SHORT PAPER

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Regiospecific synthesis of carboxylated and simple α-tetralones with homophthalates and various acrylates by a simple condensation method

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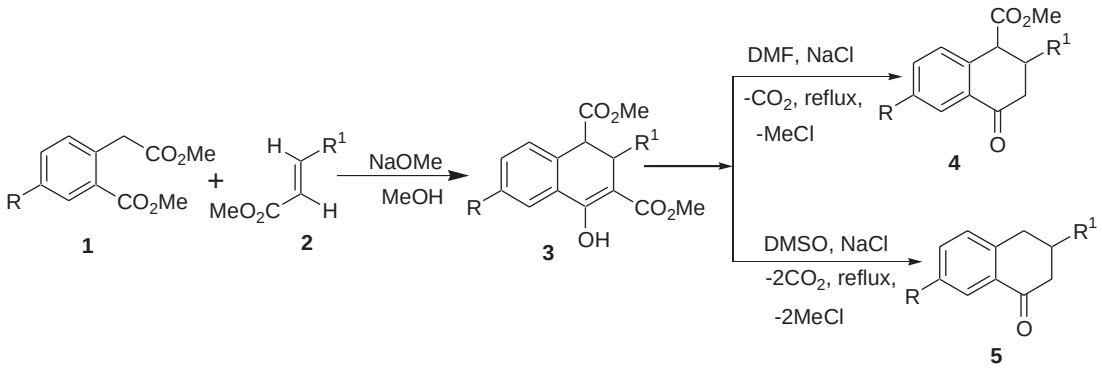
Various carboxylated enols (**3**) have been prepared by taking advantage of simple condensation reactions of various homophthalates (**1**) with different acrylates (**2**), and these enols under heating with two different solvents (DMF and DMSO), afforded ten specific α-tetralones in good to moderate yields using NaCl as a catalyst.

Keywords: carboxylated enols, condensation reactions, homophthalates, acrylates

It has been reported that α-tetralones are the basic units of several tricyclic or tetracyclic natural products.[1, 2] Notable work has been done by Vasavaraju *et al.*[3] who have prepared some substituted tetralones and used them to construct various tricyclic products. In that particular work they have also reported the antimitotic activity of tetralones. After that, until now, very little work has been reported on the efficient synthesis of tetralones.[1,3] In most cases, the routes are lengthy and are not general. An emerging group of antibiotics popularly known as angucyclines, active antitumor agents, in most cases contain a tetralone as one of the basic units.[4–6] In 1984 Rao *et al.*[7] developed a good method for the synthesis of some benzo[*a*]fluorenes and naphthofluorenes via tetralone formation, reacting with some substituted cinnamates and some homophthalates in the presence of NaOEt base. Although the routes are not lengthy, this is not a general procedure for tetralone synthesis. On considering this earlier work, we opted to establish a simple condensation procedure for the synthesis of α-tetralones using different homophthalates and acrylates and have chosen two different acrylates. Also we have selectively chosen two different solvents (DMF and DMSO) for the regiospecific generation of two tetralones in good yields. It has been reported that simple homophthalates, when reacted with crotonates in the presence of NaOMe, afford enols without aromatisation.[8] So, to improve the efficiency of annulation of this simple homophthalate condensation reaction, we have prepared three different homophthalates **1a**, **1b** and **1c** by literature procedures.[9, 10]

All three homophthalic acids were quantitatively esterified by $SOCl_2$ / MeOH treatment.[11] As a test case, the simple homophthalic ester **1a** was subject to condensation with methyl crotonate **2b**. This methyl crotonate was obtained in a slightly different way from commercially available crotonic acid by MeOH and H_2SO_4 (few drops) treatment. The NaOMe base was prepared by direct Na-metal addition to dry methanol. Due to the volatility of crotonate, it was added to the homophthalate anion at 0 °C. After 2 h, this reaction mixture was kept over heated water bath with an efficient $CaCl_2$ guard tube. The reaction was complete after 4 h, confirmed by tlc analysis with 5% $FeCl_3$ solution. The reaction mixture on tlc plates produced a violet colouration, indicating the formation of enol **3a**. The enol was isolated by ether extraction and subsequent column chromatography to give an oily liquid in 80% yield. The IR spectra of the neat liquid showed a –OH peak at $\bar{\nu}$ = 3136 cm^{-1}. Analysis by ^1H NMR spectroscopy proton magnetic resonance spectra showed this –OH as a singlet at δ = 12.37 ppm and few aliphatic protons were found in the region δ = 3.84–3.47 ppm. These two different precis of data firmly indicated the formation of enol **3a** instead of an aromatic compound.



