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ORIGINAL ARTICLE

Efficient and selective α-bromination of carbonyl compounds with *N*-bromosuccinimide under microwave

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Abstract A highly efficient method for the synthesis of α-halocarbonyl compounds has been achieved via selective monobromination of aromatic and aliphatic carbonyl compounds with *N*-bromosuccinimide catalyzed by *p*-toluenesulfonic acid under microwave irradiation within 30 min.

1. Introduction

α-Bromination of carbonyl compounds is a direct method for the preparation of α-bromoalkanones, which has attracted considerable attention in the synthetic organic chemistry (Larock, 1999; Erian et al., 2003; Yunus and Winterfeldt, 2007; Dogo-Isonagie et al., 2007; Salama and Novak, 2011), because the resulting α-brominated products are important intermediates for the synthesis of various useful molecules such as pesticides, pharmaceuticals, surfactants and biologically active heterocyclic compounds (Talegaonkar et al., 1982; Zhang et al., 2002). α-Bromination is also a key step for introducing a functional group into a molecule for further transformation reactions (Harwood, 1962). Bromine has been previously used as a basic brominating reagent for the α-bromination of carbonyl compounds (Langley, 1932; Bigelow and Hanslick, 1943), but it is not considered as a friendly choice for the bromination reaction due to its unfavorable properties such as being irritating, toxic, corrosive and difficult to handle. Moreover, its high reactivity can lead to highly exothermic and non-selective reactions (Salama and Novak, 2011). To overcome these limitations, several different reagents such as copper (II) bromide (King and Ostrum, 1964), tribromoacetophenone (krohnke and Ellegast, 1953), 1,4-dioxane bromooxonium bromide (Yanovskaya et al., 1953), pyridium (Fieser and Fieser, 1967) and tetrabutylammonium tribromide (Kajigaeshi et al., 1987) have also been employed as alternatives to bromine, but most of these methods still suffer from drawbacks such as long reaction time, low reactivity, high cost, etc. The development of a more efficient and economic method for bromination is still highly desirable.

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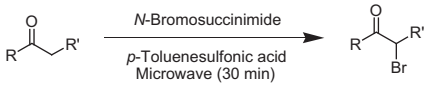
Recently, the use of *N*-bromosuccinimide (NBS) as a brominating reagent has become popular in organic synthesis, because it is easy to handle and user-friendly (Lee and Bae, 2003; Lee et al., 2003; Das et al., 2005; Yang et al., 2002; Tanemura et al., 2004). Furthermore, the resulting succinimide as the byproduct of NBS-bromination is recyclable. Carbonyl compounds can be brominated by NBS via either a radical pathway mediated by radical initiators or via ionic pathway catalyzed by acids. For instance, Paul and co-workers have reported the selective α-bromination of carbonyl compounds using NBS mediated by silica-supported perchloric acid (Gupta et al., 2008). Samant et al. described a highly efficient α-bromination of acetophenones with NBS catalyzed by *p*-toluenesulfonic acid (PTSA) under ultrasound (Adhikari and Samant, 2002). Stavber and co-workers also demonstrated the directed regioselective α-brominaiton of ketones with NBS in the presence of PTSA under solvent-free condition (Pravst et al., 2006).

Over the last decade, microwave irradiation has been proven to be a powerful and well-controlled heating source for a wide variety of organic transformations. In many cases, the reaction time can be significantly reduced with improved yields and/or selectivity (Zhang et al., 2006; Loupy, 2004; Hayes, 2002, 2004; Kappe and Stadler, 2006; Perreux and Loupy, 2001; Lidström et al., 2001; Caddick and Fitzmaurice, 2009; Sharma et al., 2011; Al-Hazimi et al., 2012). A lot of effort has been made to the development of environmentally friendlier synthetic alternatives for α-halogenations using microwave. For example, a selective α-brominaiton of ketones with dioxane-dibromide and silica gel was achieved under microwave irradiation (Paul et al., 2002). The Lee group recently reported microwave induced α-iodination of ketones with *N*-iodosuccinimide and *p*-toluenesulfonic acid (Lee et al., 2003). The same group also demonstrated that the sequential treatment of carbonyl compounds with [hydroxy(tosyloxy)iodo]benzene followed by magnesium halides was an efficient method for α-halogenation of ketones (Lee et al., 2004). Here, we describe an efficient method for the preparation of α-bromoalkanones via the reaction of carbonyl compounds with NBS catalyzed by *p*-toluenesulfonic acid under microwave irradiation conditions (Scheme 1).

