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α -Iodination of Ketones with MnO_2/I_2 Reagent Combination: A New Environmentally Friendly Procedure

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Abstract: In alcoholic media, α -iodination of ketones was accomplished using MnO_2/I_2 reagent combination in a new environmentally friendly procedure. The reactions carried out under thermal conditions or microwave irradiation afforded α -iodoketones in reasonable to good yields.

Key words: iodine, manganese oxide, halogenation, ketones.

α -Iodoketones have attracted a great deal of interest, due to their application as reaction intermediates in various organic transformations.¹ In contrast to their chloro- or bromo- analogs, α -iodoketones have received little attention. While this is partly due to their relative instability, it should also be noted that there are only few satisfactory syntheses available. Their preparation, starting from ketones and related derivatives (enol ethers, enol esters, halo-ketones...) have been achieved under various procedures. Typically, they were obtained by treatment of ketones with elemental iodine in the presence of a strong base.² More often, indirect routes were used including either iodination of enol acetates with iodine chloride or N-iodosuccinimide (NIS)³, as well as iodination of enol ethers or silylenol-ethers with NaI .⁴ α -Iodoketones were also obtained by direct exchange of bromine⁵ or chlorine⁶ with iodine using metallic iodides, principally NaI , but this methodology is subject to pronounced steric effects. Marginal routes such as decomposition of diazo-ketones in the presence of I_2 or KI were also described.⁷

Recently, many efforts have been made to the development of efficient reaction conditions towards α -iodination of ketones including oxidizing agents able to convert molecular iodine to a more reactive species, notably the iodonium (I^+) ion (e.g. thallium (I) acetate,⁸ silver acetate,⁹ pyridinium chlorochromate,¹⁰ CAN ,¹¹...). While these reactions are suitable methods, most of them suffer from drawbacks such as cumbersome workup procedures and the use of toxic or expensive oxidizing agents is very often required. Despite the various efforts already carried out, it remains desirable to develop novel iodination methods of practical value.

As part of a program aimed at developing new reagents for the preparation of α -haloketones¹², we have recently reported an efficient α -iodination of ketones using SeO_2/I_2 couple^{12a} but again, the reaction conditions used required the presence of toxic selenium dioxide.¹³ To circumvent this drawback, we have reinvestigated this reaction with several couples oxidant/ I_2 and found that commercially available MnO_2 ¹⁴ promoted α -iodination of carbonyl compounds with I_2 in alcoholic media. Herein we report the results of this study under classical thermal conditions as well as under microwave irradiation.

This new environmentally friendly procedure, which allows the formation of the corresponding α -iodoketones in good yields, is characterized by the mildness of reaction conditions and inexpensive reactants. Moreover, the ready availability associated with the ease of handling MnO_2/I_2 reagents provide a particular advantage of the present method.

At first, we studied the α -iodination reaction of 5-methoxytetralone **1a** as a model substrate under conventional thermal conditions. We found that the yield of α -iodoketone **2a** depends greatly on the solvent, temperature, and molar ratio of the MnO_2/I_2 reagent. Table 1 summarizes the results of these experiments.

