Hypervalent iodine oxidation of α-substituted 2,4-dihydroxyacetophenones: synthesis of novel *o*-iodophenoxy ethers via rearrangement of iodonium ylides

Om Prakash, Vijay Sharma, and Madan P. Tanwar

Abstract: α-Substituted 2,4-dihydroxyacetophenones (**1a–1i**) have been oxidized with phenyliodonium diacetate (PIDA) under three conditions (*i*) PIDA–KOH–MeOH (basic) at 0°C, (*ii*) PIDA–MeOH (neutral) at reflux temperature, and (*iii*) PIDA–AcOH (acidic) at reflux temperature, to afford novel *o*-iodophenoxy ethers (**4a–4i**) via rearrangement of respective iodonium ylides (**5a–5i**).

Key words: hypervalent iodine reagents, phenyliodonium diacetate, α-substituted 2,4-dihydroxyacetophenones, α-substituted 2-hydroxy-3-iodo-4-phenoxyacetophenones, iodonium ylides rearrangement.

Résumé : On a oxydé des 2,4-dihydroxyacétophénones α-substituées (**1a–1i**) à l'aide du diacétate de phényliodonium (« PIDA ») dans trois conditions : (*i*) PIDA–KOH–MeOH (basique), à 0°C; (*ii*) PIDA–MeOH (neutre), à la température de reflux et (*iii*) PIDA–AcOH (acide), à la température de reflux; ces oxydations conduisent à de nouveaux éthers *o*-iodophénoy (**4a–4i**) par le biais d'un réarrangement des ylures d'iodonium respectifs (**5a–5i**).

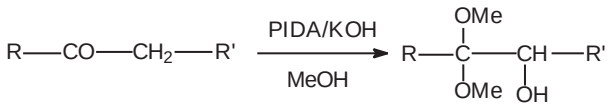
Mots clés : réactifs hypervalents de l'iode, diacétate de phényliodonium, 2,4-dihydroxyacétophénones α-substituées, 2-hydroxy-3-iodo-4-phénoxyacétophénones α-substituées, réarrangement d'ylures d'iodonium.

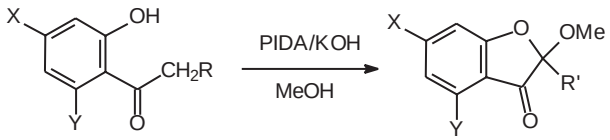
[Traduit par la Rédaction]

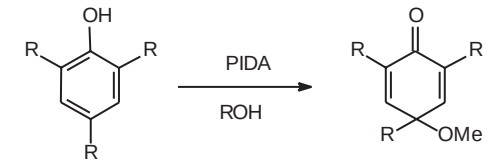
Introduction

A surge of interest in the use of hypervalent iodine reagents in organic synthesis has been witnessed by its explosive growth during few last decades (1–5). Major areas of interest are hypervalent iodine oxidation of carbonyl compounds (4), phenolic oxidations (2*a*, 5), oxidative rearrangements (1*e*), etc. These studies have provided a large number of new and convenient methods for the synthesis of a wide variety of organic compounds, including various heterocyclic compounds and bridgehead compounds (3, 6).

A notable hypervalent iodine oxidation of enolizable ketones with PIDA and potassium hydroxide in methanol leads to the formation of α-dimethylacetals (7, 8) (eq. [1]). However, the presence of a phenolic group at the *ortho* position of ketones such as acetophenone, etc. affords coumaran-3-ones by intramolecular participation in this oxidation (9, 10) (eq. [2]). On the other hand, hypervalent iodine oxidation of various phenols has provided attractive routes for the synthesis of valuable *p*-quinones, *o*-quinones, quinone acetals, etc., which are otherwise difficult to obtain (eq. [3]) (11, 12).

