# Acylation of Aroyl Chlorides *via* a Template Friedel–Crafts Process: Synthesis of Indan-1,3-diones[1]

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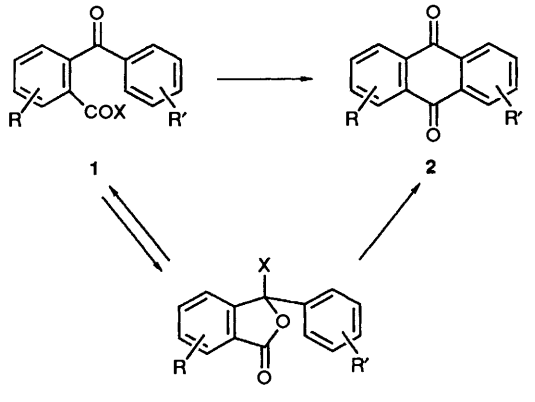
Variously substituted indan-1,3-diones have been regioselectively prepared in a one-pot synthesis by sequential cross-condensation–cycloacylation of aromatic acyl chlorides and acetyl chloride or malonyl dichloride, electrophilic acylation of aromatic acyl chlorides representing the key step in the process. The synergism of the aromatic carbonyl enolization and the organization of the reacting system promoted by the metal is emphasized to account for the mild reaction conditions.

Intramolecular electrophilic acylation has received much attention being a fundamental route to polycyclic ketones and quinones.[2] Several factors affect the yield and selectivity of the reaction, some of which are recognized in the size of the ring to be formed, the nature of the substituents, their position on the aromatic nucleus and the nature of the catalyst. In general, the cycloacylation occurs with ease, except when aromatic substrates highly deactivated toward electrophilic substitution are involved. In such cases drastic experimental conditions are needed.[3,4]

During our studies on the electrophilic acylation of metal phenolates,[5] we evidenced that these unfavoured reactions (Scheme 1, route **1** ⟶ **2**) are easily accomplished *via* a process

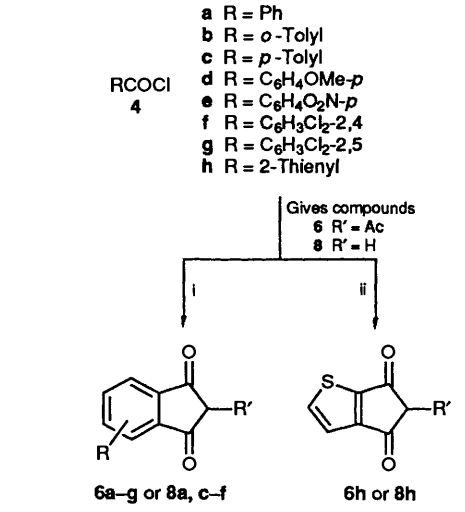



**Scheme 1**

that involves the temporary loss of the electron withdrawing effect of the carbonyl group (Scheme 1, route **1** ⟶ **3** ⟶ **2**).[6] These results prompted us to investigate the possibility of gaining activation and control in the cycloacylation of aromatic and heteroaromatic β-keto acid chlorides.

We now report the synthesis of variously substituted indan-1,3-diones **6** and **8** by sequential cross-condensation–cycloacylation of various acyl chlorides. The complete process requires an unusual electrophilic acylation of aromatic acyl chlorides (Scheme 2). Compounds such as **6** and **8** have been found to be extremely useful in quinone and heterocyclic syntheses.[7]

The most common method of synthesizing indan-1,3-diones has been *via* sodium ethoxide-catalysed condensation of ethyl phthalates and phthalides with carbonyl compounds.[8] These processes suffer from one fundamental drawback due to the limited availability of conveniently substituted starting materials. The reaction here described represents a direct and highly selective route to indan-1,3-diones by electrophilic acylation of easily accessible aromatic acyl chlorides.