1a, R = H

1b, R = OMe

1c, R = OCH_2Ph

2a, R^1 = H

2b, R^1 = CH_3

2c, R^1 = Ph

3a, 4a & 5a, R = H, R^1 = CH_3

3b, 4b & 5b, R= H, R^1 = H

3c, 4c & 5c, R = OCH_3, R^1 = CH_3

3d, 4d & 5d, R = OCH_3, R^1 = H

3e, 4e & 5e, R = OCH_2Ph, R^1 = CH_3

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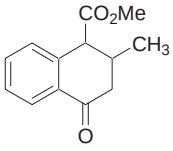
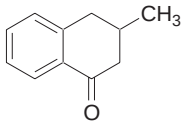
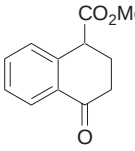
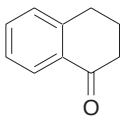
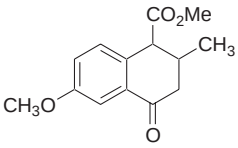
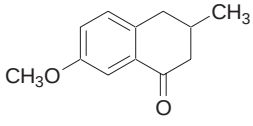
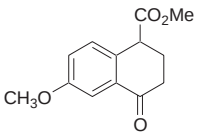
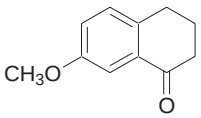
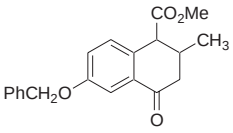
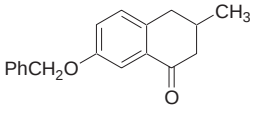
As a test case, to synthesise α-tetralones, the isolated enol **3a** was heated at reflux for 1–1.5 h in DMF containing a catalytic amount of NaCl. At the end of the reaction, no violet colouration was developed on tlc plates by $FeCl_3$ addition and the product was isolated by successive ether extractions. Evaporation of the combined extract and subsequent purification by column chromatographic filtration on silica gel eluting with petroleum ether-ethyl acetate (5: 1), gave a neat liquid in 89% yield. The IR spectra of this neat liquid showed two sharp peaks at ν = 1733 and 1642 cm^{-1} for $–CO_2Me$ and >C=O groups respectively. Moreover, ^1H NMR spectra indicated two important peaks at δ = 3.71 and 1.08 ppm for the $–CO_2Me$ & $–CH_3$ groups respectively. ^{13}C NMR spectra indicated 13 carbons, most importantly, >C=O carbon at 198 ppm. All these data clearly demonstrated the formation of tetralone **4a**, with a carboxylate group at C–1. It is well known that β-ketoesters undergo quick decarboxylation on heating, so, tetralone **4a** is the ultimate result of this reaction. In order to obtain simple α-tetralone **5a,** enol **3a** was heated at a higher temperature in DMSO. Our expectation was that, at higher temperature, due to peri-effect and quicker β-keto acid decarboxylation, the two-carboxylate groups would be removed at the same time. Interestingly, in this way we fortunately obtained a simple α-tetralone **5a**. In favour of the product being **5a,** several spectra (IR, ^1H NMR and ^{13}C NMR) were taken. The IR spectra showed one sharp peak at ν = 1683 cm^{-1} for a >C=O group and no ester functionality. The ^1H NMR spectra indicated one methyl group giving a doublet at δ = 1.20 ppm. The ^{13}C NMR spectra indicated 11 carbons, especially the >C=O carbon at δ=198.76 ppm. According to our assumption, all the individual data supported the unambiguous formation of the simple tetralone **5a**.