[1] 

[2] 

[3] 

Results and discussion

Based upon these observations, we became interested in oxidizing those compounds containing *ortho* and *para* phenolic groups and an enolizable ketonic moiety, for example, 5-acetyl-2,4-dihydroxyacetophenone (**1a**), with phenyliodonium diacetate (PIDA) and KOH in MeOH. The attempted reaction yielded a yellow crystalline solid (mp 143–145°C; yield 75%), which separated out during the stirring of the reaction mixture. Instead of obtaining bis-coumaranones **2** (via a route analogous to eq. [2]) or compound **3** (via a route analogous to eq. [3]), interestingly, a novel formation of 5-

Received April 7, 1998.[1]

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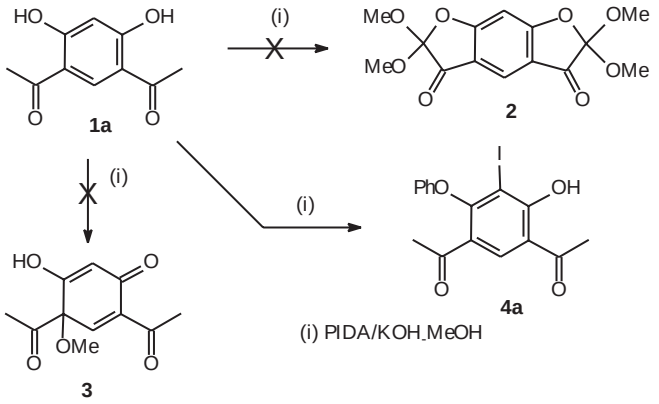
Table 1. Physical data for **4a–4i**, **5c**, and **5g**.

Compound no.	X	R	Melting point (°C)	Yield (%)[a]		
				(i)[b]	(ii)[b]	(iii)[b]
4a	$COCH_3$	H	143–145	75	40	64
4b	H	H	116–118	20	10	15
4c	NO_2	H	190–191	77	40	60
4d	Br	H	160–161	35	10	25
4e	H	CH_3	136–138	68	40	55
4f	COC_2H_5	CH_3	119–120	75	40	60
4g	NO_2	CH_3	190–192	48	27	41
4h	Br	CH_3	146–147	38	10	20
4i	H	C_6H_5	129–130	65	52	58
5c	NO_2	H	>200	82	—	—
5g	NO_2	CH_3	196–198	56	—	—

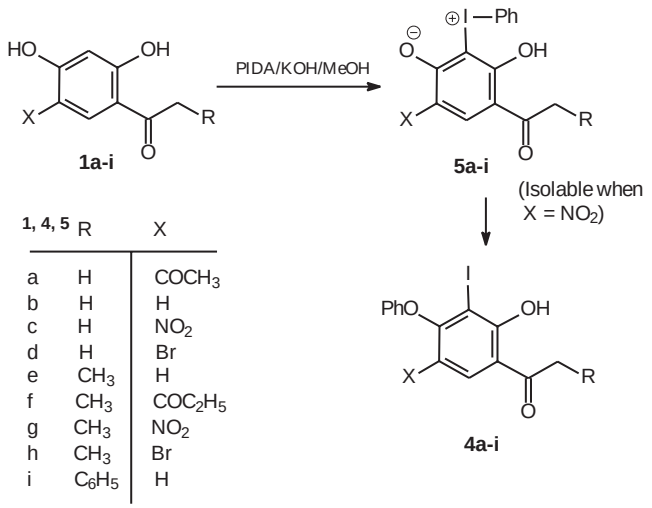
[a]Yields are based upon the pure products.

[b]Reaction conditions: (i) PIDA – KOH in MeOH; (ii) PIDA–MeOH; (iii) PIDA–AcOH.

Scheme 1.



Scheme 2.



acetyl-2-hydroxy-3-iodo-4-phenoxyacetophenone (**4a**) was observed (Scheme 1).

A preliminary account of this study was reported earlier (13). This observation, coupled with the fact that the rearranged products from this reaction i.e., *o*-iodo ethers, are potential precursors for the synthesis of a variety of interesting compounds such as dibenzofurans, which are associated with diverse physiological activities (14), prompted us to study the scope of this novel route.

In the present study, a full account of our detailed investigations on the oxidation of α-substituted 2,4-dihydroxyacetophenones (**4a–4i**) with phenyliodonium diacetate under different conditions is described.