2. Experimental

2.1. Reagents and analysis

All solvents were purified by standard methods. All ^{1}H NMR, and ^{13}C NMR spectra were recorded using a Bruker AVIII 400 or AVIII 500 spectrometer in $CDCl_3$ unless otherwise noted. Tetramethylsilane (TMS) served as internal standard ($\delta = 0$) for ^{1}H NMR, and $CDCl_3$ was used as internal standard ($\delta = 77.0$) for ^{13}C NMR. Chemical shifts are reported in parts per million as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad). The NMR data were processed using the topspin program version 2.1. The α-bromination of carbonyl compounds with *N*-bromosuccinimide was performed in a CEM Matthews WC Discover microwave reactor (model no. 908010 DV9068 equipped with programmable pressure and temperature controller). Solvents were freshly dried and degassed according to "Purification of Laboratory Chemicals" prior to use. Column chromatography purifications were performed by flash chromatography using Merck silica gel 60.



Scheme 1 Synthesis of α-bromoalkanones under microwave irradiation.

2.2. General procedure

In a dry 10 mL flask with a Teflon stir bar were introduced 0.2 mmol of the carbonyl compound, NBS (0.2 mmol, 1 equiv), and PTSA (0.02 mmol, 10 mol%). Anhydrous DCM (2.0 mL) was added, and then the flask was sealed and the mixture was stirred and heated under microwave. After 30 min, the reaction mixture was cooled and treated with 10 mL of distilled water, and extracted with 3×10 mL of CH_2Cl_2. The organic layers were separated, dried over $MgSO_4$, and purified by flash chromatography to give the corresponding product.

2.2.1. 2-Bromo-1-phenylethanone (*1b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.46 (s, 2H), 7.47–7.51 (m, 2H), 7.59–7.72 (m, 1H), 7.97–7.99 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.67, 128.51, 128.56, 133.52, 133.63, 190.93. HRMS (ESI) *m*/*z* 197.9680, calc. for [C_8H_7BrO] 197.9683.

2.2.2. 2-Bromo-1-(4-nitrophenyl)ethanone (*2b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.46 (s, 2H), 8.15–8.17 (m, 2H), 8.34–8.35 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 29.77, 123.69, 129.72, 137.94, 150.29, 189.52. HRMS (ESI) *m*/*z* 242.9531, calc. for [$C_8H_6BrNO_3$] 242.9536.

2.2.3. 2-Bromo-1-(4-bromophenyl)ethanone (*3b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.40 (s, 2H), 7.63–7.65 (m, 2H), 7.84–7.86 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.01, 128.97, 130.06,131.86, 132.21, 190.07. HRMS (ESI) *m*/*z* 275.8785, calc. for [$C_8H_6Br_2O$] 275.8783.

2.2.4. 2-Bromo-1-(4-methoxyphenyl)ethanone (*4b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.88 (s, 3H), 4.40 (s, 2H) 6.94–6.96 (m, 2H), 7.96–7.97 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.38, 55.21, 113.68, 126.48, 131.00, 163.74, 189.61. HRMS (ESI) *m*/*z* 227.9786, calc. for [$C_9H_9BrO_2$] 227.9790.

2.2.5. 2-Bromo-1-(4-fluorophenyl)ethanone (*5b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.41 (s, 2H), 7.14–7.18 (m, 2H), 8.00–8.03 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.13, 115.65, 115.83, 129.90, 129.92, 131.33, 131.40, 164.74, 166.78, 189.48. ^{19}F NMR (376 MHz, $CDCl_3$) δ −103.19. HRMS (ESI) *m*/*z* 215.9589, calc. for [C_8H_6BrFO] 215.9589.