Successful preparation of the tetralones **4a** and **5a** prompted us to react the homophthalate **1a** with the acrylate **2a**. In this case, the acrylate was added to homophthalate anion at 0 °C and it was maintained at this temperature for 2 h. The reaction mixture was heated at 54–58 °C with efficient ice cold water circulation through the condenser. Using this procedure, the yield of enol **3a** was improved to 70%. The self-polymerisation tendency of acrylates at higher temperatures gave poor yields of the condensation product **3b**. In a similar fashion, acrylate **2a** and crotonate **2b** were reacted with the three homophthalates **1a, 1b** and **1c** and afforded the respective enols in fair yields. One anomaly was found using methyl cinnamate. Here no enol (<< 7%) was isolated at all. The reaction conditions and the reacting base were changed, but unfortunately, in this case no improved result was obtained. With these prepared enols **3**, similar treatment in DMF and DMSO afforded the various tetralones with good to moderate yields (shown in Table 1). Despite this failure, the simple homophthalate condensation methods not only furnished simple α-tetralones but also at the same time, some rare carboxylated α-tetralones have been synthesised equally effectively. Therefore, the simple homophthalate condensation procedure should complement other available methods.

Experimental

General: The melting points were determined on a capillary melting point apparatus and are uncorrected. Infrared spectra were recorded using KBr pellets for solids and neat liquids on FTIR-8400 and Perkin-Elmer 883 grating spectrometers. ^1H NMR and ^{13}C NMR spectra were taken on AC Bruker 200 MHz spectrometer in $CDCl_3$, containing TMS as the internal standard. Mass spectra were taken on Kratos MS 80 and JEOL JMS-DX 303/JMA-DA-5000 system. All *J* values are given in Hz, chemical shifts in δ units and column chromatography were carried out on 60–120 mesh E. Merck silica gel. No hazardous materials and / or procedures have been adopted during this works.

Table 1

No.	Tetralone obtained in DMF (not dried)	M.p.	Yield/%	No.	Tetralone obtained in DMSO (not dried)	M.p.	Yield/%
4a		liquid	89	**5a**		liquid	92
4b		liquid	53	**5b**		m.p.= 5°C (lit.[12] 5.3–6 °C)	54
4c		low melting solids	70	**5c**		m.p.=37–39 °C	88
4d		low melting solids	58	**5d**		m.p.= 61–63 °C) (lit.[13] = 61-63 °C	65
4e		m.p. 87 – 89 °C	80	**5e**		m.p.=121–123 °C	70

General procedure for enol preparation: Homophthalic ester (1 equiv) was added first at 0 °C to freshly prepared NaOMe (1.5 equiv) in MeOH (5 ml) and then acrylate was added slowly to it. The mixture was heated at 80–85 °C for 4 h and then ice cold 2M HCl (2 ml) was added slowly to it. The reaction mixture was then extracted with diethyl ether (2 × 15 ml). The combined ether extract was worked-up in the usual manner and the resulting residue was purified by column chromatography on silica gel.

General procedure for tetralone preparation: Enols were dissolved into DMF / DMSO (10 ml) and a catalytic amount of NaCl was added to it. This mixture was then heated (157 °C for DMF and 200 °C for DMSO) until the completion of the reaction (1–1.5 h) as indicated by 5% $FeCl_3$ on TLC plates. After that, the reaction mixture was extracted with diethyl ether (3 × 25 ml). The ether extract was worked up in the usual manner and the resulting residue was purified by column chromatography on silica gel**.**

Dimethyl-1, 2-dihydro-4-hydroxy-2-methylnaphthalene-1, 3-dicarboxylate **3a:** Oils. IR: 2936, 1757, 1733, cm^{-1}. ^1H NMR: 12.37 (s, 1 H, enolic -OH) 7.86–7.84 (m, 1 H Ar-H), 7.41–7.34 (m, 2 H, Ar-H) 7.28–7.23 (m, 1 H, Ar-H), 3.84–3.78 (m, 4 H, $-CO_2Me$ and. -CH), 3.78–3.47 (m, 4 H, $-CO_2Me$ and –CH), 1.02 (d, 3 H, *J*= 6.2, $–CH_3$). ^{13}C NMR: 219, 172, 167, 159.5, 145.24, 144.6, 130.32, 128.42, 127.5, 126.8, 121.22, 120.84, 51.6, 50.02, 20.11. (Calc. for $C_{15}H_{16}O_5$: C, 65.13, H, 5.83; found: C, 65.22, H, 5.89 %).