Initially, the reaction of 5-methoxytetralone **1a** with MnO_2 (2 equiv) and I_2 (1 equiv) was evaluated in acetonitrile, which has been shown to be the best solvent for the α -iodination with SeO_2/I_2 system.^{12a} Thus, at room temperature no reaction occurred and starting material **1a** was recovered unchanged. However, in boiling MeCN , α -iodination proceeded smoothly to give the corresponding α -iodotetralone **2a** in moderate yield (46%, entry 1). Changing the solvent to AcOH resulted in lower yield (25%, entry 2). Of the other solvents tested, EtOH gave the highest yield of **2a** (76%, entry 4). Treatment of **1a** with MnO_2/I_2 in *n*- BuOH , *i*- BuOH , or H_2O at 80 °C gave unsatisfactory yields of product **2a** (entries 5-7). It should be noted that all attempts to react **1a** with I_2 (1 equiv) in refluxing ethanol without MnO_2 were unsuccessful even after prolonged reaction times (entry 8), only unchanged starting material was recovered. Next, the influence of $\text{MnO}_2/\text{I}_2/\text{1a}$ molar ratio was also investigated. When the amount of MnO_2 was increased (6 equiv) the yield of **2a** was not improved (72%, entry 10). Similar results were obtained when the amount of I_2 was doubled (70%, entry 11). After a lot of trials, the best yield of α -iodinated ketone **2a** was obtained when **1a** was stirred in refluxing EtOH for 18h¹⁵ with MnO_2 (1.1 equiv) and I_2 (1.5 equiv) (83%, entry 12). Nevertheless, even if the yield of **2a** was improved, some starting material¹⁶ ($\pm 10\%$) was recovered unchanged and longer reaction time (48 h) resulted in lower yield of iodinated ketone **2a** (74%, entry 13). We then decided to apply subsequently these optimum conditions (e.g. $\text{MnO}_2/\text{I}_2 = 1.1/1.5$ in refluxing EtOH for 18h) to other commercially available tetralones **1b-d**. As expected, α -iodotetralones **2b-d** were obtained in fair to good yields (76-80%, entries 14-16). It should be noted that in all cases studied, while using an excess of I_2 , no product resulting from iodination of activated aromatic ring could be detected.

In the last decade, organic reactions promoted by microwave irradiation received much attention due to the advantages over conventional methods in term of rapid reaction rates and improved conversion. For that purpose and concerning iodination reactions, Lee and Bae¹⁷ reported an efficient α -iodination of carbonyl compounds using NIS and *p*-toluenesulfonic acid under microwave irradiation.

Because no aromatic ring iodination and no α - α -diiodination were detected under the aforementioned optimum thermal conditions and, in order to succeed in α -iodination with shorter reaction times, we have examined the α -iodination of arylated ketones using microwave irradiation. The molar ratio $\text{MnO}_2/\text{I}_2 = 1.1/1.5$ was fixed and attempts were carried out in ethanol at 78 °C, as for the above thermal classical conditions. In this way, we were pleased to observe that a reaction time of 5 min was sufficient to reach up to 95% conversion of starting ketones. The results summarized in Table 2 indicate that α -iodoketones **2** are produced in good yields.

Treatment of various tetralones **1** with MnO_2/I_2 for 5 minutes at 78 °C afforded the corresponding α -iodotetralones in fair to good yields and the results displayed in Table 2 clearly indicate the general applicability of the procedure. We have noticed that, on the contrary of thermal classical conditions, *n*-BuOH and *i*-BuOH could be used successfully as solvent for the α -iodination process under microwave irradiation. For examples, when tetralones **1b-d** were treated with the couple MnO_2/I_2 in *n*-BuOH instead of EtOH, the yield was improved (compare entries 2/3, 4/5, and 6/7). We also successfully applied these conditions to functionalized tetralones and yields were satisfactory using *n*-BuOH or *i*-BuOH (entries 10-17). Thus, iodination of allyl-substituted tetralone **1f** was achieved successfully without iodination of the allylic moiety (80%, entry 11). We also observed that the reaction with pivaloyl ester **1g** was efficient but principally when hindered alcohol solvents were used in order to prevent any *trans* esterification as observed with EtOH (compare entries 12-14). Moreover, under these conditions, if no reaction occurred with 6-hydroxy-tetralone, its silylated homologue **1h** was transformed with satisfactory yield (60%, entry 16). Reactions were also performed with either chromanone **1i** or indanone **1j** and the corresponding α -iodo compounds were obtained in reasonable yields (resp. 75% and 66%) in *n*-BuOH (entries 19 and 21).

Finally, this iodination reaction was evaluated in the case of acyclic carbonyl derivatives particularly with aryl-alkyl ketone **1k** and aryl-benzyl ketone¹⁸ **1l** (Scheme 1).

First attempts in EtOH showed that iodination of **1k** was effective even if some starting material (easily separable on SiO_2 column chromatography) was recovered unchanged after stirring for 10 minutes under microwave irradiation (conversion yield 67%). More interestingly, under these conditions, the aryl-benzyl ketone **1l** contrary to expectations was transformed into the ethyl ester **3l**¹⁹. This result is consistent with an EtOH esterification of the intermediate **2l** followed by an aryl migration and

a subsequent substitution of the iodine atom. The development of this latter transformation is currently under study in our Laboratory and will be reported in due course.