2.2.6. 2-Bromo-1-(4-(trifluoromethyl)phenyl)ethanonenyl)ethanone (*6b*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.40 (s, 2H), 7.70–7.72 (m, 2H), 8.04–8.05 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.00, 30.02, 121.91, 124.08, 125.55, 125.58, 128.96, 134.61, 134.87, 136.15, 190.05. ^{19}F NMR (376 MHz, $CDCl_3$) δ −63.29. HRMS (ESI) *m*/*z* 265.9554, calc. for [$C_9H_6BrF_3O$] 265.9559.

*2.2.7. 2-Bromo-1-(3-hydroxyphenyl)ethanone (**7b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.46 (s, 2H), 6.11 (s, 1H), 7.12–7.14 (m, 1H), 7.35–7.38 (m, 1H), 7.52–7.53 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.83, 114.88, 121.09, 121.19, 129.82, 134.81, 155.92, 191.28. HRMS (ESI) *m*/*z* 213.9629, calc. for [$C_8H_7BrO_2$] 213.9635.

*2.2.8. 2-Bromo-1-(2-bromophenyl)ethanone (**8b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 4.49 (s, 2H), 7.35–7.37 (m, 1H), 7.39–7.42 (m, 1H), 7.46–7.48 (m, 1H), 7.62–7.64 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 33.59, 118.72, 127.22, 129.36, 132.17, 133.34. 138.18, 194.58. HRMS (ESI) *m*/*z* 275.8785, calc. for [$C_8H_6Br_2O$] 275.8788.

*2.2.9. 2-Bromo-1-(2-methoxyphenyl)ethanone (**9b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.91 (s, 3H), 4.38 (s, 2H), 6.95–7.02 (m, 2H), 7.47–7.51 (m, 1H), 7.79–7.82 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.46, 55.36, 111.20, 120.67, 124.39, 131.16, 134.43, 158.40, 191.92. HRMS (ESI) *m*/*z* 227.9786, calc. for [$C_9H_9BrO_2$] 227.9792.

*2.2.10. Ethyl 2-bromo-3-oxobutanoate (**10b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 1.29–1.32 (t, J = 7.1 Hz, 3H), 2.34 (s, 2H), 4.26–4.30 (q, J = 7.1 Hz, 2H), 4.75 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.53, 26.08, 48.74, 62.85, 164.78, 196.08. HRMS (ESI) *m*/*z* 207.9735, calc. for [$C_6H_9BrO_3$] 207.9738.

*2.2.11. Ethyl 2-bromo-3-oxo-3-phenylpropanoate (**11b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 1.23–1.26 (t, J = 7.1 Hz, 3H), 4.26–4.30 (q, J = 7.1 Hz, 2H), 5.65 (s, 1H), 7.48–7.51 (m, 2H), 7.61–7.62 (m, 1H), 7.98–8.00 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.51, 45.96, 62.97, 128.55, 128.83, 132.93, 133.94, 164.81, 187.75. HRMS (ESI) *m*/*z* 269.9892, calc. for [$C_{11}H_{11}BrO_3$] 269.9896.

*2.2.12. 2-Bromo-3,4-dihydronaphthalen-1(2H)-one (**12b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 2.45–2.54 (m, 2H), 2.90–2.94 (m, 1H), 3.28–3.35 (m, 1H), 4.73 (s, 1H), 7.27–7.29 (m, 1H), 7.34–7.37 (m, 1H), 7.51–7.54 (m, 1H), 8.08–8.10 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 25.72, 31.50, 50.06, 126.75, 128.30, 128.39, 129.51, 133.78, 142.62, 190.26. HRMS (ESI) *m*/*z* 223.9837, calc. for [$C_{10}H_9BrO$] 223.9843.

*2.2.13. 2-Bromo-2,3-dihydro-1H-inden-1-one (**13b**)*

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.40–3.44 (dd, J = 2.8, 18 Hz, 1H), 3.81–3.86 (dd, J = 7.6, 18 Hz, 1H), 4.64–4.66 (m, 1H), 7.41–7.45 (m, 2H), 7.65–7.68 (m, 1H), 7.83–7.84 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.59, 43.70, 124.72, 126.07, 127.93, 133.16, 135.63, 150.77, 199.32. HRMS (ESI) *m*/*z* 209.9680, calc. for [C_9H_7BrO] 209.9685.

