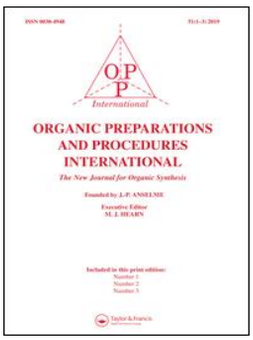

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An Effective and Environmentally Friendly Synthesis of 1,3-Keto-alcohols

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The 1,3-hydroxycarbonyl group is an essential synthon for natural products and bioactive organic molecules. In order to obtain this structure, a number of reaction methods have been developed and, among them, the aldol reaction is one of the most popular and effective.[1–6] Green chemistry methods have contributed greatly to the progress of aldol reactions in recent years. Harmful effects are reduced by solvent-free catalytic aldol reactions[7,8] or catalytic aldol reactions in water,[9] supercritical fluids[10–12] or ionic liquids.[13,14] The use of catalysts can be made more effective by using heterogeneous catalysis,[15] synthetic chiral catalysts,[16] biocatalysts[17,18] and biomimetic catalysts.[19,20]

Dimethyl sulfoxide (DMSO), in the presence of a little water, can be used as a polar aprotic solvent in organocatalyzed asymmetric aldol reactions.[21–27] According to the U.S. Environmental Protection Agency (EPA), DMSO has also been classified as a nontoxic solvent that poses no human health hazard.[28]

In this study, our aim was to synthesize new tetralone- and indanone-derived 1,3-keto-alcohols, which can be used as important prochiral intermediates. In the past, these 1,3-keto-alcohols were only synthesized by the Mukaiyama aldol reaction using silyl enol ethers instead of ketones.[29–33] In contrast, because of the reversibility and the difficulty of controlling the enolate geometry, acid- or base-promoted classical aldol reactions give a complex mixture of products. Thus, when the classical aldol reaction is used to obtain tetralone or indanone-derived 1,3-keto-alcohols, α-β-unsaturated ketone derivatives generally occur.

When we tried to prepare the ketones with the classic aldol procedure[34] and reagents we always saw the formation of alkene derivatives. Because of high conjugation, tetralone-derived α-β-unsaturated ketones are very stable, so in the typical classic strongly acidic or basic environment, the keto-alcohol immediately turns into an alkene by dehydration (Scheme 1).

We thus found a new method by testing different conditions (Table 1).

As seen in Table 1, when NaOH was used as the base, there was no keto-alcohol product **3a** and only the α,β-unsaturated ketone occurred at 25 °C (Entry 1). On the other hand, when K_2CO_3 was used as the base at room temperature, we observed **3a** as

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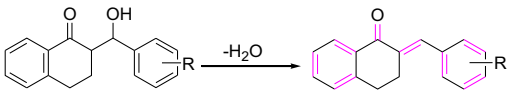
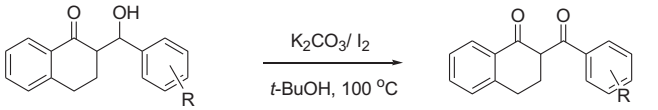


Scheme 1. Dehydration of the 1,3-keto-alcohol and highly conjugated product.

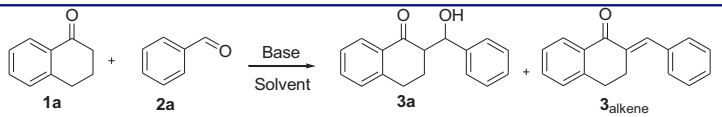


R=3-Me (**3b**), 4-NO_2 (**3d**)

Yield: 85% **6b** (with **3b**)
91% **6d** (with **3d**)

Scheme 2. The oxidation reaction of keto alcohols.

Table 1. Aldol reaction conditions.[a]



1a **2a** **3a** 3_{alkene}

Entry	Base	Solvent	T (°C)	Time (h)	**Yield** (%)[b] **(3a)**	*dr(anti/syn)*[c] (3a)	**Yield** (%)[b] **(3_{alkene})**
1	NaOH	MeOH	25	5	0	–	98
2	NaOH	MeOH	0	5	25	52:48	71
3	NaOH	MeOH	−10	2	55	62:38	34
4	NaOH	DMSO	−10	2	50	67:33	25
5	KOH	MeOH	−10	2	10	63:37	75
6	Na_2CO_3	MeOH	25	48	52	65:35	0
7	K_2CO_3	MeOH	25	48	75	76:24	0
8	K_2CO_3	DMSO	25	48	80	70:30	0
9	K_2CO_3	H_2O	25	48	10	58:42	0
10	K_2CO_3	DMSO-H_2O (8:2)	25	24	95	85:15	0

[a]Conditions: **1a** (10 mmol), **2a** (7.5 mmol), and base (10 eq %) in solvent (2.5 mL) were stirred at the corresponding temperature.
[b]Yield of isolated product. [c] The *dr* values were determined by HPLC analysis using a chiral column.

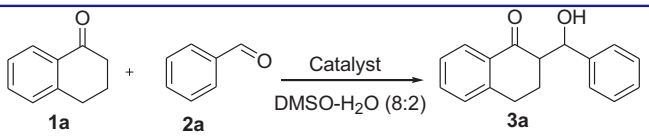
the only product with good yield. The solvent mixture DMSO-H_2O (8:2) showed the best result with 95% yield in 24 hours (Entry 10). Also very good diastereoselectivity was achieved with diasteromeric ratio (*dr*) 85:15 using K_2CO_3 and DMSO-H_2O (8:2) in the aldol reaction.

We tried some Lewis acids ($FeCl_3$, $CoCl_2$, $ZnCl_2$ and $MnCl_2$) and organic Brønsted acids (benzoic acid, trifluoroacetic acid (TFA) and *N*-triflylphosphoramide (NTPA)) as catalysts in this reaction and compared these acids with K_2CO_3. The results are summarized in Table 2 and as seen clearly the other catalysts are not better than K_2CO_3.

With the best conditions in hand, we prepared 1,3-keto-alcohols using tetralone and different aldehydes. The new 1,3-keto-alcohols and their yields are given in Table 3. A number of these compounds are novel, but several have been synthesized before using the Mukaiyama procedure, including **3a**,[31–33] **3c**,[35] **3d**,[36] and **3e.**[29]

Table 3 shows that the yields change according to the substituents and their positions. Yields increased with electron-withdrawing groups such as -NO_2 (Entry 4). With strong electron-donating groups such as –MeO the yields were reduced (Entries 5 and 6). When the aldehydes were substituted with weak electron-donating groups such as Me, the yields were good (Entries 2 and 3). While good yields were obtained with phenyl

Table 2. The effect of different Lewis acid or Brønsted acid in Aldol reaction with tetralone and benzaldehyde.[a,b]

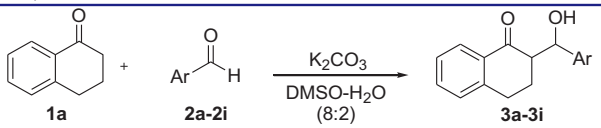


1a **2a** **3a**

Entry	Catalyst	Time (h)	**Yield** (%)[c] **(3a)**	***dr(anti/syn)***[d] **(3a)**
1	$FeCl_3$	48	35	59:41
2	$CoCl_2$	48	0	–
3	$ZnCl_2$	48	0	–
4	$MnCl_2$	48	0	–
5	Benzoic acid	48	0	–
6	TFA	48	0	–
7	NTPA	48	5	75:25

[a]Conditions: **1a** (10 mmol), **2a** (7.5 mmol), and acid (10 eq %) in solvent (2.5 mL) were stirred at 25 °C.
[b]3_{alkene} was not observed in these experiments.
[c]Yield of isolated product.
[d]The *dr* values were determined by HPLC analysis using a chiral column.

