Me_{Me} \rightarrow Me_{Me}$	60	88	273-275
2	CHO N Cl	Me N He Me	30	85	265-267
3	CHO	$Me_{N} \xrightarrow{OMe}_{N} Me_{N}$	100	85	246-248
4	CHO	$Me_{N} \xrightarrow{V} Me_{Me} \xrightarrow{V} Me$	60	91	199-201
5	CHO Cl	$Me_{N} \xrightarrow{Cl} Me_{N} \xrightarrow{N} Me_{Me} \xrightarrow{N} Me_{Me}$	25	82	258-259
6	CHO F		120	61	266-268
7	CHO	O OMe O O O O O O O O O	120	66	199–201
8	CHO CHO N H	O O O	180	63	268-269

Entry	Aldehyde	Product	Time (min)	Yield ^{<i>a</i>} (%)	M.P. (°C)
9	CHO S Me	o o o o o	180	73	232-233
10	CHO Me	Me O O O O	120	79	259-261
11	CHO NO ₂		120	75	252-254
^{<i>a</i>} Isolated yi	eld.				



Scheme 2 Suggested mechanism for synthesis of pyran derivatives.

3.73 (s, 3H, OMe), 5.05 (s, 1H, CH), 6.82–7.07 (s, 4H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 28.1, 28.2, 29.4, 30.1, 55.6, 56.0, 86.0, 90.6, 113.8, 126.7, 130.3, 150.571, 151.3, 158.6, 159.8, 162.6, 163.6, 166.0; MS *m/z*: 412 (M⁺).

9-(2-Chlorophenyl)-9*H*-2,4,5,7-tetramethyl-diuracilopyran (Table 2, entry 4); white powder 91%; m.p. 199–201 °C; elem. anal. found: C, 52.97; H, 3.54; N, 12.70 calc. for $C_{19}H_{17}ClN_4O_5$: C, 53.75; H, 4.11; N, 13.44%. IR (KBr, ν_{max}/cm^{-1}): 3415, 3069, 2959, 1766, 1702, 1693, 1667; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 2.51 (s, 3H, OMe), 3.11 (s, 3H, OMe), 3.20 (s, 3H, OMe), 3.40 (s, 3H, OMe), 5.26 (s, 1H, CH), 7.23–7.37 (m, 3H, ArH), 7.46–7.48 (d, *J* 8, 1H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 28.1, 28.2, 29.2, 30.2, 52.8, 85.5, 89.3, 127.7, 129.4, 130.9, 131.9,

131.9, 133.3, 150.5, 151.3, 158.4, 163.0, 163.3, 165.9; MS *m*/*z*: 415 (M⁺-1).

9-(2,4-Dichlorophenyl)-9*H*-2,4,5,7-tetramethyl-diuracilopyran (Table 2, entry 5); white powder 82%; m.p. 258–259 °C; elem. anal. found: C, 48.82; H, 2.93; N, 12.22 calc. for C₁₉H₁₆Cl₂N₄O₅: C, 50.57; H, 3.57; N, 12.42 IR (KBr, ν_{max}/cm^{-1}): 3446, 3099, 2957, 1765, 1708, 1670; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 2.59 (s, 3H, OMe), 3.11 (s, 6H, 2 OMe), 3.40 (s, 3H, OMe), 5.23 (s, 1H, CH), 7.29–7.40 (m, 2H, ArH), 7.68–7.68 (d, *J* 2, 1H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 28.1, 28.2, 28.3, 28.4, 28.9, 29.2, 30.2, 52.2, 85.4, 89.1, 122.5, 127.1, 128.0, 128.7, 128.9, 128.9, 131.3, 132.1, 133.2, 133.3, 134.1, 134.3, 134.6, 135.9, 149.8, 150.5, 151.3, 158.4, 163.1, 163.2, 165.7, 166.3; MS *m/z*: 449 (M⁺-1).

9-(4-Fluorophenyl)-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)dione (Table 2, entry 6); white powder 61%; m.p. 266–268 °C; elem. anal. found: C, 72.75; H, 5.30; calc. for C₁₉H₁₇FO₃: C, 73.06; H, 5.49%. IR (KBr, $\nu_{\rm max}/{\rm cm}^{-1}$): 2955, 1721, 1655; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 1.80–1.89 (m, 2H, CH₂), 1.91– 1.98 (m, 2H, CH₂), 2.21–2.34 (m, 4H, 2CH₂), 2.55–2.70 (m, 4H, 2CH₂), 4.56 (s, 1H, CH), 7.00–7.04 (t, *J* 8.8, 2H, ArH), 7.19–7.22 (q, 2H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 20.3, 26.8, 30.7, 36.8, 114.9, 115.1, 115.7, 130.2, 130.3, 141.1, 141.1, 159.8, 162.2, 165.3, 196.8; MS *m/z*: 312 (M⁺).