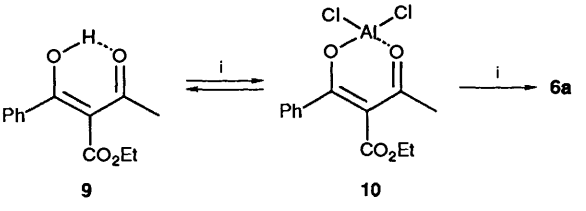



**a**, R = H; **b** R = 4-Me; **c**, R = 5-Me; **d**, R = 5-OMe; **e**, R = $NO_2$; **f**, R = 4,6-$Cl_2$; **g**, R = 4,7-$Cl_2$

**Scheme 2** *Reagents:* i, $AlCl_3$, 2 AcCl **5**; ii, $AlCl_3$, $CH_2(COCl)_2$ **7**

## Results and Discussion

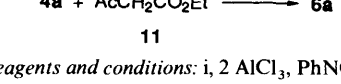
Compound **9** was assumed to be the key intermediate according to the hypothetical reaction pathway reported in Scheme 3. We




**Scheme 3** *Reagent:* i, $AlCl_3$

thought that, due to the extensive enolization of the tricarbonyl moiety of the compound **9** and particularly of the complex **10**, there might be the steric and electronic requisites for the easy cycloacylation **9** ⟶ **6a**.

Ethyl 2-benzoyl-3-oxobutanoate **9** was prepared by selective *C*-benzoylation of the Mg-chelate of ethyl acetoacetate.[9] Further treatment of **9** with $AlCl_3$ (molar ratio 1:2) in dry nitrobenzene at 80 °C for 5 h induced the desired cycloacylation to afford the 2-acetylindan-1,3-dione **6a** in 45% yield. This result




**Scheme 4** *Reagents and conditions:* i, 2 $AlCl_3$, $PhNO_2$, 80 °C, 5 h

**Table 1** Reaction of aromatic acyl chlorides with acetyl chloride

| Entry | ArCOCl | Product | Yield[a] (%) |
|---|---|---|---|
| a | **4a** | **6a** | 87 |
| b | **4b** | **6b** | 68 |
| c | **4c** | **6c** | 80 |
| d | **4d** | **6d** | 75 |
| e | **4e** | **6e** | 35 |
| f | **4f** | **6f** | 67 |
| g | **4g** | **6g** | 58 |
| h | **4h** | **6h** | 72 |

[a] Isolated.

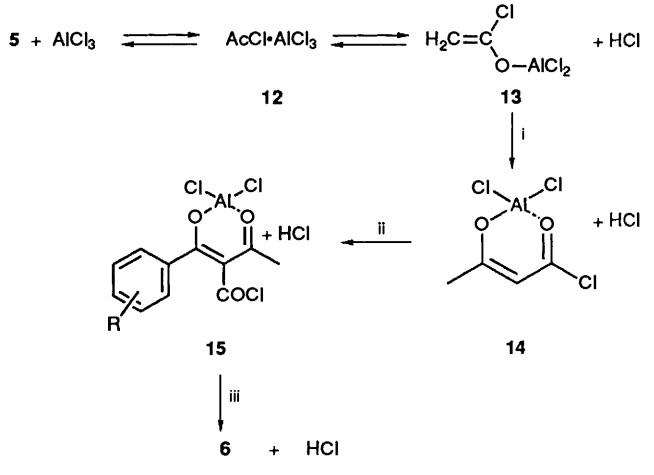
**Table 2** Reaction of aromatic acyl chlorides with malonyl dichloride

| Entry | ArCOCl | Product[a] | Yield[b] (%) |
|---|---|---|---|
| a | **4a** | **8a** | 90 |
| b | **4c** | **8c** | 88 |
| c | **4d** | **8d** | 75 |
| d | **4e** | **8e** | 37[c] |
| e | **4f** | **8f** | 60 |
| f | **4h** | **8h** | 78 |

[a] Variable amounts of bis-indones due to the self-condensation of compounds **8** were also obtained. [b] Isolated. [c] 4-Nitrobenzoic acid was recovered, after quenching, in 6% yield.

prompted us to investigate the direct reaction of ethyl acetoacetate and benzoyl chloride in the presence of $AlCl_3$: the reaction directly afforded compound **6a** in 48% yield. In order to increase the yield of the process and to obtain more detailed information on the mechanism involved, we decided to utilize acetoacetyl chloride which was expected to be more electrophilic and, consequently, more reactive toward cycloacylation than the corresponding ethyl ester.