Dimethyl-1, 2-dihydro-4-hydroxynaphthalene-1, 3-dicarboxylate **3b:** Oils. IR: 3427, 1758, 1737, cm^{-1}. ^1H NMR: 12.39 (s, 1 H, enol), 9.02 (dd, 1 H, *J* = 2.4, 8.6, Ar-H), 8.64–8.46 (m, 2 H, Ar-H), 7.75 (dd, 1 H, *J* = 2.1, 8.2, Ar-H), 4.03 (s, 3 H, $-CO_2Me$), 3.48 (s, 3 H, $-CO_2Me$) 3.21–3.07 (m, 1 H, ring- CH), 3.00–2.90 (m, 2 H, ring- CH_2). ^{13}C NMR: 218, 172.5, 168, 154.23, 152.44, 136.8, 128.63, 127.5, 127.15, 121.2, 120.87, 51.9, 51.63, 30.32. (Calc. for $C_{14}H_{14}O_5$: C, 64.09, H, 5.37; found: C, 64.13, H, 5.42 %).

Dimethyl-1, 2-dihydro-4-hydroxy-6-methoxy-2-methylnaphthalene-1, 3-dicarboxylate **3c:** Oils. IR: 2969, 1755, 1732, cm^{-1}. ^1H NMR: 12.42 (s, 1 H, enolic -OH), 7.39 (d, 1 H, *J*= 2.5, Ar-H), 7.18 (d, 1 H, *J*= 8.1, Ar-H), 6.95 (dd, 1 H, *J*= 2.5, 8.1, Ar-H), 3.96 (s, 3 H, -OMe), 3.86 (s, 3 H, $-CO_2Me$), 3.59 (s, 3 H, $-CO_2Me$), 3.83–3.47 (m, 1 H, -CH), 3.45 - 3.40 (m, 1 H, -CH), 1.05 (d, 3 H, *J*= 7.2 $–CH_3$). ^{13}C NMR: 220, 180.6, 172.96, 167, 159.57, 152, 144.51, 131.26, 125.97, 117.07, 100.87, 55.40, 52.22, 44.49, 29.42, 19.42. (Calc. for $C_{16}H_{18}O_6$: C, 62.66, H, 5.91; found: C, 62.72, H, 5.95 %).

Dimethyl-1, 2-dihydro-4-hydroxy-6-methoxynaphthalene-1, 3-dicarboxylate **3d:** Oils. IR: 3430, 1755, 1731, cm^{-1}. ^1H NMR: 12.33 (s, 1 H, enolic -OH), 8.95 (d, 1 H, *J*= 9.2, Ar-H), 7.75 (d, 1 H, *J*= 2.8, Ar-H), 7.39 (dd, 1 H, *J*= 2.8, 9.2, Ar-H), 4.03 (s, 3 H, Ar-OMe), 3.97 (br s, 6 H, 2 × $-CO_2Me$), 3.24–3.10 (m, 1 H, ring–CH), 3.08–2.80 (d, 2 H, ring–CH_2). ^{13}C NMR: 218, 173.2, 167.36, 152.8, 144.5, 142.35, 129.65, 128.7, 127, 120.33, 119.85, 108, 52.1, 51.6, 30.21. (Calc. for $C_{15}H_{16}O_6$: C, 61.56, H, 5.51; found: C, 61.62, H, 5.62 %).

Dimethyl-1, 2-dihydro-4-hydroxy-6-benzyloxy-2-methyl-naphthalene-1,3-dicarboxylate **3e**: Oils. IR: 3441, 1750, 1735, cm^{-1}. ^1H NMR: 12.40 (s, 1 H, enolic -OH), 7.45–7.38 (m, 5 H, Ar-H), 7.37 (d, 1 H, *J*= 2.5, Ar-H), 7.15 (dd, 1 H, *J*= 2.4, 8.1, Ar-H), 6.90 (dd, 1 H, *J*= 2.5, 8.2, Ar-H), 5.06 (s, 2 H, $-OCH_2$), 3.84 (s, 3 H, $-CO_2Me$), 3.60 (s, 3 H, $-CO_2Me$), 3.58–3.47 (m, 1 H, ring–CH), 3.35–3.30 (m, 1 H, ring–CH), 1.03 (d, 3 H, *J*= 7.2, $–CH_3$). ^{13}C NMR: 210, 180.65, 172.61, 167.54, 165.22, 152.44, 144.25, 136.33, 133.52, 130.5, 128.65. 128.54, 128.38, 128.16, 127.8, 127.53, 121.2, 119.6, 70.2, 51.9, 51.78, 20.1. (Calc. for $C_{22}H_{22}O_6$: C, 69.03, H, 5.79; found: C, 69.12, H, 5.85 %).