9-(3-Methoxyphenyl)-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)dione (Table 2, entry 7); white powder 66%; m.p. 199–201 °C; elem. anal. found: C, 74.01; H, 6.07; calc. for C₂₀H₂₀O₄: C, 74.06; H, 6.21%. IR (KBr, ν_{max}/cm^{-1}): 3434, 2969, 2935, 2835, 1670, 1654; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 1.82–1.87 (m, 2H, CH₂), 1.92–1.98 (m, 2H, CH₂), 1.98–2.35 (m, 4H, 2CH₂), 2.56– 2.70 (m, 4H, 2CH₂), 3.69 (s, 3H, OCH₃), 4.56 (s, 1H, CH), 6.68– 6.71 (q, 2H, ArH), 6.73–6.75 (d, *J* 8, 1H, ArH), 7.10–7.14 (t, *J* 10, 1H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 20.3, 26.8, 31.1, 36.8, 55.2, 111.3, 114.8, 115.8, 120.5, 129.4, 146.4, 159.3, 165.3, 196.7; MS *m*/*z*: 324 (M⁺).

9-(1*H*-Indole-3-yl)-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)dione (Table 2, entry 8); opalescent powder 63%; m.p. 268– 269 °C; elem. anal. found: C, 74.94; H, 5.49; N, 3.83; calc. for $C_{21}H_{19}NO_3$: C, 75.66; H, 5.74; N, 4.20%.IR (KBr, ν_{max}/cm^{-1}): 3415, 3058, 2946, 2895, 1676, 1656; ¹H-NMR (400 MHz, DMSOd6) $\delta_{\rm H}$ (ppm): 1.79 (broad, 2H, CH₂), 1.92 (broad, 2H, CH₂), 2.24 (m, 4H, 2CH₂), 2.64–2.65 (m, 4H, 2CH₂), 4.87 (s, 1H, CH), 6.94– 6.95 (t, *J* 7.2, 1H, ArH), 7.00 (m, 2H, ArH), 7.25–7.27 (d, *J* 8, 1H, ArH), 7.55–7.57 (d, *J* 8, 1H, ArH), 10.79 (s, 1H, NH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 20.4, 22.3, 26.9, 36.9, 111.8, 116.2, 118.5, 118.7, 119.3, 120.9, 124.2, 126.1, 136.6, 164.7, 196.8; MS *m/z*: 333 (M⁺).

9-(5-Methylthiophen-2-yl)-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)-dione (Table 2, entry 9); pale yellow powder 73%; m.p. 232–233 °C; elem. anal. found: C, 68.44; H, 5.73; S, 9.11; calc. for $C_{18}H_{18}O_3S$: C, 68.76; H, 5.77; O, 15.27; S, 10.20%. IR (KBr, ν_{max}/cm^{-1}): 3313, 2957, 2936, 2892, 2818, 1671, 1618; ¹H-NMR (400 MHz, DMSO-d6) δ_{H} (ppm): 1.85–1.93 (m, 2H, CH₂), 1.95–2.01 (m, 2H, CH₂), 2.29 (s, 3H, CH₃), 2.32–2.35 (m, 4H, 2CH₂), 2.55–2.68 (m, 4H, 2CH₂), 4.79 (s, 1H, CH), 6.47–6.50 (q, 2H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) δ_{C} (ppm): 15.3, 20.3, 25.8, 26.8, 36.8, 115.5, 124.3, 125.1, 137.7, 146.1, 165.5, 196.6; MS (*m*/*z*): 314 (M⁺).

9-*p*-Tolyl-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)-dione (Table 2, entry 10); white powder 79%; m.p. 259–261 °C; elem. anal. found: C, 77.99; H, 6.63 calc. for C₂₀H₂₀O₃: C, 77.90; H, 6.54%. IR (KBr, ν_{max}/cm^{-1}): 3032, 2954, 2893, 1656, 1617; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 1.78–1.88 (m, 2H, CH₂), 1.92–1.97 (m, 2H, CH₂), 2.20 (s, 3H, CH₃), 2.22–2.33 (m, 4H, 2CH₂), 2.56–2.69 (m, 4H, 2CH₂), 4.54 (s, 1H, CH), 6.99–7.01 (d, *J* 8, 2H, Ph), 7.05–7.07 (d, *J* 8, 2H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 20.3, 21.0, 26.8, 30.8, 36.8, 116.1, 128.3, 128.9, 135.6, 142.1, 165.1, 196.7; MS (*m*/*z*): 308 (M⁺).

9-(4-Nitrophenyl)-3,4,6,7-tetrahydro-2*H*-pyran-1,8(5*H*,9*H*)dione (Table 2, entry 11); white powder 75%; m.p. 252–254 °C; elem. anal. found: C, 67.18; H, 4.91; N, 3.85; calc. for $C_{19}H_{17}NO_5$: C, 67.25; H, 5.05; N, 4.13%. IR (KBr, ν_{max}/cm^{-1}): 3071, 2949, 2874, 1660, 1617; ¹H-NMR (400 MHz, DMSO-d6) $\delta_{\rm H}$ (ppm): 1.86–1.91 (m, 2H, CH₂), 1.93–1.99 (m, 2H, CH₂), 2.22–2.36 (m, 4H, 2CH₂), 2.58–2.71 (m, 4H, 2CH₂), 4.66 (s, 1H, CH), 7.47–7.49 (d, *J* 8.4, 2H, ArH), 8.07–8.09 (d, *J* 8.4, 2H, ArH); ¹³C-NMR (100 MHz, DMSO-d6) $\delta_{\rm C}$ (ppm): 20.2, 26.9, 32.0, 36.7, 114.8, 123.6, 129.9, 146.3, 152.4, 165.8; MS (*m*/*z*): 339 (M⁺).

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