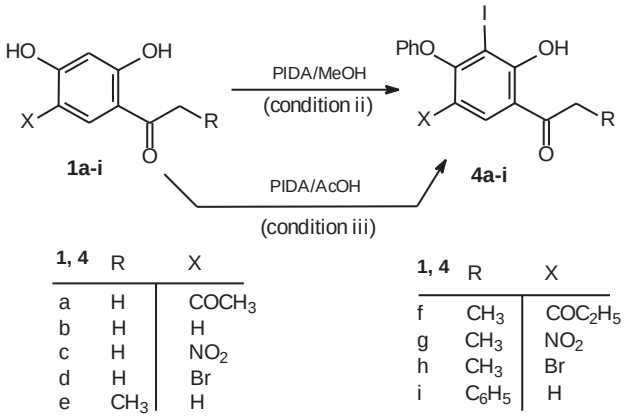
Accordingly, we first carried out oxidation of 2,4-dihydroxyacetophenones (**1b–1d**) with PIDA – KOH in MeOH, to afford the corresponding rearranged *o*-iodo ethers (**4b–4d**). This rearrangement occurred via the intermediacy of iodonium ylides of the type **5b–5d** as evidenced by the isolation of ylide **5c** as a high-melting yellow solid, from the oxidation of ketone **1c**. Acidification of **5c** with hydrochloric acid, followed by refluxing the methanolic solution of the resultant solid (mp 153–154°C), afforded a rearranged product **4c** (mp 190–191°C; yield 77%). However, our attempt to isolate similar intermediates **5a**, **5b**, **5d** in other cases ended up with no success, as only the rearranged products **4a**, **4b**, **4d** were obtained from these reactions. The physical data for the products **4a–4d** are listed in Table 1.

Similarly, oxidation of 2,4-dihydroxy-5-nitropropiophenone (**1g**) with PIDA with KOH in MeOH at 0°C (condition (i)) afforded iodonium ylide **5g** as a yellow crystalline solid (based on ^{1}H NMR and mp). This ylide, on acidification with hydrochloric acid followed by refluxing, gave 2-hydroxy-3-iodo-4-phenoxy-5-nitroacetophenone (**4g**) in 48% yield.

2,4-dihydroxypropiophenone (**1e**), 2,4-dihydroxy-5-propionylpropiophenone (**1f**), 5-bromo-2,4-dihydroxypropiophenone (**1h**), and 2,4-dihydroxydeoxybenzoin (**1i**) also underwent smooth transformation to 2-hydroxy-3-iodo-4-phenoxypropiophenone (**4e**) 2-hydroxy-3-iodo-4-phenoxy-5-propionylpropiophenone (**4f**), 5-bromo-2-hydroxy-3-iodo-4-phenoxypropiophenone (**4h**), and 2-hydroxy-3-iodo-4-phenoxydeoxybenzoin (**4i**), respectively. The intermediates, iodonium ylides **5e**, **5f**, **5h**, and **5i**, could not be isolated.

In view of the marked effect of conditions on the course of various hypervalent iodine-mediated transformations, it was considered worthwhile to investigate the role of other reaction conditions on the oxidation of compounds **1a–1i**.

Scheme 3.



1, 4	R	X
a	H	$COCH_3$
b	H	H
c	H	NO_2
d	H	Br
e	CH_3	H

1, 4	R	X
f	CH_3	COC_2H_5
g	CH_3	NO_2
h	CH_3	Br
i	C_6H_5	H

Thus substrate **1a** was also oxidized with PIDA under condition (ii), viz. PIDA in methanol (neutral) at reflux temperature, and under condition (iii), PIDA in acetic acid (acidic) at reflux temperature (Scheme 3). The product, obtained after the usual work-up under these conditions, was characterized as **4a,** which was found to be identical to that prepared under reaction condition (i). However, yields of the products were found to be lower in conditions (ii) and (iii) than in condition (i).