Table 3. Scope of aldehydes for the aldol reaction method with tetralone.[a]



1a **2a-2i** **3a-3i**

Entry	Ar	Aldehyde	Product	**Yield** (%)[b]	***dr(anti/syn)***[c]
1	C_6H_5	**2a**	**3a**	95	85:15
2	3-MeC_6H_4	**2b**	**3b**	96	81:19
3	2-MeC_6H_4	**2c**	**3c**	70	73:27
4	4-$NO_2C_6H_4$	**2d**	**3d**	95	76:24
5	4-$MeOC_6H_4$	**2e**	**3e**	60	62:38
6	2-$MeOC_6H_4$	**2f**	**3f**	66	81:19
7	2-Naphthyl	**2g**	**3g**	80	55:45
8	2-Furyl	**2h**	**3h**	55	80:10
9	2-Thienyl	**2i**	**3i**	68	65:35
10	2-FC_6H_4	**2j**	**3j**	94	81:19
11	2-BrC_6H_4	**2k**	**3k**	91	79:21

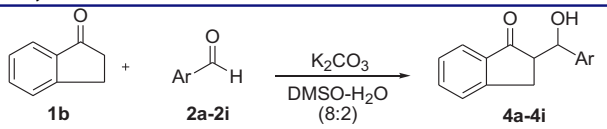
[a]Conditions: **1a** (10 mmol), **2a-2i** (7.5 mmol), and K_2CO_3 (10 eq %) in DMSO-H_2O (8:2) (2.5 mL) were stirred at room temperature.
[b]Yield of isolated product.
[c]*dr* values were determined by HPLC analysis using a chiral column.

and naphthyl as aryl groups (Entries 1 and 7), moderate yields were obtained with the substrates containing heteroaryl groups such as furyl and thienyl (Entries 8 and 9); that is, alkene derivatives were also formed in these reactions.

We have also used indanone for our aldol reaction method. The results are shown in Table 4. The yields are between 45-86%. The best yield was obtained with benzaldehyde and 4-nitrobenzaldehyde with 87 and 86% yield respectively in the reaction of indanone. With the exceptions of **4a**,[33] **4d**[36] and **4e**,[30] the other indanone derivatives are novel.

We also tried the aldol reaction method with 2-heptanone as an aliphatic ketone (Tables 5 and 6). Compound **5a**[37] was previously known, and **5d** was synthesized for the first time in this study.

Table 4. Scope of aldehydes for the aldol reaction method with indanone.[a]

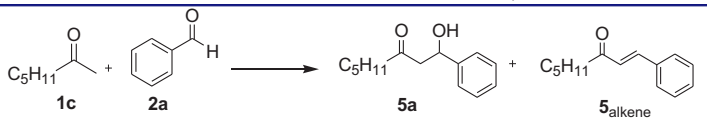


1b **2a-2i** **4a-4i**

Entry	Ar	Aldehyde	Product	**Yield** (%)[b]	***dr(anti/syn)***[c]
1	C_6H_5	**2a**	**4a**	87	81:19
2	3-MeC_6H_4	**2b**	**4b**	75	82:18
3	2-MeC_6H_4	**2c**	**4c**	69	75:25
4	4-$NO_2C_6H_4$	**2d**	**4d**	86	78:22
5	4-$MeOC_6H_4$	**2e**	**4e**	60	65:35
6	2-$MeOC_6H_4$	**2f**	**4f**	45	61:39
7	2-Naphthyl	**2g**	**4g**	83	83:17
8	2-Furyl	**2h**	**4h**	45	77:13
9	2-Thienyl	**2i**	**4i**	62	69:31
10	2-FC_6H_4	**2j**	**4j**	86	78:22
11	2-BrC_6H_4	**2k**	**4k**	75	76:24

[a]Conditions: **1b** (10 mmol), **2a-2i** (7.5 mmol), and K_2CO_3 (10 eq %) in DMSO-H_2O (8:2) (2.5 mL) were stirred at room temperature.
[b]Yield of isolated product.
[c]*dr* values were determined by HPLC analysis using a chiral column.

Table 5. Aldol reaction conditions for 2-heptanone and benzaldehyde.[a]

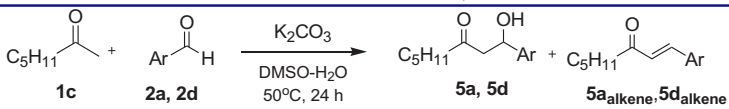


1c **2a** **5a** $\mathbf{5}_{alkene}$

Entry	Base	Solvent	T(°C)	Time (h)	Yield (%)[b] (5a)	Yield (%)[b] (5_{alkene})
1	K_2CO_3 (5 eq %)	DMSO-H_2O (8:2)	25	48	5	25
2	K_2CO_3 (5 eq %)	DMSO-H_2O (8:2)	50	4	25	45
3	K_2CO_3 (10 eq %)	DMSO-H_2O (8:2)	50	24	45	50
4	Na_2CO_3 (10 eq %)	DMSO-H_2O (8:2)	50	24	25	66
5	aq. NaOH (40%, 5 mL)	MeOH	25	24	0	84
6	aq. NaOH (40%, 5 mL)	MeOH	−10	5	0	20
7	aq.NaOH (40%, 0.1 mL)	MeOH	−18	2	15	75

[a]Conditions: **1c** (10 mmol), **2a** (7.5 mmol), and base in solvent (2.5 mL) were stirred at the corresponding temperature.
[b]Yield of isolated product.

Table 6. Aldol reaction of 2-heptanone with aromatic aldehydes.[a]



1c **2a, 2d** **5a, 5d** $\mathbf{5a}_{alkene}$,$\mathbf{5d}_{alkene}$

Entry	Ar	Aldehyde	Product	**Yield** (%)[b] **Alcohol product**	**Yield** (%)[b] **Alkene product**
1	C_6H_5	**2a**	**5a**	45	50
2	4-$NO_2C_6H_4$	**2d**	**5d**	75	23

[a]Conditions: **1c** (10 mmol), **2a, 2d** (7.5 mmol), and K_2CO_3 (10% equiv.) in DMSO-H_2O (8:2) (2.5 mL) were stirred at 50 °C.
[b]Yield of isolated product.