*2.2.14. 2-Bromo-1-(furan-2-yl)ethanone (**14b**)*

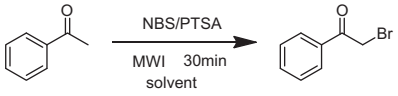
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.32 (s, 2H), 6.59–6.60 (m, 1H), 7.33–7.34 (m, 1H), 7.64 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.07, 112.91, 119.20, 147.34, 150.33, 180.40. HRMS (ESI) *m*/*z* 187.9473, calc. for [$C_6H_5BrO_2$] 187.9476.

*2.2.15. 2-Bromo-1-(thiophen-2-yl)ethanone (**15b**)*

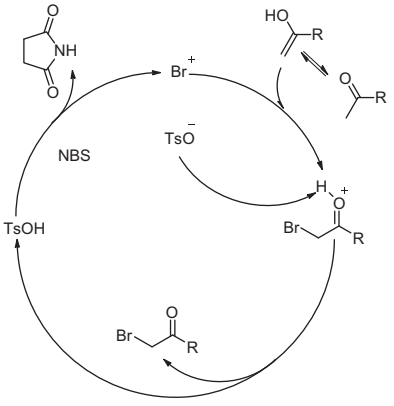
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.36 (s, 2H), 7.16–7.18 (m, 1H), 7.72–7.73 (m, 1H), 7.80–7.81 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.24, 128.05, 133.19, 134.93, 140.37, 184.07. HRMS (ESI) *m*/*z* 203.9244, calc. for [C_6H_5BrOS] 203.9250.

3. Results and discussion

On the onset of our study, we selected acetophenone as a model substrate for the optimization of the reaction conditions for the microwave promoted α-bromination of carbonyl compounds with NBS and PTSA (Scheme 2). In the absence of the PTSA catalyst, no reaction took place. While 10 mol% of PTSA was added, α-bromoacetophenone (Scheme 3) was obtained in 95% yield after 30 min of microwave irritation



Scheme 2 α-Bromination of acetophenone with NBS and PTSA under microwave irradiation.



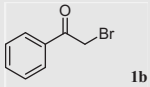
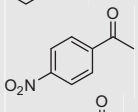
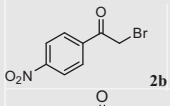
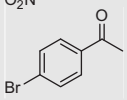
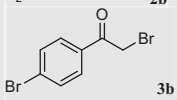
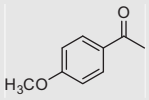
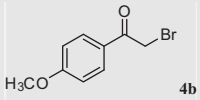
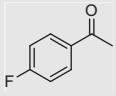
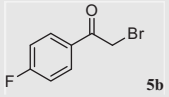
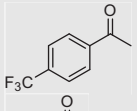
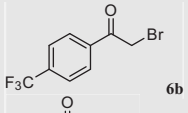
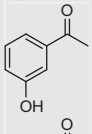
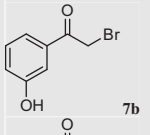
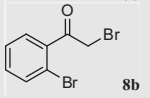
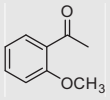
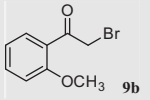
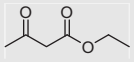
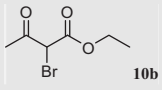
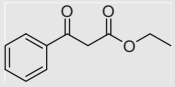
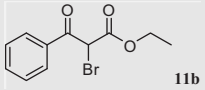
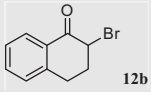
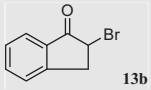
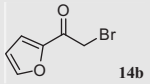
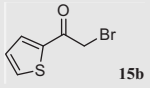
Scheme 3 Proposed mechanism for microwave assisted α-bromination of carbonyl compounds with NBS catalyzed by PTSA.

Table 1 Effect of solvent and temperature on the α-bromination of acetophenone with NBS under microwave irradiation[a].