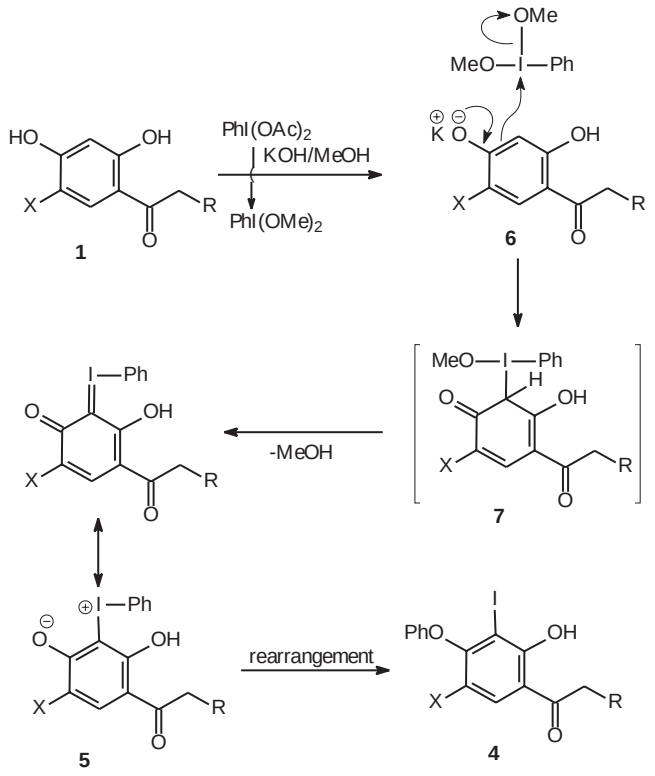
Similarly, applying conditions (ii) and (iii), other phenolic ketones **1b–1i** were oxidatively rearranged to the corresponding *o*-iodophenoxy ethers **4b**–**4i**. The physical data for the products (% yields and melting points under different conditions) are given in Table 1.

A plausible mechanistic pathway for **1**→**4** via rearrangement of iodonium ylide (15, 16) of type **5** is outlined in Scheme 4. Under strong basic conditions (KOH in MeOH) PIDA is converted into dimethoxyiodobenzene (17), ($PhI(OMe)_2$), while substrate **1** changes in situ into the corresponding potassium phenolate **6**. Dimethoxyiodobenzene attacks at position 3 of phenolate **6** to form intermediate **7**, which loses methanol to produce iodonium ylide **5**. The iodonium ylide **5** finally rearranges to product **4**. It is to be noted that under conditions (ii) and (iii) the electrophilic attack of PIDA itself takes place, on substrate **1** (in place of dimethoxyiodobenzene), to afford the final product via intermediates of type **5** and **7**. Since PIDA in acetic acid gives the products in lower yields (5–18%), basic conditions presumably assist the removal of a proton from intermediate **7**. Thus $PhI(OMe)_2$ may not be an obligatory reagent for the products **4** and **5**.

It is relevant to mention that while our detailed investigation of this rearrangement was in progress and a preliminary communication had also appeared (13), Mallik and Mallik reported the oxidation of 2,4-dihydroxyacetophenone and other phenolic ketones with PIDA–MeOH at reflux temperature to afford *o*-iodophenoxy ethers (18). Although these reaction conditions also afforded the same *o*-iodo ethers **4**, PIDA – KOH in MeOH (our condition i) gave better results in terms of yields and isolation of products.

Finally, the formation and rearrangement of phenolic iodonium ylides containing an enolizable ketone moiety offers a novel route for the synthesis of new *o*-iodophenoxy ethers that are otherwise difficult to prepare using conventional methods. The most striking feature of this approach is that the enolizable ketonic group present in compounds **1a–1i** remains unaffected under these reaction conditions, which normally oxidize the α-position of the enolizable ketones (7, 8). Another noteworthy feature of this oxidative rearrangement is that phenolic ketones having electron-withdrawing substituents ($COCH_2R$ and NO_2) at the 5-position yield better results. The products obtained from this study are potential precursors of various organic compounds, especially heterocyclic compounds. For example, *o*-iododiaryl ethers are useful intermediates for dibenzofuran synthesis as they could be readily cyclized either photochemically (15) or chemically through the use of Ph(II) salts(16), *n*-Bu_3SnH–AIBN (19), etc.

Scheme 4.



Experimental

Melting points were measured in open capillaries in a sulfuric acid bath and are uncorrected. IR spectra were recorded on a Beckmann IR-20 spectrophotometer. ^1H NMR spectra were taken on a Perkin Elmer (90 MHz) instrument. EI mass spectra were recorded on a mass spectrophotometer operating at 70 eV. 2,4-Dihydroxyacetophenone (**1b**) (20) and its analogues (5-bromo-2,4-dihydroxyacetophenone (**1d**) (21), 2,4-dihydroxy-5-nitroacetophenone (**1c**) (22), 5-acetyl-2,4-dihydroxyacetophenone (**1a**) (20, 21)), 2,4-dihydroxypropiophenone (**1e**) (20) and its analogues 5-bromo-2,4-dihydroxypropiophenone (**1h**) (23), 2,4-dihydroxy-5-nitropropiophenone (**1g**) (22*a*), 5-propionyl-2,4-dihydroxypropiophenone) (**1f**) (21), and 2,4-dihydroxydeoxybenzoin (**1i**) (24) were prepared according to literature procedure.