We further showed our 1,3-keto-alcohols can be oxidized to the related 1,3-diketones.[38,39] Compounds **3b** and **3d** were converted to the corresponding diketones **6b** and **6d** with K_2CO_3/I_2 (Scheme 2).[40] Compound **6b**[41] was obtained previously by Claisen condensation of tetralone and the appropriate aryl methyl ester. The diketone **6d** is new.

In summary, we have prepared some new 1,3-keto-alcohols, using a method that is consistent with green principles. These materials can be useful as prochiral intermediates. We hope that their preparation may stimulate further research in the synthesis of chiral compounds or chiral catalysts.

Experimental section

Chemicals were commercially available from Merck, Acros or Aldrich. All novel pruducts were purified by crystallization or column chromatography and were characterized by IR, ^{1}H-NMR, ^{13}C-NMR, elemental analysis and GC-MS. The reactions were monitored by TLC using silica gel plates and the products were purified by flash column chromatography on silica gel (Merck; 230–400 mesh), eluted with hexane-ethyl acetate (v/v 9:1). NMR spectra were recorded at 500 MHz for ^{1}H and 125 MHz for ^{13}C using Me_4Si as the internal standard in $CDCl_3$. GC-MS spectra were recorded on Shimadzu/QP2010 Plus. IR spectra were recorded on a Bruker Vertex 70 IR spectrometer. Melting points were determined with Büchi Melting Point B-540 and are uncorrected. For determination of *dr* values, HPLC was performed on a Shimadzu/DGU-20A_5 HPLC apparatus fitted with a 25 cm Chiralcel OD, Chiralcel OD-H, Chiralcel OJ-H and Chiralpac AD-H chiral columns.

General experimental procedure for aldol reaction

Aromatic aldehyde (7.5 mmol), ketone (10 mmol), K_2CO_3 (10% equiv.) and 2.5 mL of DMSO:H_2O (8:2) were added to a flask then the mixture was stirred at room temperature for 1-2 days, as specified by TLC. The reaction was extracted with dichloromethane (3 X 10 mL). Then the combined extracts were washed with saturated brine and dried over anhydrous Na_2SO_4; after filtration the solvents were removed under reduced pressure. Reactions were monitored by thin layer chromatography and visualized by using UV light. For determination of the isolated yield, flash chromatography was performed on silica gel (Merck; 230–400 mesh) with hexane-ethyl acetate (v/v 9:1) as the mobile phase.

General procedure for oxidation of keto alcohols

Diketones were synthesized by oxidation of their corresponding 1,3- keto alcohols using I_2/K_2CO_3 in t-BuOH, according to this procedure: keto-alcohol (0.1 mmol), iodine (0.2 mmol), K_2CO_3 (0.2 mmol) and 4 mL of *t*-BuOH are added to a flask and the mixture refluxed at 100° C for 16 hours. When the reaction was finished (TLC, hexane-ethyl acetate (v/v 9:1)), an excess of saturated sodium bisulfite solution was added. Then the mixture was stirred at room temperature until the color of iodine was gone. Extraction was then carried out with dichloromethane. The products were purified by column chromatography on silica gel with hexane-ethyl acetate (v/v 9:1).

2-(Hydroxy-phenyl-methyl)-3,4-dihydro-2H-naphthalen-1-one (3a)

Colorless oil. IR (neat, cm^{-1}): 3577, 3459, 3024, 2903, 1664, 1655, 1595, 1451, 1223, 1048, 745, 705 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.90-1.95 (m, 1H), 2.08-2.17 (m, 1H), 2.81–2.85 (m, 1H), 2.89-2.91 (m, 1H), 2.92–2.95 (m, 1H), 4.98 (s, 1H), 5.72 (s, 1H), 7.22–7.24 (d, 1H, $J = 8.1$ Hz), 7.33-7.42 (m, 6H), 7.47–7.54 (m, 1H), 8.08-8.11 (m, 1H). ^{13}C-NMR ($CDCl_3$): δ 22.2, 28.9, 54.7, 71.6, 125.9–133.7 (9C), 141.1–144.4 (3C), 200.0. MS *m/z*: 253 (M + 1), 233, 202, 115, 91.

Anal. Calcd. for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39. Found: C, 80.75; H, 6.51.

2-(Hydroxy-m-tolyl-methyl)-3,4-dihydro-2H-naphthalen-1-one (3b)

Pale orange solid, mp 89.0-90.0 °C. IR (neat, cm^{-1}): 3479, 2963, 2888, 1664, 1598, 1460, 1445, 1367, 1313, 1290, 1232, 1154, 1059, 1013, 803, 760, 728 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.80–2.06 (m, 2H), 2.27 (s, 3H), 2.66–2.85 (m, 3H), 4.82–4.84 (d, 1H, $J = 8.5$ Hz), 5.57 (d, 1H $J = 2.5$ Hz), 6.98-7.41 (m, 7H), 7.96-7.99 (dd, 1H, $J_1 = 8.0$, $J_2 = 1.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 20.5, 21.2, 27.9, 53.7, 70.5, 123.9–143.3 (12C), 198.9. MS *m/z*: 265 (M-1), 233, 247, 202, 128.

Anal. Calcd. for $C_{18}H_{18}O_2$: C, 81.17; H, 6.81. Found: C, 80.88; H, 6.96.

2-(Hydroxy-o-tolyl-methyl)-3,4-dihydro-2H-naphthalen-1-one (3c)

Pale orange solid, mp 113-114 °C. IR (neat, cm^{-1}): 3525, 3064, 2957, 2920, 2859, 1676, 1598, 1454, 1364, 1290, 1223, 1085, 762, 742 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.82-1.87 (m, 1H), 2.10–2.19 (dd, 1H, $J_1 = 5.0$ Hz, $J_2 = 10.0$ Hz), 2.23 (s, 3H), 2.44 (d, 1H, $J = 3.9$ Hz), 2.59–2.63 (ddd, 1H, $J_1 = 13.3$, $J_2 = 2.0$, $J_3 = 6.7$ Hz), 2.75-2.90 (m, 2H), 5.92 (d, 1H, $J = 6.0$ Hz), 7.07–7.25 (m, 5H), 7.38-7.44 (m, 1H), 7.50 (d, 1H, $J = 7.6$ Hz), 8.00 (m, 1H). ^{13}C-NMR ($CDCl_3$): δ 17.9, 20.7, 27.9, 51.3, 66.8, 124.7–138.9 (11C), 143.4, 198.7. MS *m/z*: 265 (M-1), 278, 262, 232, 202, 128, 115, 90.

Anal. Calcd. for $C_{18}H_{18}O_2$: C, 81.17; H, 6.81. Found: C, 80.95; H, 7.05.