Although acetoacetyl chloride is extremely unstable and difficult to prepare in satisfactory yield,[10] evidence of its accessibility is provided by studies of acetyl chloride in solution in the presence of Lewis acids. Indeed the complex $AcCl{\cdot}AlCl_3$ when heated in dry nitrobenzene gave substantial quantities of tricarbonyl and polycarbonyl derivatives, depending on the experimental conditions,[11] a reaction assumed to proceed by the intermediacy of acetoacetyl chloride. With this in mind we thought that with judicious choice of reagents, catalyst and experimental conditions it would be possible to trap the unstable intermediate **14** by selective *C*-benzoylation at the active methylene carbon and open a direct 2-acetylindan-1,3-dione synthesis. Thus benzoyl chloride **4a** (0.01 mol) was treated with acetyl chloride **5** (0.025 mol) in the presence of $AlCl_3$ (0.03 mol) in dry nitrobenzene at 80 °C for 5 h to give 2-acetylidan-1,3-dione **6a** in 87% yield (Scheme 5).

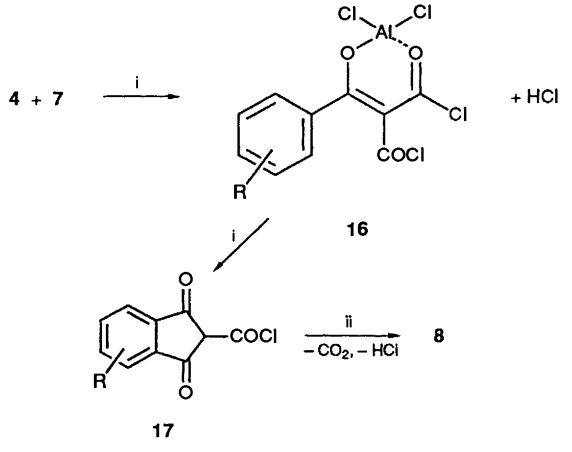



**Scheme 5** *Reagents:* i, $AlCl_3$, **5**; ii, **4**; iii, $AlCl_3$

To study the reaction in depth, aromatic acid chlorides with a variety of substitution patterns on the aromatic ring, were examined. Results from Table 1 show that the process represents an extremely useful synthetic method since it provides a direct, one-pot entry into compounds **6** from acetyl chloride and aromatic acyl chlorides, even for substrates deactivated toward electrophilic substitution (Table 1, entries e, f and g). Published data[11] supports the hypothesis that an $AlCl_3$-promoted aldol-like self-condensation of the acetyl chloride **5** affords the Al-chelate acetoacetyl chloride **14**. *C*-Selective benzoylation of the adduct **14** affords the tricarbonyl derivative **15**.

Recent multinuclear NMR investigations suggest that the major part of the enolization of compounds such as **15** may be ascribed to the β-dicarbonyl framework involving the benzoyl group.[9] This enolization reduces the electron withdrawing power of the aromatic C=O. The selectivity of the process was found to depend more on the sequence of mixture of the reagents and catalyst. Thus, the yield of the compound **6a** rose to 87%, if the acetyl chloride was added dropwise to a solution of benzoyl chloride and aluminium chloride in dry nitrobenzene at 80 °C; if the benzoyl chloride was added, under the above conditions, to a solution of the acetyl chloride and aluminium chloride the product **6a** was obtained in 48% yield accompanied by variable amounts of acetylacetone and polycarbonyl derivatives. Under the above conditions the trimerization of the acetyl chloride competed with the cross-condensation with the benzoyl chloride.[1]

In order to generalize the process, similar reactions with different β-dicarbonyl compounds were investigated. Accordingly, malonyl dichloride **7** was treated with various aromatic acyl chlorides **4** under $AlCl_3$ catalysis.* Selective *C*-benzoylation followed by intramolecular electrophilic acylation




**Scheme 6** *Reagents:* i, $AlCl_3$; ii, $H^+$, $H_2O$

produced the cyclic tricarbonyl derivative **17** which underwent dechlorocarbonylation during the acid quench to afford the indan-1,3-diones **8** in good yields as reported in Table 2.