Oxidation of 5-substituted 2,4-dihydroxyacetophenones (1a–1d), 5-substituted 2,4-dihydroxypropiophenones (1e–1h), and 2,4-dihydroxydeoxybenzoin (1i) with phenyliodonium diacetate – potassium hydroxide in methanol (condition i). General procedure

To a stirred solution of potassium hydroxide (0.03 mol) in methanol (20 mL) at 0°C in a conical flask placed in an ice bath was added 5-substituted 2,4-dihydroxyacetophenone, 5-substituted 2,4-dihydroxypropiophenone, or 2,4-dihydroxydeoxybenzoin (**1**) (0.01 mol). PIDA (0.011 mol) was slowly added to the resulting solution and the stirring was continued overnight. Water was added to the resulting reaction mixture followed by dilute HCl. Subsequently, the mixture was extracted with dichloromethane (3 × 50 mL). The combined organic phases were washed with water and dried over anhydrous sodium sulphate. The solvent was distilled off at reduced pressure. The crude product, so obtained, was purified by passing through a column using silica gel (60–120 mesh size) and ethyl acetate – petroleum ether (1:10) as eluent to afford pure product **4**.

The yields and melting points of products **4a**–**4i** are given in Table 1.

*Isolation of iodonium ylides **5c** and **5g***

To a stirred solution of potassium hydroxide (1.680 g, 0.03 mol) in methanol (20 mL) at 0°C in a conical flask placed in an ice bath was added 2,4-dihydroxy-5-nitroacetophenone (**1c**, 1.970 g, 0.01 mol) or 2,4-dihydroxy-5-nitropropiophenone (**1g**, 2.110 g, 0.01 mol). The reaction mixture was stirred for 10 min. PIDA (3.542 g, 0.011 mol) was added slowly to the resulting solution and the stirring was continued for half an hour. A yellow crystalline solid (separated out of the solution) was isolated by filtration, followed by washing with methanol. **5c**: yield, 3.272 g, 82%, mp >200°C. **5g**: yield 2.312 g, 56%, mp 196–198°C; ^{1}H NMR (DMSO) δ 1.2 (t, 3H, C_1-$COCH_2C\mathbf{H}_3$), 2.97(q, 2H, C_1-$COC\mathbf{H}_2CH_3$), 6.70–7.25(m, 5H, C_4-I-$C_6\mathbf{H}_5$), 8.6(s, 1H, C_6-**H**).

Oxidation of 5-substituted 2,4-dihydroxyacetophenones (1a–1d), 5-substituted 2,4-dihydroxypropiophenones (1e–1h), and 2,4-dihydroxydeoxybenzoin (1i) with phenyliodonium diacetate in methanol (condition ii). General procedure

PIDA (0.01 mol) was added to a solution of 5-substituted 2,4-dihydroxyacetophenone, 5-substituted 2,4-dihydroxypropiophenone, or 2,4-dihydroxydeoxybenzoin **1** (0.01 mol) in methanol (20 mL) and the reaction mixture was refluxed. The progress of the reaction was monitored by TLC. After about 12 h the solvent was removed in vacuo To the resulting mixture was added water, followed by extraction with dichloromethane (3 × 50 mL). The combined organic phases were washed with water and dried over anhydrous sodium sulphate. The solvent was distilled at reduced pressure to obtain the crude product, which was purified by passing through a column using silica gel (60–120 mesh size) and petroleum ether – ethyl acetate (10:1) as an eluent to afford pure product **4.**

The physical data of the pure products are listed in Table 1.