2-[Hydroxy-(4-nitro-phenyl)-methyl]-3,4-dihydro-2H-naphthalen-1-one (3d)

Pale brown solid, mp 140.9–141.8 °C. IR (neat, cm^{-1}): 3580, 3447, 3113, 3081, 2931, 1670, 1595, 1511, 1344, 1223, 1065, 1013, 858, 748, 705 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.68–1.73 (m, 1H), 1.99-2.08 (m, 1H), 2.76-3.01 (m, 3H), 5,04 (d, 1H, $J = 8.0$ Hz), 5.72 (s, 1H), 7.16–7.31 (m, 2H), 7.40–7.53 (m, 3H), 7.97 (d, 1H, $J = 7.5$ Hz), 8.14–8.16 (m, 2H). ^{13}C-NMR ($CDCl_3$): δ 22.1, 28.8, 54.5, 70.9, 123.5–134.3 (10C), 144.2, 149.5, 199.3. MS *m/z*: 279 (M-H_2O), 278, 262, 248, 232, 202, 115, 90.

Anal. Calcd. for $C_{17}H_{15}NO_4$: C, 68.68; H, 5.09; N, 4.71. Found: C, 68.83; H, 5.25, N, 4.52.

2-[Hydroxy-(4-methoxy-phenyl)-methyl]-3,4-dihydro-2H-naphthalen-1-one (3e)

Pale yellow solid, Mp 124.0–125.0 °C. IR (neat, cm^{-1}): 3441, 2966, 2911, 2842, 1678, 1598, 1511, 1457, 1304, 1246, 1220, 1171, 1085, 1030, 797, 768, 742, 662 cm^{-1}. ^{1}H-NMR ($CDCl_3$):

δ 1.93-1.98 (m, 1H), 2.05–2.14 (m, 1H), 2.79-2.84 (m, 1H), 2.91–2.96 (m, 2H), 3.83 (s, 3H), 5.62 (s, 1H), 6.91-6.94 (m, 2H), 7.23–7.24 (d, 1H, $J = 7.6$ Hz), 7.28–7.36 (m, 4H), 7.43–7.52 (m, 1H), 8.07–8.09 (m, 1H). ^{13}C-NMR ($CDCl_3$): δ 22.5, 29.0, 54.6, 55.2, 71.5, 113.6-134.0 (10C), 144.4, 158.71, 200.26. MS *m/z*: 282 (M), 281, 263, 249, 233, 121, 90.

Anal. Calcd. for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.73; H, 6.24.

2-[Hydroxy-(2-methoxy-phenyl)-methyl]-3,4-dihydro-2H-naphthalen-1-one (3f)

Beige solid, mp 162–163 °C. IR (neat, cm^{-1}): 3482, 3070, 3032, 2978, 2937, 2865, 2839, 1676, 1598, 1488, 1454, 1439, 1362, 1290, 1223, 1051, 1025, 1002, 970, 742, 725, 656 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.73–2.09 (m, 2H), 2.75–2.87 (m, 3H), 2.88–2.92 (dd, 1H, $J_1 = 15.0$ Hz, $J_2 = 10.0$ Hz), 3.70 (s, 3H), 5.90 (s, 1H), 6.77–7.45 (m, 7H), 8.1 (d, 1H, $J = 10.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 21.2, 27.9, 51.1, 54.1, 65.6, 108.9, 119.3, 125.4–132.3 (8C), 143.4, 154.4, 199.3. MS *m/z*: 282 (M), 233, 202, 115, 90.

Anal. Calcd. for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.82; H, 6.36.

2-(Hydroxy-naphthalen-2-yl-methyl)-3,4-dihydro-2H-naphthalen-1-one (3g)

Yellow solid, mp 146–148 °C. IR (neat, cm^{-1}): 3490, 2960, 2920, 1661, 1601, 1454, 1359, 1264, 1226, 1091, 1016, 869, 803, 751, 734, 676, 656 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.52–2.07 (m, 2H), 2.71-2.79 (m, 3H), 4.94 (s, 1H), 5.04 (d, 1H, $J = 8.5$ Hz), 7.07 (d, 1H, $J = 7.5$ Hz), 7.18–7.24 (m, 1H), 7.32–7.39 (m, 4H), 7.71–7.78 (m, 4H), 7.99 (d, 1H, $J = 8.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 25.2, 27.9, 52.8, 74.6, 123.0–133.0 (16C), 201.3. MS *m/z*: 284 (M-H_2O), 267, 252, 239, 141, 128, 90.

Anal. Calcd. for $C_{21}H_{18}O_2$: C, 83.42; H, 6.00. Found: C, 83.63; H, 6.25.

2-(Furan-2-yl-hydroxy-methyl)-3,4-dihydro-2H-naphthalen-1-one (3h)

Brown oil. IR (neat, cm^{-1}): 3430, 3122, 2937, 2900, 2877, 1670, 1601, 1511, 1454, 1362, 1324, 1295, 1226, 1148, 1123, 1099, 1062, 1007, 955, 918, 809, 774, 739, 665 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.64–1.70 (m, 1H), 1.95-2.01 (m, 1H), 2.85–2.99 (m, 3H), 3.54 (br s, 1H), 5.33 (d, 1H, $J = 2.5$ Hz), 6.22–6.27 (m, 2H), 7.13–7.26 (m, 3H), 7.33–7.43 (m, 1H), 7.96 (d, 1H, $J = 8.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 23.9, 28.9, 51.9, 67.8, 106.9, 110.3, 126.6–134.1 (5C), 141.6, 144.3, 155.08, 199.7. MS *m/z*: 224 (M-H_2O), 206, 195, 170, 165, 152, 128, 118, 90, 81, 63, 51.

Anal. Calcd. for $C_{15}H_{14}O_3$: C, 74.36; H, 5.82. Found: C, 74.56; H, 6.14.

2-(Hydroxy-thiophen-2-yl-methyl)-3,4-dihydro-2H-naphthalen-1-one (3i)

Green oil. IR (neat, cm^{-1}): 3433, 3067, 2929, 2871, 1667, 1601, 1454, 1356, 1226, 1157, 1030, 912, 826, 702 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.61–1.79 (m, 1H), 1.95–2.03 (m, 1H), 2.68–2.91 (m, 3H), 4.93 (s, 1H), 5.2 (s, 1H), 6.88–6.93 (m, 2H), 7.13–7.26 (m, 3H), 7.38–7.43 (m, 1H), 7.98 (d, 1H, $J = 8.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 25.0, 27.7, 53.4, 70.4, 123.3–133.0 (9C), 143.4, 200.5. MS *m/z*: 256 (M-2), 207, 193, 147, 118, 103, 90, 82, 43, 41.

Anal. Calcd. for $C_{15}H_{14}O_2S$: C, 69.74; H, 5.46; S, 12.41. Found: C, 69.88; H, 5.57; S, 12.23.