## Conclusions

The $AlCl_3$-promoted reaction of acetyl chloride and malonyl

* Activated aromatic substrates with an appropriate substitution pattern gave indan-1,3-diones in moderate yields by bis-acylation with malonyl dichloride.[12]

dichloride with a variety of aromatic acyl chlorides provides a novel and direct route to regiospecifically substituted indan-1,3-diones **6** and **8** through the selective *C*-benzoylation of the active methylene carbon of the acetoacetyl chloride or malonyl dichloride followed by cycloacylation. The reaction can be accomplished in a one-pot process by adding the aliphatic acyl chloride to a preformed complex between aluminium chloride and the aromatic acyl chloride. In particular the cycloacylation of the Al-chelate tricarbonyl compounds **15** and **16** (Scheme 5, step **15** ⟶ **6** and Scheme 6, step **16** ⟶ **8**) represents the crucial step of the process. The synergism of the aromatic carbonyl enolization and the organization of the reacting system mediated by the metal, as in the key intermediates **15** and **16**, is emphasized to account for the mild reaction conditions. The process opens the route to the electrophilic acylation of extremely deactivated aromatic substrates.

## Experimental

M.p.s were obtained on an Electrothermal melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer 298 spectrophotometer. UV spectra for solutions in 95% EtOH were measured on a JASCO UVIDEC 505 spectrophotometer. $^1$H NMR spectra were recorded on a Bruker CXP 200 spectrometer at 200 MHz. Chemical shifts are expressed in ppm relative to $Me_4Si$ as internal standard; *J*-values in Hz. Mass spectra were obtained in EI mode on a Finnigan 1020 instrument at 70 eV. Microanalyses were carried out at the Istituto di Chimica Farmaceutica dell'Università degli Studi di Parma, Italy. TLC analyses were performed on Stratocrom SIF silica gel plates (Carlo Erba).

All reagents were of commercial quality from freshly opened containers. $AlCl_3$ was sublimed and all liquid acid chlorides were distilled before use.

*2-Acetylindan-1,3-diones* **6**. *General Procedure.*—Acetyl chloride (2.3 $cm^3$, 0.03 mol) dissolved in dry nitrobenzene (50 $cm^3$) was added, with stirring, at room temperature and under a gentle stream of dry nitrogen to dry $AlCl_3$ (3.99 g, 0.03 mol) and the selected aromatic acyl chloride (0.01 mol) dissolved in dry nitrobenzene (50 $cm^3$). The mixture was stirred at 80 °C for 5 h and then cooled to room temp. and quenched with aqueous oxalic acid (10%, 100 $cm^3$). The solution was extracted with $Et_2O$ (3 × 80 $cm^3$) and then treated with aqueous $Na_2CO_3$ (10%; 2 × 100 $cm^3$). The aqueous layer was washed with $Et_2O$ (50 $cm^3$) and acidified with aqueous HCl (10%) and the resulting mixture was extracted with $Et_2O$ (2 × 100 $cm^3$). The organic extract was dried ($Na_2SO_4$) and evaporated and the product was purified by TLC with PhMe–AcOEt–AcOH (90:5:5) or $CHCl_3$–MeOH (80:20) as eluent.

*Indan-1,3-diones* **8**. *General Procedure.*—These were prepared as described in the preceding method by using freshly distilled malonyl dichloride (1.4 g, 0.01 mol) instead of AcCl. Variable amounts of bis-indones, due to the self-condensation of compounds **4** were also obtained.[13]

*2-Acetylindan-1,3-dione* **6a**. Yellow crystals, m.p. 109–110 °C (EtOH) (lit.,[8a] 109–111 °C); $\nu_{max}$(KBr)/$cm^{-1}$ 1710 (CO) and 1670 (CO).