Methyl-1, 2, 3, 4-tetrahydro-2-methyl-4-oxo-naphthalene carboxylate **4a:** Liquid. IR: 1733, 1642, cm^{-1}. ^1H NMR: 7.99 (dd, 1 H, *J*= 2.1, 8, Ar-H), 7.95–7.73 (m, 3 H, Ar-H), 3.71 (s, 3 H, $-CO_2CH_3$), 2.93–2.80 (m, 1 H, ring–H), 2.70–2.59 (m, 2 H, ring–CH_2), 2.38–2.22 (m, 1 H, ring–H), 1.08 (d, 3 H, *J*= 6.6, $–CH_3$). ^{13}C NMR: 198.0, 180, 177, 140.13, 130.7, 128, 125.2, 123.7, 49.9, 47.12, 35.6, 31.07, 20.2. (Calc. for $C_{13}H_{14}O_3$: C, 71.48, H, 6.46; found: C, 71.37, H, 6.40 %), *m/z* (218).

Methyl-1, 2, 3, 4-tetrahydro-4-oxo-naphthalene carboxylate **4b:** Liquid. IR: 1735, 1690. cm^{-1} ^1H NMR: 7.9 (dd, 1 H, *J*= 2.4, 8, Ar-H), 7.34–7.22 (m, 3 H, Ar-H), 3.75 (s, 3 H, $-CO_2Me$), 3.25–3.1 (m, 1 H, ring–H), 3.0–2.85 (m, 2 H, ring–CH_2), 2.70–2.65 (m, 2 H, ring–CH_2). ^{13}C NMR: 199, 181, 173, 133, 132, 128, 125, 122, 47.5, 39, 33, 32. (Calc. for $C_{12}H_{12}O_3$: C, 70.57, H, 5.92; found: C, 70.43, H, 5.80 %), *m/z* (204).

Methyl–1, 2, 3, 4-tetrahydro-2-methyl-4-oxo-6-methoxynaphthalene carboxylate **4c:** Low melting white colour solids. IR: 1735, 1642, cm^{-1}. ^1H NMR: 7.54 (d, 1 H, *J*= 2.1, Ar –H), 7.16 (s, 1 H, Ar-H), 7.12 (dd, 1 H, *J*= 2.7, 10, Ar –H), 3.80 (s, 3 H, Ar-OMe), 3.76 (s, 3 H, $-CO_2Me$), 3.00–2.97 (m, 1 H –CH), 2.86–2.78 (m, 2 H, $-CH_2$), 2.63–2.34 (m, 1 H, CH), 1.10 (d, 3 H, *J*= 6.0, $–CH_3$). ^{13}C NMR: 199.93, 173.73, 159.27, 152.91, 131.94, 129.94, 122.03, 109.39, 55.45, 52.17, 51.86, 43.77, 32.39, 19.71. (Calc. for $C_{14}H_{16}O_4$: C, 67.73, H, 6.50; found: C, 67.65, H, 6.42 %), *m/z* (248).

Methyl-1, 2, 3, 4-tetrahydro-4-oxo-6-methoxy naphthalene carboxylate **4d**: Low melting white solids. IR: 1733, 1686. ^1H NMR: 7.6 (d, 1 H, *J*= 2.2, Ar-H), 7.55 (s, 1 H, Ar-H), 7.43–7.25 (m, 1 H, Ar-H), 3.83 (s, 3 H, Ar-OMe), 3.75 (s, 3 H, $-CO_2Me$), 2.92–2.80 (m, 4 H, ring 2 ×–CH_2), 2.75–2.71 (m, 1 H, ring–CH). ^{13}C NMR: 195, 170, 160, 152, 130, 128, 120, 107, 53, 51, 49, 42, 31. (Calc. for $C_{13}H_{14}O_4$: C, 66.66, H, 6.02; found: C, 66.55, H, 6.00 %), *m/z* (234).