2-[(2-Fluoro-phenyl)-hydroxy-methyl]-3,4-dihydro-2H-naphthalen-1-one (3j)

Beige solid, mp 98.4-99.8 °C. IR (neat, cm^{-1}): 3442, 3057, 2949, 2914, 1672, 1598, 1481, 1369, 1224, 1174, 1055, 871, 794, 767, 659 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.88–1.91 (m, 1H), 2.04–2.18 (m, 1H), 2.92–3.00 (m, 4H), 5.98 (d, 1H, $J = 4.0$ Hz), 7.00–7.07 (m, 1H), 7.16–7.35 (m, 4H), 7.48 (ddd, 1H, $J_1 = J_2 = 12.0$ Hz, $J_3 = 2.0$ Hz), 7.62 (ddd, 1H, $J_1 = J_2 = 13.0$ Hz, $J_3 = 3.5$ Hz), 8.07 (dd, 1H, $J_1 = 13.0$ Hz, $J_2 = 2.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 22.4, 28.9, 52.9, 66.0, 114.8, 115.1, 124.0-133.6 (7C), 144.3, 157.4, 160.7, 199.8. MS *m/z*: 269 (M-1), 234, 208, 193, 147, 118, 103, 90, 82, 43, 41.

Anal. Calcd. for $C_{17}H_{15}FO_2$: C, 75.54; H, 5.59. Found: C, 75.79; H, 5.52.

2-[(2-Bromo-phenyl)-hydroxy-methyl]-3,4-dihydro-2H-naphthalen-1-one (3k)

White solid, mp 138.2–141.9 °C. IR (neat, cm^{-1}): 3446, 3057, 2951, 2914, 1674, 1598, 1454, 1363, 1222, 1128, 1089, 923, 748, 732, 655 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.75–1.83 (m, 1H), 2.11–2.25 (m, 1H), 2.70 (br s, 1H), 2.90-2.95 (m, 2H), 3.05-3.11 (ddd, 1H, $J_1 = 23.0$ Hz, $J_2 = 7.0$ Hz, $J_3 = 4.0$ Hz), 6.06 (d, 1H, $J = 3.5$ Hz), 7.15–7.51 (m, 5H), 7.55 (dd, 1H, $J_1 = 13.5$ Hz, $J_2 = 2.0$ Hz), 7.65 (dd, 1H, $J_1 = 13.0$ Hz, $J_2 = 3.0$ Hz), 8.10 (dd, 1H, $J_1 = 13.0$ Hz, $J_2 = 1.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 25.9, 28.9, 51.6, 70.2, 121.1, 126.6–136.9 (9C), 140.7, 144.3, 199.5. MS *m/z*: 329 (M-1), 236, 207, 193, 147, 118, 103, 90, 82, 43, 41.

Anal. Calcd. for $C_{17}H_{15}BrO_2$: C, 61.65; H, 4.56. Found: C, 61.72; H, 4.67.

2-(Hydroxy-phenyl-methyl)-indan-1-one (4a)

Beige solid, mp 156–157 °C. IR (neat, cm^{-1}): 3064, 3029, 2906, 1699, 1604, 1462, 1275, 1203, 1094, 999, 843, 751, 711 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.63 (br s, 1H), 2.93 (dd, 1H, $J_1 = 4.0$, $J_2 = 17.0$ Hz), 3.38 (dd, 1H, $J_1 = 8.0$, $J_2 = 17.0$ Hz), 3.68 (ddd, 1H, $J_1 = 4.0$, $J_2 = J_3 = 8.0$ Hz), 5.32 (d, 1H, $J = 8.0$ Hz), 7.19–7.39 (m, 7H), 7.50–7.56 (m, 1H), 7.71 (d, 1H, $J = 7.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 31.0, 48.8, 70.1, 123.8–141.3 (11C), 153.4, 207.4. MS *m/z*: 220 (M-H_2O), 191, 165, 132, 155, 104, 91, 77.

Anal. Calcd. for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.45; H, 6.16.

2-(Hydroxy-m-tolyl-methyl)-indan-1-one (4b)

White solid, mp 117–118 ^{0}C. IR (neat, cm^{-1}): 2920, 2850, 1696, 1626, 1603, 1473, 1301, 1268, 1238, 1091, 964, 930, 892, 777, 737, 690, 673 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.25 (s, 1H), 4.36 (s, 5H), 4.86–4.95 (m, 1H), 5.24-5.28 (m, 1H), 9.07–9.79 (m, 8H). ^{13}C-NMR ($CDCl_3$): δ 20.5, 25.6, 53.8, 70.8, 121.5–141.7 (11C), 153.8, 206.3. MS *m/z*: 253 (M + 1), 233, 219, 191, 165, 115, 89.

Anal. Calcd. for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39. Found: C, 81.12; H, 6.47.

2-(Hydroxy-o-tolyl-methyl)-indan-1-one (4c)

Beige solid, mp 205.3–206.4 ^{0}C. IR (neat, cm^{-1}): 3523, 2920, 2851, 1699, 1604, 1468, 1261, 1099, 1016, 803, 748, 667 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.27 (s, 3H), 2.57 (d, 1H, $J = 5.5$ Hz), 2.78–2.87 (m, 1H), 2.91–2.98 (m, 1H), 3.18 (dd, 1H, $J_1 = 17.0$ Hz, $J_2 = 4.5$ Hz), 5.46 (br s, 1H), 7.01 (d, 1H, $J = 7.0$ Hz), 7.11–7.34 (m, 5H), 7.45–7.52 (m, 1H), 7.65 (d, 1H, $J = 8.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 25.6, 28.9, 53.8, 70.8, 121.5–141.7 (11C), 153.8, 206.3. MS *m/z*: 252 (M), 244, 132, 103, 77.

Anal. Calcd. for $C_{17}H_{16}O_2$: C, 80.93; H, 6.39. Found: C, 81.18; H, 6.43.

2-[Hydroxy-(4-nitro-phenyl)-methyl]-indan-1-one (4d)

Orange solid, mp 175–176 °C. IR (neat, cm^{-1}): 3375, 3110, 3078, 2880, 1693, 1601, 1509, 1341, 1290, 1206, 1088, 797, 754, 705, 673 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.61 (d, 1H, $J = 4.5$ Hz), 2.78 (dd, 1H, $J_1 = 8.0$ Hz, $J_2 = 17.0$ Hz), 2.98–3.01 (m, 1H), 3.09 (dd, 1H, $J_1 = 4.5$ Hz, $J_2 = 17$ Hz), 4.02 (d, 1H, $J = 1.5$ Hz), 5.63 (t, 1H, $J = 3.5$ Hz), 7.28–7.41 (m, 2H), 7.68 (d, 1H, $J = 8.5$ Hz), 7.73 (d, 1H, $J = 8.5$ Hz), 8.16 (d, 2H, $J = 8.5$ Hz), 8.23 (d, 1H, $J = 8.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 31.3, 53.5, 54.2, 70.1, 122.6–146.3 (6C), 146.7, 148.3, 148.9, 153.3, 192.6, 205.3. MS *m/z*: 282 (M-1), 281, 264, 248, 218, 189, 178, 165, 115, 89, 82, 63, 51.