In conclusion, MnO_2/I_2 in alcoholic media is an efficient reagent combination for α -iodination of ketone derivatives. This method is complementary to the existing procedures and sometimes could be the method of choice because of its simplicity, rapidity, and the high yields obtained under thermal classical methods as well as under microwave irradiation. Moreover, the use of the non toxic heterogeneous mineral MnO_2 offers the advantage to be easily removed from the reaction medium by simple filtration and, therefore, purifications of products are easier.

IR spectra were recorded on a Perkin-Elmer 841 spectrometer. ¹H and ¹³C NMR spectra were measured with a Bruker AC 200 (200 MHz and 50 MHz, for ¹H and ¹³C, respectively). ¹H chemical shifts are reported in ppm from an internal standard TMS or residual chloroform ($\delta = 7.27$ ppm). ¹³C chemical shifts are reported from the central peak of deuteriochloroform ($\delta = 77.1$ ppm). Elemental analyses were performed with a Perkin-Elmer 240 analyzer. Melting points were recorded on Büchi B-450 apparatus and are uncorrected. Analytical TLC were performed on Merck pre-coated silica gel 60F plates. Merck silica gel 60 (230-400 mesh) was used for column chromatography. All microwave experiments were performed using an Emrys Optimizer in 2-5 mL pyrex reaction vessels. Each contained a Teflon stir bar and Teflon coated reaction vessel cap. MnO_2 was purchased from Acros (99+% powder).

Thermal conditions, typical procedure:

To a flask containing α -tetralone (1 mmol; 1 equiv) and EtOH (10 mL) were added MnO_2 (96 mg; 1.1 equiv) and I_2 (371 mg; 1.5 equiv). Then the reaction mixture was stirred at 78 °C for 18 h and filtered over a pad of celite to remove manganese derivatives. The solution was diluted with H_2O (30 mL) and extracted with CH_2Cl_2 . The organic phase was washed with 30 mL of a aqueous saturated $\text{Na}_2\text{S}_2\text{O}_3$ solution and then with H_2O . After drying, the solvents were removed and the residue purified by column chromatography to yield **2** (light sensitive).

Microwave conditions, typical procedure:

To an Emrys Optimizer 2-5 mL pyrex reaction vessel were added ketone (1 mmol), MnO_2 (96 mg; 1.1 equiv), I_2 (371 mg; 1.5 equiv) in the appropriate solvent (1 mL). The reaction vessel was then placed in the Emrys Optimizer and exposed to microwave irradiation according to the following specifications: temperature: 78 °C, time 600 s, fixed hold time: on, sample absorption: high, pre-stirring: 60 s. After cooling to room temperature, the crude mixture was treated as above.

3,4-Dihydro-2*H*-2-iodo-6-methoxy-naphthalen-1-one (**2c**)

Beige crystals; mp 104-105 °C.

IR (neat): 1657, 1593, 1258, 856 cm^{-1} .

¹H NMR (CDCl_3 , 200 MHz) δ = 8.02 (d, 1H, *J* = 8.8 Hz), 6.86 (dd, 1H, *J* = 8.8 Hz, *J* = 2.1 Hz), 6.70 (d, 1H, *J* = 2.1 Hz), 4.95 (t, 1H, *J* = 3.6 Hz), 3.84 (s, 3H), 3.18-3.01 (m, 2H), 2.31-2.00 (m, 2H).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 190.7, 164.0, 145.3, 131.1, 122.7, 113.7, 112.4, 55.4, 32.8, 30.9, 28.2.

Anal. Calcd for $\text{C}_{11}\text{H}_{11}\text{IO}_2$: C, 43.73; H, 3.67. Found C, 43.91; H, 3.77.

5-Bromo-3,4-dihydro-2H-2-iodo-naphthalen-1-one (2e)

Beige crystals; mp 78–81 °C.

IR (neat): 1672, 1439, 610 cm^{-1} .