Oxidation of 5-substituted 2,4-dihydroxyacetophenones (1a–1d), 5-substituted 2,4-dihydroxypropiophenones (1e–1h), and 2,4-dihydroxydeoxybenzoin (1i) with phenyliodonium diacetate in acetic acid (condition iii). General procedure

PIDA (0.01 mol) was added to a solution of 5-substituted 2,4-dihydroxyacetophenone, 5-substituted 2,4-dihydroxypropiophenone, or 2,4-dihydroxydeoxybenzoin **1** (0.01 mol) in acetic acid (20 mL) and the reaction mixture was refluxed. The progress of the reaction was monitored by TLC. After about 8 h the solvent was removed in vacuo. To the resulting mixture was added water, followed by extraction with dichloromethane (3 × 50 mL). The combined organic extracts were washed with water and dried over anhydrous sodium sulphate. The solvent was distilled at reduced pressure to obtain the crude product, which was purified by passing through a column using silica gel (60–120 mesh size) and petroleum ether – ethyl acetate (10:1) as an eluent to afford pure product **4**.

The physical data of the pure products are listed in Table 1.

Spectral and analytical data of products 4a–4i

*5-Acetyl-2-hydroxy-3-iodo-4-phenoxyacetophenone (**4a**)*: IR (Nujol, cm^{-1}): 1630, 1670 (C=O). ^{1}H NMR ($CDCl_3$) δ: 2.47 (s, 3H, C_1-$COC\mathbf{H}_3$), 2.72 (s, 3H, C_5-$COC\mathbf{H}_3$), 6.80–7.42 (m, 5H, C_4-$OC_6\mathbf{H}_5$), 8.45 (s, 1H, C_6-**H**). MS (70 eV) *m*/*z*: 396 (M^+, 34), 305 (26), 303 (100), 287 (9). Anal. calcd. for $C_{16}H_{13}IO_4$: C 48.48, H 3.28; found: C 48.34, H 3.35.

*2-Hydroxy-3-iodo-4-phenoxyacetophenone (**4b**)*: IR (Nujol, cm^{-1}): 1625 (C=O). ^{1}H NMR ($CDCl_3$) δ: 2.60 (s, 3H, C_1-$COC\mathbf{H}_3$), 6.25 (d, 1H, C_5-**H**, J = 8.9 Hz), 7.00–7.50 (m, 5H, C_4-$OC_6\mathbf{H}_5$), 7.63 (d, 1H, C_6-**H**). Anal. calcd. for $C_{14}H_{11}IO_3$: C 47.45, H 3.10; found: C 47.14, H 2.80.

*2-Hydroxy-3-iodo-5-nitro-4-phenoxyacetophenone (**4c**)*: IR (Nujol, cm^{-1}): 1645 (C=O). ^{1}H NMR ($CDCl_3$) δ: 2.70 (s, 3H, C_1-$COC\mathbf{H}_3$), 6.80–7.32 (m, 5H, C_4-$OC_6\mathbf{H}_5$), 8.50 (s, 1H, C_6-**H**), 13.30 (bs, 1H, C_2-O**H**). Anal. calcd. for $C_{14}H_{10}NIO_5$: C 42.10, H 2.50; found: C 41.39, H 2.45.

*5-Bromo-2-hydroxy-3-iodo-4-phenoxyacetophenone (**4d**)*: IR (Nujol, cm^{-1}): 1630 (C=O). ^{1}H NMR ($CDCl_3$) δ: 2.20 (s, 3H, C_1-$COC\mathbf{H}_3$), 6.50–7.30 (m, 5H, C_4-$OC_6\mathbf{H}_5$), 7.86 (s, 1H, C_6-**H**), 13.40 (bs, 1H, C_2-O**H**). MS (70 eV) *m*/*z*: 434 (M^+, 8.9), 434 (M^+, 7.7), 433 (49), 431 (48), 342 (15), 340 (100), 338 (92), 291 (12). Anal. calcd. for $C_{14}H_{10}BrIO_3$: C 38.80, H 2.31; found: C 38.70, H 2.40.

*2-Hydroxy-3-iodo-4-phenoxypropiophenone (**4e**)*: IR (Nujol, cm^{-1}): 1635 (C=O). ^{1}H NMR ($CDCl_3$) δ: 0.80–0.96 (t, 3H, C_1-$COCH_2$-$C\mathbf{H}_3$), 2.50–3.00 (q, 2H, C_1-$COC\mathbf{H}_2CH_3$), 6.50–7.90 (m, 7H, C_4-$OC_6\mathbf{H}_5$, C_5-**H** and C_6-**H**). Anal. calcd. for $C_{15}H_{13}IO_3$: C 48.91, H 3.53; found: C 48.71, H 3.36.