Anal. Calcd. for $C_{16}H_{13}NO_4$: C, 67.84; H, 4.63; N, 4.94. Found: C, 67.64; H, 4.75, N, 4.71.

2-[Hydroxy-(4-methoxy-phenyl)-methyl]-indan-1-one (4e)

Yellow oil. IR (neat, cm^{-1}): 3433, 2917, 2839, 1690, 1604, 1514, 1468, 1246, 1171, 1030, 832, 751 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.63 (br s, 1H), 2.96 (dd, 1H, $J_1 = 17.0$, Hz, $J_2 = 17.0$ Hz), 3.04–3.09 (m, 1H), 3.84 (s, 3H), 4.76 (d, 1H, $J = 10.0$ Hz), 5.55 (s, 1H), 6.93 (dd, 2H, $J_1 = 3.0$, Hz, $J_2 = 8.5$ Hz), 7.36–7.46 (m, 4H), 7.57–7.63 (m, 1H), 7.80 (dd, 1H, $J_1 = 11.0$, Hz, $J_2 = 11.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 30.0, 54.7, 55.3, 71.9, 113.8, 113.9, 123.8–135.4 (8C), 154.7, 158.9, 209.8. MS *m/z*: 267 (M-1), 250, 235, 219, 207, 178, 152, 89, 76, 63, 51.

Anal. Calcd. for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found: C, 76.27; H, 6.18.

2-[Hydroxy-(2-methoxy-phenyl)-methyl]-indan-1-one (4f)

Beige solid, mp 150.4–151.7 °C. IR (neat, cm^{-1}): 3548, 2929, 2911, 2839, 1704, 1687, 1598, 1460, 1241, 1148, 1085, 1022, 964, 751 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.50 (br s, 1H), 2.74–2.84 (m, 1H), 3.15–3.20 (m, 2H), 3.79 (s, 3H), 5.75 (s, 1H), 6.82 (d, 1H, $J = 8.5$ Hz), 6.95 (dd, 1H, $J_1 = J_2 =$ 7.5 Hz), 7.19-7.36 (m, 3H), 7.43-7.50 (m, 2H), 7.71 (d, 1H, $J = 8.0$). ^{13}C-NMR ($CDCl_3$): δ 26.1, 51.2, 54.2, 66.9, 109.1, 119.5–136.3 (9C), 153.8, 154.8, 206.5. MS *m/z*: 267 (M-1), 282, 253, 249, 207, 193, 147, 119, 103, 96, 73, 59, 45.

Anal. Calcd. for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found: C, 76.34; H, 6.22.

2-(Hydroxy-naphthalen-2-yl-methyl)-indan-1-one (4g)

Orange solid, mp 40.0–40.5 °C. IR (neat, cm^{-1}): 3587, 3052, 2923, 1701, 1606, 1468, 1278, 1059, 909, 817, 754, 662 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.57 (s, 1H), 2.70–2.79 (m, 2H), 5.03 (d, 1H, $J = 8.8$ Hz), 5.76 (s, 1H), 7.07 (d, 1H, $J = 7.5$ Hz), 7.18–7.24 (m, 1H), 7.32–7.39 (m, 4H), 7.71–7.78 (m, 4H), 7.99 (dd, 1H, $J_1 = J_2 =$ 7.5 Hz). ^{13}C-NMR ($CDCl_3$): δ 27.7, 52.8, 74.6, 123.0-138.4 (15C), 143.3, 201.3. MS *m/z*: 270 (M-H_2O), 252, 239, 226, 215, 207, 195, 177, 120, 82, 65, 50, 40.

Anal. Calcd. for $C_{20}H_{16}O_2$: C, 83.31; H, 5.59. Found: C, 83.26; H, 5.61.

2-(Furan-2-yl-hydroxy-methyl)-indan-1-one (4h)

Brown oil. IR (neat, cm^{-1}): 3625, 2955, 2920, 2851, 1704, 1627, 1606, 1471, 1385, 1258, 1108, 1022, 737 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.88–2.98 (m, 3H), 3.54 (br s, 1H), 5.33 (d, 1H, $J = 2.5$ Hz), 6.22 (d, 1H, $J = 3.5$ Hz), 6.24-6.27 (m, 1H), 7.15 (d, 1H, $J = 7.5$ Hz), 7.21–7.26 (m, 2H), 7.38–7.42 (m, 1H), 7.97 (dd, 1H, $J_1 = 8.0$ Hz, $J_2 = 1.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 29.9, 51.9, 67.8, 106.9, 110.2, 126.6–144.3 (7C), 155. 0, 199.7. MS *m/z*: 210 (M-H_2O), 284, 253, 210, 192, 148, 133, 119, 105, 92, 78, 64, 42.

Anal. Calcd. for $C_{14}H_{12}O_3$: C, 73.67; H, 5.30. Found: C, 73.86; H, 5.14.

2-(Hydroxy-thiophen-2-yl-methyl)-indan-1-one (4i)

Green oil, IR (neat, cm^{-1}): 3389, 3070, 2920, 2851, 1687, 1604, 1471, 1428, 1295, 1203, 1151, 1097, 1019, 958, 938, 754, 713, 656 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.67 (br s, 1H), 3.10–3.19 (m, 3H), 3.36 (dd, 1H, $J_1 = 12.5$ Hz, $J_2 =$ 9.0 Hz), 5.79 (d, 1H, $J = 4.5$ Hz), 6.98-7.06 (m, 2H), 7.27 (dd, 1H, $J_1 = 1.0$ Hz, $J_2 =$ 5.5 Hz), 7.36–7.64 (m, 2H), 7.78 (d, 1H, $J = 7.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 27.4, 54.4, 69.2, 123.8–127.4 (6C), 135.1, 136.9, 146.4, 154.7, 206.8. MS *m/z*: 226 (M-H_2O), 197, 165, 152, 89, 76, 63, 51.

Anal. Calcd. for $C_{14}H_{12}O_2S$: C, 68.83; H, 4.95; S, 13.12. Found: C, 68.97; H, 5.16; S, 13.08.

2-[(2-Fluoro-phenyl)-hydroxy-methyl]-indan-1-one (4j)

Beige solid, mp 149.5–151.5 °C. IR (neat, cm^{-1}): 3456, 3056, 2943, 1689, 1620, 1483, 1454, 1228, 1101, 1055, 977, 748, 732, 678 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.80 (br s, 1H), 4.07–4.21 (m, 1H), 4.96 (d, 1H, $J = 2.0$ Hz), 5.08 (d, 1H, $J = 7.5$ Hz), 5.50 (d, 1H, $J = 8.0$ Hz), 7.10-7.21 (m, 2H), 7.27–7.49 (m, 4H), 7.70–7.83 (m, 1H), 7.98–7.92 (m, 1H). ^{13}C-NMR ($CDCl_3$): δ 30.7, 47.5, 67.5, 111.4, 111.5, 123.9-139.0 (7C), 148.4, 159.7, 161.0, 193.5. MS *m/z*: 238 (M-H_2O), 222, 197, 165, 152, 89, 76, 63,51.