^1H NMR (CDCl_3 , 200 MHz) δ = 8.14 (d, 1H, J = 7.8 Hz), 7.84 (d, 1H, J = 7.8 Hz), 7.32 (t, 1H, J = 7.8 Hz), 5.04 (t, 1H, J = 3.8 Hz), 3.25–2.92 (m, 2H), 2.45–2.11 (m, 2H).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 191.0, 141.6, 137.7, 131.2, 128.0, 127.9, 124.5, 31.3, 28.6, 28.3.

Anal. Calcd for $\text{C}_{10}\text{H}_8\text{BrIO}$: C, 34.22; H, 2.30. Found C, 34.31; H, 2.38.

6-Allyl-3,4-dihydro-2H-2-iodo-naphthalen-1-one (2f)

Yellow crystals; mp 67–71 °C.

IR (neat): 1659, 1590, 1493, 1431, 1245, 1163, 1082 cm^{-1} .

^1H NMR (CDCl_3 , 200 MHz) δ = 8.10 (d, 1H, J = 8.8 Hz), 6.87 (dd, 1H, J = 8.8 Hz J = 2.2 Hz), 6.73 (d, 1H, J = 2.2 Hz), 6.13–5.94 (m, 1H), 5.46–5.29 (m, 2H), 4.97 (t, 1H, J = 3.6 Hz), 4.59 (d, 2H, J = 5.0 Hz), 3.19–3.03 (m, 1H), 2.86–2.74 (dt, 1H, J = 17.0 Hz J = 3.6 Hz), 2.33–2.01 (m, 2H).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 190.7, 163.1, 145.2, 132.3, 131.2, 122.8, 188.2, 114.3, 113.3, 68.6, 32.9, 30.8, 28.3.

Anal. Calcd for $\text{C}_{13}\text{H}_{13}\text{IO}_2$: C, 47.58; H, 3.99. Found C, 47.66; H, 3.89.

2,2-Dimethyl-propionic acid-6-iodo-5-oxo-5,6,7,8-tetrahydro-naphthalen-2-yl-ester (2g)

Beige crystals; mp 124–125 °C.

IR (neat): 1752, 1672, 1600, 1246, 1104 cm^{-1} .

^1H NMR (CDCl_3 , 200 MHz) δ = 8.13 (d, 1H, J = 8.4 Hz), 7.07–7.00 (m, 2H), 5.01 (t, 1H, J = 3.6 Hz), 3.24–3.05 (m, 1H), 2.95–2.75 (dt, 1H, J = 17.0 Hz J = 3.6 Hz), 2.34–2.06 (m, 2H), 1.37 (s, 9H).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 190.6, 176.3, 155.5, 144.5, 130.5, 126.8, 121.2, 120.5, 39.1, 32.5, 30.1, 27.8, 26.9 (3).

Anal. Calcd for $\text{C}_{15}\text{H}_{17}\text{IO}_3$: C, 48.40; H, 4.60. Found C, 48.43; H, 4.72.

6-(*t*-Butyl-dimethylsilanyloxy)-3,4-dihydro-2H-2-iodo-naphthalen-1-one (2h)

Beige crystals; mp 58–60 °C.

IR (neat): 1676, 1597, 1254, 966 cm^{-1} .

^1H NMR (CDCl_3 , 200 MHz) δ = 8.00 (d, 1H, J = 8.6 Hz), 6.78 (dd, 1H, J = 8.6 Hz, J = 2 Hz), 6.68 (d, 1H, J = 2 Hz), 4.97 (t, 1H, J = 3.4 Hz), 3.17–3.01 (m, 1H), 2.83–2.71 (m, 1H), 2.32–2.02 (m, 2H), 0.99 (s, 9H), 0.24 (s, 6H).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 190.8, 160.8, 145.2, 131.1, 123.2, 119.2, 118.9, 32.9, 30.8, 28.0, 25.5 (3), 18.1, –4.4 (2).

Anal. Calcd for $\text{C}_{16}\text{H}_{23}\text{IO}_2\text{Si}$: C, 47.76; H, 5.76. Found C, 47.62; H, 5.69.

2-Iodo-1-(4-methoxyphenyl)pentan-1-one (2k)

Yellow oil

IR (neat): 1667, 1598, 1253, 842 cm^{-1} .