Entry	Solvent	T (°C)	Conversion %[b]
1	CH_3CN	80	46
2	Et_2O	80	35
3	THF	80	52
4	*n*-hexane	80	30
5	H_2O	80	< 5
6	CH_2Cl_2	80	95
7	CH_2Cl_2	60	60
8	CH_2Cl_2	100	80

[a] Substrate (0.2 mmol), NBS (0.2 mmol, 1 equiv), PTSA (0.02 mmol, 10 mol%), solvent (2 mL).
[b] Determined by ^{1}H-NMR.

Table 2 Microwave promoted α-bromination of carbonyl compounds with NBS catalyzed by PTSA.

Entry	Substrate	Product	Yield %[b]
1		 **1b**	94
2		 **2b**	91
3		 **3b**	95
4		 **4b**	90
5		 **5b**	89
6		 **6b**	85
7		 **7b**	93
8		 **8b**	91
9		 **9b**	94
10		 **10b**	93
11		 **11b**	96
12		 **12b**	96
13		 **13b**	93
14		 **14b**	93
15		 **15b**	95

[a]Substrate (0.2 mmol), NBS (0.2 mmol, 1 equiv), PTSA (0.02 mmol, 10 mol%), Solvent (2 mL), 80 °C, MWI time (30 min).
[b] Isolated yield after column chromatography.

Table 3 Comparative results under microwave or thermal conditions.

Product	Method	Time (min)	Temp (°C)	Conversion %[b]
1b	MW	30	80	94
1b[a]	Δ	30	80	31
1b[a]	Δ	120	80	62
3b	MW	30	80	95
3b[a]	Δ	30	80	33
3b[a]	Δ	120	80	55

[a] Substrate (0.2 mmol), NBS (0.2 mmol, 1 equiv), PTSA (0.02 mmol, 10 mol%), CH_2CL_2 (2 mL).
[b] Determined by ^{1}H-NMR.

at 80 °C. Among all solvents examined, dichloromethane was identified to be the best choice. The reaction proceeded smoothly with excellent selectivity in dichloromethane: only the monobromo product was obtained (Table 1, entries 5), whereas the reactions in CH_3CN, Et_2O, THF and *n*-hexane afford significantly lower yields under similar conditions (Table 1, entries 1–4). The best result was obtained at the reaction temperature of 80 °C, but the conversion decreased at either a lower or higher temperature.

After optimizing the reaction conditions, we tested the generality of this microwave promoted α-bromination of carbonyl compounds with NBS and PTSA for various carbonyl compounds. The results are summarized in Table 2. Both electron-donating (entries 4, 7, 9) and electron-withdrawing (entries 2, 3, 5, 6, and 8) substituents on the phenyl ring of acetophenones at either the *para*-, *ortho*- or *meta*-position show little influence on the excellent selectivity and reactivity. Bromo, fluoro, nitro, methoxyl, and the hydroxyl group containing acetophenones were monobrominated selectively to afford the corresponding α-bromo ketones in excellent yields. The reaction was next extended to 1,3-dicarbonyl compounds, because α-brominated 1,3-dicarbonyl compounds are key intermediates and building blocks in organic synthesis (Verhé and De Kimpe, 2010). The reactions proceeded smoothly to produce α-brominated 1,3-dicarbonyl compounds in high yields (entries 10 and 11). For the carbocyclic ketones, such as 1-indanone and 1-tetralone, α-bromination products were also obtained in high yields (entries 12 and 13). It is noteworthy that heteroaromatic rings such as furan and thiophene were also compatible with this reaction condition (entries 14 and 15).

To interrogate the differences between the microwave irradiation and the thermal heating, synthesis of bromoacetophenones **1b** and **3b** using a thermostated oil-bath under otherwise identical conditions as those employed for the microwave-assisted method were studied (Table 3). Lower yields with a longer reaction time were observed under thermal conditions, suggesting that the microwave irradiation condition offers much better efficacy.

4. Conclusion

In summary, we have demonstrated an efficient methodology for the synthesis of α-bromoalkanones from carbonyl compounds using NBS catalyzed by *p*-toluenesulfonic acid under microwave irradiation conditions. This method can be applied to a wide range of carbonyl compounds.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.arabjc.2014.01.024.

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