Anal. Calcd. for $C_{16}H_{13}FO_2$: C, 74.99; H, 5.11. Found: C, 74.77; H, 5.35.

2-[(2-Bromo-phenyl)-hydroxy-methyl]-indan-1-one (4k)

Pale yellow solid, mp 180.1–182.7 °C. IR (neat, cm^{-1}): 3396, 3086, 2951, 1687, 1645, 1465, 1307, 1298, 1249, 1095, 1058, 1022, 970, 748, 734 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 4.85 (br s, 1H), 5.16 (d, 1H, $J = 4.0$ Hz), 5.29 (d, 1H, $J = 3.0$ Hz), 5.37 (d, 1H, $J = 3.0$ Hz),

6.16 (d, 1H, $J = 12.5$ Hz), 7.02–7.74 (m, 7H), 7.92-7.96 (m, 1H). ^{13}C-NMR ($CDCl_3$): δ 45.2, 72.2, 75.3, 121.0–136.5 (10C), 148.2, 151.2, 193.7. MS *m/z*: 298 (M-H_2O), 222, 197, 165, 152, 89, 76, 63, 51.

Anal. Calcd. for $C_{16}H_{13}BrO_2$: C, 60.59; H, 4.13. Found: C, 60.71; H, 4.22.

1-Hydroxy-1-phenyl-octan-3-one (5a)

Colorless oil. IR (neat, cm^{-1}): 3470, 2933, 2885, 1699, 1607, 1451, 1307, 1235, 763, 742 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 0.80 (t, 3H, $J = 7.0$ Hz), 1.13-1.27 (m, 5H), 1.46–1.52 (m, 2H), 2.33 (t, 2H, $J = 7.5$ Hz), 2.67 (dd, 1H, $J_1 = 3.0$ Hz, $J_2 = 14.0$ Hz), 2.77 (dd, 1H, $J_1 = 9.0$ Hz, $J_2 = 17.5$ Hz), 5.05 (dd, 1H, $J_1 = 3.0$ Hz, $J_2 = 9.5$ Hz), 7.16–7.20 (m, 1H), 7.25–7.26 (m, 4H). ^{13}C-NMR ($CDCl_3$): δ 14.0, 22.9, 25.4, 30.8, 42.6, 51.7, 73.7, 126.7, 126.7, 128.2, 128.2, 128.3, 143.8, 209.7. MS *m/z*: (M) 220, 202, 187, 173, 149, 131, 105, 77, 58.

Anal. Calcd. for $C_{14}H_{20}O_2$: C, 76.33; H, 9.15. Found: C, 76.54; H, 9.25.

1-Hydroxy-1-(4-nitro-phenyl)-octan-3-one (5d)

Pale yellow oil. IR (neat, cm^{-1}): 3338, 2923, 2853, 1615, 1515, 1469, 1384, 1307, 1253, 1176, 1038, 969, 746 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 0.82 (t, 3H, $J = 7.0$ Hz), 1.15–1.25 (m, 4H), 1.49-1.55 (m, 2H), 2.37 (t, 2H, $J = 7.0$ Hz), 2.73–2.76 (m, 2H), 3.56 (brs, 1H), 5.19 (dd, 1H, $J_1 = 4.0$ Hz, $J_2 = 8.5$ Hz), 7.46 (d, 2H, $J = 8.5$ Hz), 8.14 (d, 2H, $J = 9.0$ Hz). ^{13}C-NMR ($CDCl_3$): δ 14.0, 22.5, 23.4, 31.4, 43.8, 50.4, 69.2, 123.8, 124.0, 126.5, 126.7, 147.5, 150.3, 211.3. MS *m/z*: (M-1) 254, 247, 208, 195, 153, 141, 127, 113, 101, 87, 77, 63, 51.

Anal. Calcd. for $C_{14}H_{19}NO_4$: C, 63.38; H, 7.22; N, 5.28. Found: C, 63.51; H, 7.36; N, 5.09.

2-(3-Methyl-benzoyl)-3,4-dihydro-2H-naphthalen-1-one (6b)

Pale orange oil. IR (neat, cm^{-1}): 2985, 2930, 2861, 1726, 1682, 1608, 1528, 1469, 1313, 1208, 1116, 865, 753, 718, 683, 627 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.89 (ddd, 1H, $J_1 = 13.5$ Hz, $J_2 = 8.5$ Hz, $J_3 = 4.5$ Hz), 2.49-2.43 (m, 1H), 2.39 (s, 2H), 2.84 (dd, 2H, $J_1 = 3.5$ Hz, $J_2 = 1.5$ Hz), 4.34 (t, 1H, $J = 1.5$ Hz), 7.18-7.21 (m, 3H), 7.24–7.31 (m, 2H), 7.37 (dd, 1H, $J_1 = J_2 = 8.5$ Hz), 7.52 (ddd, 1H, $J_1 = 7.5$ Hz, $J_2 = 1.5$ Hz, $J_3 = 15$ Hz), 8.13 (dd, 2H, $J_1 = 8.0$ Hz, $J_2 = 1.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 21.3, 25.3, 27.0, 64.2, 123.7, 126.9, 127.2, 128.2, 128.7, 128.8, 129.1, 132.7, 134.0, 134.2, 138.0, 143.3, 193.6, 197.9. MS *m/z*: (M) 264, 247, 233, 221, 203, 193, 178, 131, 119, 115, 103, 91, 78.

Anal. Calcd. for $C_{18}H_{16}O_2$: C, 81.79; H, 6.10. Found: C, 81.63; H, 6.27.

2-(4-Nitro-benzoyl)-3,4-dihydro-2H-naphthalen-1-one (6d)

Pale brown oil. IR (neat, cm^{-1}): 2955, 2920, 2854, 1727, 1696, 1601, 1517, 1462, 1347, 1301, 1232, 1108, 875, 751, 739, 713, 693, 665 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.69–1.74 (m, 1H), 2.34–2.40 (m, 1H), 2.70-2.86 (m, 2H), 4.40 (t, 1H, $J = 1.0$ Hz), 7.17 (d, 1H, $J = 10.0$ Hz), 7.37 (t, 1H, $J = 8.5$ Hz), 7.46–7.51 (m, 3H), 8,04 (d, 1H, $J = 9.5$ Hz), 8.19 (d,

2H, $J = 8.5$ Hz). ^{13}C-NMR ($CDCl_3$): δ 25.2, 28.0, 29.3, 123.9, 123.9, 127.2, 128.8–129.0 (4C), 134.5, 135.7, 141.6, 142.7, 149.3, 191.9, 196.7. MS *m/z*: 295 (M), 269, 265, 208, 192, 178, 145, 131, 115, 103, 90, 63, 51.

Anal. Calcd. for $C_{17}H_{13}NO_4$: C, 69.15; H, 4.44; N, 7.74. Found: C, 69.36; H, 4.52; N, 7.56.