*2-Acetyl-4-methylindan-1,3-dione* **6b**. Yellow crystals, m.p. 115–116 °C (EtOH) (Found: C, 71.1; H, 4.9. $C_{12}H_{10}O_3$ requires C, 71.28; H, 4.99%); $\nu_{max}$(KBr)/$cm^{-1}$ 1700 (CO) and 1650 (CO); $\delta_H$(200 MHz, $CDCl_3$) 2.56 (3 H, s, $CH_3$), 2.71 (3 H, s, $CH_3Ar$), 7.43 (1 H, d, 5-H, *J* 7.5), 7.57 (1 H, t, 6-H, *J* 7.5) and 7.68 (1 H, d, 7-H, *J* 7.5); *m/z* 202 ($M^+$, 48%), 187 (100), 118 (14) and 103 (15).

*2-Acetyl-5-methylindan-1,3-dione* **6c**. Yellow crystals, m.p. 78–80 °C (EtOH) (Found: C, 71.4; H, 4.9. $C_{12}H_{10}O_3$ requires C, 71.28; H, 4.99%); $\nu_{max}$(KBr)/$cm^{-1}$ 1710 (CO) and 1650 (CO); $\delta_H$(200 MHz, $CDCl_3$) 2.50 (3 H, s, $CH_3Ar$), 2.55 (3 H, s, $CH_3CO$), 7.50 (1 H, dd, 6-H, *J* 7.6, 0.8), 7.63 (1 H, d, 4-H, *J* 0.8), 7.74 (1 H, d, 7-H, *J* 7.6) and 12 (1 H, br s, enolic OH); *m/z* 202 ($M^+$, 54%), 187 (100), 119 (7) and 103 (11).

*2-Acetyl-5-methoxyindan-1,3-dione* **6d**. Orange crystals, decomp. before melting (EtOH) (Found: C, 65.9; H, 4.5. $C_{12}H_{10}O_4$ requires C, 66.05; H, 4.62%); $\nu_{max}$(KBr)/$cm^{-1}$ 1710 (CO) and 1680 (CO); $\delta_H$(100 MHz, $CDCl_3$) 2.55 (3 H, s, $CH_3CO$), 3.94 (3 H, s, $CH_3O$), 7.18 (1 H, d, 6-H, *J* 7.98), 7.27 (1 H, s, 4-H), 7.76 (1 H, d, 7-H, *J* 7.98) and 12 (1 H, br s, enolic OH); *m/z* 218 ($M^+$, 64%), 203 (100), 135 (7) and 119 (12).

*2-Acetyl-5-nitroindan-1,3-dione* **6e**.[14] Yellow crystals, decomp. before melting (EtOH) (Found: C, 56.5; H, 3.1; N, 5.9. $C_{11}H_7NO_5$ requires C, 56.66; H, 3.03; N, 6.01%); $\nu_{max}$(KBr)/$cm^{-1}$ 1700 (CO) and 1630 (CO); $\delta_H$(100 MHz, DMSO) 2.51 (3 H, s, $CH_3$), 7.74 (1 H, br s, 7-H), 8.11 (1 H, br s, 6-H) and 8.41 (1 H, br s, 4-H); *m/z* 233 ($M^+$, 73%), 218 (100), 172 (57) and 103 (10).

*2-Acetyl-4,6-dichloroindan-1,3-dione* **6f**. Yellow crystals, m.p. 222 °C (decomp.) (EtOH) (Found: C, 51.25; H, 2.2; Cl, 27.4. $C_{11}H_6Cl_2O_3$ requires C, 51.39; H, 2.35; Cl, 27.58%); $\nu_{max}$(KBr)/ $cm^{-1}$ 1730 (CO) and 1660 (CO); $\delta_H$(100 MHz, $CDCl_3$) 2.59 (3 H, s, $CH_3$), 7.61 (1 H, d, 5-H, *J* 1.7), 7.72 (1 H, d, 7-H, *J* 1.7) and 12 (1 H, s, enolic OH); *m/z* 260 ($M^+$ + 4, 5%), 258 ($M^+$ + 2, 27), 256 ($M^+$, 40), 241 (100), 173 (10) and 109 (12).