*2-Hydroxy-3-iodo-5-propionyl-4-phenoxypropiophenone (**4f**)*: IR (Nujol, cm^{-1}): 1630 (C=O). ^{1}H NMR ($CDCl_3$) δ: 0.70–1.30 (t, 6H, C_1- and C_5-$COCH_2$-$C\mathbf{H}_3$), 2.50–3.20 (q, 4H, C_1- and C_5-$COC\mathbf{H}_2CH_3$), 6.45–7.30 (m, 5H, C_4-$OC_6\mathbf{H}_5$), 8.25 (s,

1H, C_6-**H**), 9.55 (bs, 1H, C_2-O**H**). Anal. calcd. for $C_{18}H_{17}IO_4$: C 50.94, H 4.00; found: C 50.86, H 3.86.

*2-Hydroxy-3-iodo-5-nitro-4-phenoxypropiophenone (**4g**)*: IR (Nujol, cm^{-1}): 3300 (OH), 1645 (C=O). ^{1}H NMR ($CDCl_3$) δ: 1.17 (t, 3H, C_1-$COCH_2$-C**H**$_3$), 2.95 (q, 2H, C_1-COC**H**$_2CH_3$), 6.54–7.30 (m, 5H, C_4-OC_6**H**$_5$), 8.60 (s, IH, C_6-Ar-**H**). Exact Mass (70 eV) *m*/*z* calcd. for $C_{15}H_{12}INO_5$: 413.00; found: 412.98. MS *m*/*z*: 412.98 (77.7), 383.94 (53.8), 336.94 (27.8), 319.97 (47.9), 286.7 (39.6), 215.97 (26.0), 193.04 (100.0), 152.4 (23.0), 94.04 (68.2), 77.04 (32.6).

*5-Bromo-2-hydroxy-3-iodo-4-phenoxypropiophenone (**4h**)*: IR (Nujol, cm^{-1}): 3300 (OH), 1640 (C=O). ^{1}H NMR ($CDCl_3$) δ: 0.95 (t, 3H, C_1-$COCH_2$C**H**$_3$), 2.75 (q, 2H, C_1-COC**H**$_2CH_3$), 6.60–7.40 (m, 5H, C_4-OC_6**H**$_5$), 7.98 (s, IH, C_6-Ar-**H**). Exact Mass (70 eV) *m*/*z* calcd. for $C_{15}H_{12}BrIO_3$: 448.00; found: 447.90. MS *m*/*z*: 447.90 (74.6), 445.90 (77.1), 418.86 (99.9), 416.87 (100.0), 354.86 (52.3), 352.87 (54.8), 342.83 (25.9), 340.83 (25.6), 296.85(34.6), 294.86 (70.3), 292.85 (35.0), 291.97 (25.5), 290.97 (27.4), 289.97 (24.6), 243.98 (22.6), 216.93 (73.6), 214.94 (75.6), 77.03 (34.0).

*2-Hydroxy-3-iodo-4-phenoxydeoxybenzoin (**4i**)*: IR (Nujol, cm^{-1}): 3300 (OH), 1645 (C=O). ^{1}H NMR ($CDCl_3$) δ: 4.14 (s, 2H, C_1-COC**H**$_2C_6H_5$), 6.15 (d, 1H, C_5-Ar-**H**), 6.90–7.70 (m, 11H, C_1-$COCH_2C_6$**H**$_5$, C_4-OC_6**H**$_5$, C_6-Ar-**H**). Exact Mass (70 eV) *m*/*z* calcd.: 430.00; found: 431.00. MS *m*/*z*: 431 (4.44), 430 (19.61), 341 (5.10), 340 (37.98), 339 (100.0), 302 (0.9), 263 (5.83), 234 (2.05), 211 (16.31), 184 (16.64), 128 (14.74), 102 (3.33), 101 (1.19).

Acknowledgment

We are grateful to the Council of Scientific and Industrial Research (New Delhi) for financial assistance and to the Mass Spectrometry Facility, Department of Pharmaceutical Chemistry, San Francisco, California, for the scanning high-resolution mass spectra.

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