Methyl-1, 2, 3, 4-tetrahydro-2-methyl-4-oxo-6-benzyloxy naphthalene carboxylate **4e:** IR: 1730, 1675. ^1H NMR: 8.01 (d, 1 H, *J*= 8.4, Ar-H), 7.50–7.36 (m, 7 H, Ar-H), 5.13 (s, 2 H, Ar-CH_2O), 3.73 (s, 3 H, $-CO_2Me$), 3.19–2.98 (m, 1 H, ring–CH), 2.96–2.79 (m, 1 H, ring–CH), 2.48–2.38 (m, 2 H, ring–CH_2), 1.09 (d, 3 H, *J*= 6.4, $-CH_3$). ^{13}C NMR: 199, 177, 175, 168, 163, 158, 136, 134, 131, 129, 128 (2×CH), 127, 126 (2×CH), 65, 49, 48, 45, 20. (Calc. for $C_{20}H_{20}O_4$: C, 74.06, H, 6.21; found: C, 74.01, H, 6.10 %), *m/z* (324).

3-Methyl-3, 4-dihydro-2H–naphthalen-1-one **5a:** This compound was obtained as a colourless liquid in 92 % yield from **3a** following the procedure adopted for **4a** in DMSO solvent. IR: 1683 cm^{-1}. ^1H NMR: 8.03 (dd, 1 H, *J*= 1.80, 7.8, Ar-H), 7.50–749 (m, 1 H, Ar-H), 7.33–7.23 (m, 2 H, Ar-H), 3.00–2.76 (m, 2 H, ring–CH_2), 2.75–2.68 (m, 2 H, ring–CH_2), 2.64–2.34 (m, 1 H, ring-CH), 1.20 (d, 3 H, *J*= 6.3, – CH_3). ^{13}C NMR: 198.76, 157.91, 143.59, 132.31, 128.92, 127.09, 126.69, 47.12, 37.96, 30.39, 21.28. (Calc. for $C_{11}H_{12}O$: C, 82.43, H, 7.54; found: C, 82.32, H, 7.45 %), *m/z* (160).

3,4-Dihydro-2H-naphthalene-1-one **5b:** Low melting solids. m.p. 5–6 °C (lit[12] 5.3 - 6 °C).

2, 4-Dihydro-3-methyl–1(2H)-7-methoxynaphthalene-1-one **5c:** Solids. m.p. 37–39 °C IR: 1673 cm^{-1}. ^1H NMR: 7.80 (br s, 1 H, Ar–H), 6.80 (d, 1 H, *J*= 2.4, Ar–H), 6.67–6.59 (m, 1 H, Ar–H), 3.83 (s, 3 H, -OMe), 3.00–2.91 (m, 2 H, ring–H), 2.59–2.47 (m, 1 H, ring–H), 2.33–2.10 (m, 1 H, ring-H), 1.92–1.77 (m, 1 H, ring–H), 1.25 (d, 3 H, *J*= 6, $-CH_3$). ^{13}C NMR: 198.79, 158.47, 136.57, 133.06, 130.09, 121.94, 108.98, 55.43, 46.94, 37.17, 31.10, 21.22. (Calc. for $C_{12}H_{14}O_2$: C, 75.76, H, 7.42; found: C, 75.45, H, 7.30 %), *m/z* (190).

2, 3, 4-Trihydro-1 (2H)-7-methoxynaphthalene-1-one **5d:** Solids. m.p. 61–62 C (lit[13] 61-63 C).

2, 4-Dihydro-3-methyl-1 (2H)-7-benzyloxynaphthalene-1-one **5e:** White crystalline solids. m.p. 121-123 °C. IR: 1682. ^1H NMR: 7.8 (d, 1 H, *J*= 8.3, Ar-H), 7.70–7.59 (m, 6 H, Ar-H), 7.40 (dd, 1 H, *J*= 2.4, 8.4, Ar-H), 5.20 (s, 2 H, Ar-CH_2O), 3.10–2.89 (m, 2 H, ring–CH_2), 2.47–2.43 (m, 2 H, ring–CH_2), 2.27–2.20 (m, 1 H, ring–CH), 1.20 (d, 3 H, *J* = 6.2, $-CH_3$). ^{13}C NMR: 190, 168, 165, 160, 136, 132, 131, 130, 128, 127, 125, 124, 60, 55, 47, 43, 30, 19. (Calc. for $C_{18}H_{18}O_2$: C, 81.17, H, 6.82; found: C, 81.00, H, 6.70%), *m/z* (266).

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