^1H NMR (CDCl_3 , 200 MHz) δ = 7.98 (d, 2H, J = 8.5 Hz), 6.94 (d, 2H, J = 8.5 Hz), 5.29 (t, 1H, J = 7.4 Hz), 3.87 (s, 3H), 2.13 (m, 2H), 1.50–1.26 (m, 4H), 0.88 (t, 3H, J = 14.6 Hz).

^{13}C NMR (CDCl_3 , 50 MHz) δ = 193.2, 163.8, 130.9 (2), 126.9, 114.0 (2), 55.5, 34.9, 31.7, 25.9, 22.1, 13.8.

Anal. Calcd for $\text{C}_{13}\text{H}_{17}\text{IO}_2$: C, 47.00; H, 5.16. Found C, 46.99; H, 5.17.

(4-Methoxyphenyl) phenylacetic acid ethyl ester (3l)

3l showed identical spectral properties than previously reported (see ref 19).

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Graphical Abstract **α -Iodination of Ketones with MnO_2/I_2 Reagent Combination: A New Environmentally Friendly Procedure**

Chemical reaction scheme showing the iodination of 2-substituted-1,2,3,4-tetrahydronaphthalen-1-one (**1a-d**) to 2-iodo-2-substituted-1,2,3,4-tetrahydronaphthalen-1-one (**2a-d**). The reaction conditions are $\text{MnO}_2 / \text{I}_2$ in a solvent, over time, at temperature T° .

Entry	Ketones	MnO ₂ ^a (equiv)	I ₂ (equiv)	Solvent	Time (h)	T (°C)	Yield (%) ^b
1	1a : R = 5-OMe	2	1	CH ₃ CN	12	82	46 ^c
2	1a	2	1	AcOH	12	80	25
3	1a	2	1	DMF	12	90	43
4	1a	2	1	EtOH	12	78	76
5	1a	2	1	<i>n</i> -BuOH	12	80	40
6	1a	2	1	<i>i</i> -BuOH	12	80	49
7	1a	2	1	H ₂ O	12	80	44
8	1a	0	1	EtOH	18	78	0
9	1a	1	1	EtOH	18	78	76
10	1a	6	1	EtOH	18	78	72
11	1a	1	2	EtOH	18	78	70
12	1a	1.1	1.5	EtOH	18	78	83
13	1a	1.1	1.5	EtOH	48	78	74
14	1b : R = 6-OMe	1.1	1.5	EtOH	18	78	76
15	1c : R = 7-OMe	1.1	1.5	EtOH	18	78	80
16	1d : R = H	1.1	1.5	EtOH	18	78	79

^c No reaction occurred at room temperature.

Table 2. Microwave irradiation promoted preparation of α -iodocarbonyl compounds **2**

$\text{R}-\text{C}_6\text{H}_4-\text{C}(=\text{O})-\text{CH}_2-\text{CH}_2-\text{X} \xrightarrow[\text{Solvent, MWI, 5 min, 78 } ^\circ\text{C}]{+\text{MnO}_2 (1.1 \text{ equiv}) + \text{I}_2 (1.5 \text{ equiv})} \text{R}-\text{C}_6\text{H}_4-\text{C}(=\text{O})-\text{CH}(\text{I})-\text{CH}_2-\text{X}$

1a-j **2a-j**

Entry	Ketone	Product	Solvent	Yield ^a %
1			EtOH	91
2			EtOH	69
3			<i>n</i> -BuOH	82
4			EtOH	82
5			<i>n</i> -BuOH	85
6			EtOH	67
7			<i>n</i> -BuOH	92
8			EtOH	71
9			<i>n</i> -BuOH	86
10			EtOH	66
11			<i>n</i> -BuOH	80
12			EtOH	35
13			<i>n</i> -BuOH	58
14			<i>i</i> -BuOH	70
15			EtOH	56
16			<i>n</i> -BuOH	60
17			<i>i</i> -BuOH	33
18			EtOH	58
19			<i>n</i> -BuOH	75
20			EtOH	55
21			<i>n</i> -BuOH	66
22			<i>i</i> -BuOH	51

^a isolated yields.

Scheme 1. Iodination of aryl ketones **1k-l**

* conversion yield (not optimized).