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References

1. M. B. Smith and J. March, "*Advanced Organic Chemistry*," 5th ed., p. 1218, Wiley-Interscience, New York, 2001.
2. F. A. Carey and R. J. Sundberg, "*Advanced Organic Chemistry Part B*," 4th ed., p. 57, Kluwer Academic/PlenumPress, NewYork, 2000.
3. J. H. Furhopp and G. Li, "*Organic Synthesis*," 3rd ed., p. 44, Wiley-VCH, Weinheim, 2003.
4. A. T. Nielsen and W. J. Houlihan, "*Org. React.*," Vol. **16**, p. 1, John Wiley, New York, 1968.
5. H. B. Mekelburger and C. S. Wilcox, "*Comprehensive Organic Synthesis*," Vol. **2**, eds. B. M. Trost and I. Fleming, p. 99, Pergamon Press, 1991.
6. M. M. Green and H. A. Wittcoff, "*Organic Chemistry Principles and Industrial Practice*," 1st. Ed., passim,Wiley-VCH, Weinheim, 2003.
7. P. T. Anastas and J. C. Warner, "*Green Chemistry: Theory and Practice*," p. 29, Oxford University Press, Oxford, 1998.
8. N. Winterton, *Green Chem.*, **3**, G73, (2001).
9. A. D. Curzons, D. J. C. Constable, D. N. Mortimer and V. L. Cunningham, *Green Chem.*, **3**, 1 (2001). doi:10.1039/b007871i
10. M. Eissen, J. O. Metzger, E. Schmidt and U. Schneidewind, *Angew. Chem. Int. Edit.*, **41**, 414 (2002). doi:10.1002/1521-3773(20020201)41:3 < 414::AID-ANIE414 > 3.0.CO;2-N
11. M. Eissen and J. O. Metzger, *Chem.-Eur. J.*, **8**, 3580 (2002). doi:10.1002/1521-3765(20020816)8:16 < 3580::AID-CHEM3580 > 3.0.CO;2-J
12. M. Eissen, R. Mazur, H. G. Quebbemann and K. H. Pennemann, *Helv. Chim. Acta*, **87**, 524 (2004). doi:10.1002/hlca.200490050
13. M. Lancaster, "*Green Chemistry*," p. 69, RSC, Cambridge, 2002.
14. M. Lancaster, "*Handbook of Green Chemistry and Technology*," eds. J. Clark and D. Macquarrie, p. 10, Blackwell, Oxford, 2002.
15. R. J. Lewis, Sr., "*Hazardous Chemical Desk Reference*," 5th ed., passim, Wiley-Interscience, New York, 2002.
16. J. P. Guthrie, *J. Am. Chem. Soc.*, **113**, 7249 (1991). doi:10.1021/ja00019a024
17. J. B. Conant and N. Tuttle, "*Organic Syntheses Coll.*", Vol. **1**, p. 199, John Wiley, New York, 1941.
18. T. Yildiz, H. Yasa, B. Hasdemir and A. S. Yusufoglu, *Monatsh. Chem.*, **148**, 1445 (2017). doi:10.1007/s00706-017-1967-z
19. T. Mukaiyama and K. Narasaka, *J. Syn. Org. Chem. Jpn.*, **40**, 1002 (1982). doi:10.5059/yukigoseikyokaishi.40.1002

20. T. Mukaiyama, *Org. Reactions*, **28**, 203 (1982).
21. A. Cordova, W. Notz and C. F. Barbas, *Chem. Commun.*, 3024 (2002). doi:10.1039/B207664K
22. P. Dziedzic, W. B. Zou, J. Hafren and A. Cordova, *Org. Biomol. Chem.*, **4**, 38 (2006). doi:10.1039/B515880J
23. A. Cordova, W. B. Zou, I. Ibrahem, E. Reyes, M. Engqvist and W. W. Liao, *Chem. Commun.*, 3586 (2005). doi:10.1039/b507968n
24. A. Cordova, W. B. Zou, P. Dziedzic, I. Ibrahem, E. Reyes and Y. M. Xu, *Chem.-Eur. J.*, **12**, 5383 (2006). doi:10.1002/chem.200501639
25. Y. Hayashi, T. Itoh, N. Nagae, M. Ohkubo and H. Ishikawa, *Synlett*, 1565 (2008). doi:10.1055/s-2008-1077789
26. M. Penhoat, D. Barbry and C. Rolando, *Tetrahedron Lett.*, **52**, 159 (2011). doi:10.1016/j.tetlet.2010.11.014
27. Z. Wang, S. M. Richter, J. R. Bellettini, Y. M. Pu and D. R. Hill, *Org. Process. Res. Dev.*, **18**, 1836 (2014). doi:10.1021/op500260n
28. Dimethyl Sulfoxide Producer Association, US Environmental Protection Agency. IUCLID Data Set; Leesburg, VA, September 8, 2003, report number 201-14721A.
29. A. Yanagisawa, T. Ichikawa and T. Arai, *J. Organomet. Chem.*, **692**, 550 (2007). doi:10.1016/j.jorganchem.2006.08.048
30. Y. Orito, S. Hashimoto, T. Ishizuka and M. Nakajima, *Tetrahedron*, **62**, 390 (2006). doi:10.1016/j.tet.2005.09.074
31. T. Ooi, K. Doda and K. Maruoka, *Org. Lett.*, **3**, 1273 (2001). doi:10.1021/ol000382d
32. R. Sudha and S. Sankararaman, *J. Chem. Soc. Perk. T. 1*, 383 (1999). doi:10.1039/a900095j
33. C. H. Cheon and H. Yamamoto, *Tetrahedron*, **66**, 4257 (2010). doi:10.1016/j.tet.2010.03.120
34. D. Acetti, E. Brenna, C. Fuganti, F. G. Gatti and S. Serra, *Eur. J. Org. Chem.*, 142 (2010). doi:10.1002/ejoc.200901006
35. T. Mukaiyama, T. Takuwa, K. Yamane and S. Imachi, *B. Chem. Soc. Jpn.*, **76**, 813 (2003). doi:10.1246/bcsj.76.813
36. R. L. Gao and C. S. Yi, *ACS Catal.*, **1**, 544 (2011). doi:10.1021/cs200087c
37. D. X. Yang, J. F. Huang and B. Liu, *Eur. J. Org. Chem.*, 4185 (2010). doi:10.1002/ejoc.201000484
38. A. V. Kel'in and A. Maioli, *Curr. Org. Chem.*, **7**, 1855 (2003). doi:10.2174/1385272033486134
39. A. V. Kel'in, *Curr. Org. Chem.*, **7**, 1691 (2003). doi:10.2174/1385272033486233
40. N. Mori and H. Togo, *Tetrahedron*, **61**, 5915 (2005). doi:10.1016/j.tet.2005.03.097
41. Y. Wu, Z. C. Geng, J. J. Bai and Y. W. Zhang, *Chinese J. Chem.*, **29**, 1467 (2011). doi:10.1002/cjoc.201180267