*2-Acetyl-4,7-dichloroindan-1,3-dione* **6g**. Yellow crystals, m.p. 180 °C (decomp.) (EtOH) (Found: C, 51.3; H, 2.2; Cl, 27.4. $C_{11}H_6Cl_2O_3$ requires C, 51.39; H, 2.35; Cl, 27.58%); $\nu_{max}$(KBr)/$cm^{-1}$ 1700 (CO) and 1660 (CO); $\delta_H$(100 MHz, DMSO) 2.59 (3 H, s, $CH_3$) and 7.42 (2 H, s, ArH); *m/z* 260 ($M^+$ + 4, 3%), 258 ($M^+$ + 2, 12), 256 ($M^+$, 35), 241 (100), 214 (50), 146 (28) and 109 (48).

*5-Acetylcyclopenta[b]thiophene-4,6-dione* **6h**. Yellow solid, decomp. before melting (Found: C, 55.75; H, 3.1; S, 16.4. $C_9H_6O_3S$ requires C, 55.68; H, 3.16; S, 16.48%); $\nu_{max}$(KBr)/$cm^{-1}$ 1720 (CO) and 1670 (CO); $\delta_H$(100 MHz, $CDCl_3$) 2.28 (3 H, s, $CH_3$), 7.23 (1 H, d, 3-H, *J* 4.36), 8.00 (1 H, d, 2-H, *J* 4.36) and 12.00 (1 H, br s, enolic OH); *m/z* 194 ($M^+$, 48%), 179 (100) and 123 (7).

*Indan-1,3-dione* **8a**. Pale yellow crystals, m.p. 131–132 °C (EtOH) [authentic sample (Aldrich), 130 °C].

*5-Methylindan-1,3-dione* **8c**. Pale yellow crystals, m.p. 114 °C (lit.,[15] 114–116 °C) (EtOH) (Found: C, 75.1; H, 5.2. $C_{10}H_8O_2$ requires C, 74.99; H, 5.03%); $\nu_{max}$(KBr)/$cm^{-1}$ 1705 (CO); $\delta_H$(100 MHz, $CDCl_3$) 2.55 (3 H, s, $CH_3$), 3.23 (2 H, s, $CH_2$), 7.64 (1 H, dd, 6-H, *J* 7.74, 0.85), 7.74 (1 H, d, 4-H, *J* 0.85) and 7.88 (1 H, d, 7-H, *J* 7.74); *m/z* 160 ($M^+$, 100%), 132 (28), 118 (63) and 104 (50).

*5-Methoxyindan-1,3-dione* **8d**. Yellow solid, m.p. 116–118 °C (lit.,[16] 118–119 °C) (Found: C, 68.05; H, 4.7. $C_{10}H_8O_3$ requires C, 68.18; H, 4.58%); $\nu_{max}$(KBr)/$cm^{-1}$ 1710 (CO); $\delta_H$(100 MHz, $CDCl_3$) 3.63 (2 H, s, $CH_2$), 3.96 (3 H, s, $CH_3$), 7.33 (1 H, d, 4-H, *J* 2.30), 7.34 (1 H, dd, 6-H, *J* 9.18, 2.30) and 7.89 (1 H, d, 7-H, *J* 9.18); *m/z* 176 ($M^+$, 100%), 148 (15), 120 (28) and 106 (45).

*5-Nitroindan-1,3-dione* **8e**. Yellow solid, m.p. 115–116 °C (lit.,[17] m.p. 113 °C) (Found: C, 56.7; H, 2.7; N, 7.2. $C_9H_5NO_4$ requires C, 56.55; H, 2.64; N, 7.33%); $\nu_{max}$(KBr)/$cm^{-1}$ 1700 (CO); $\delta_H$(100 MHz, $CDCl_3$) 3.40 (2 H, s, $CH_2$), 8.17 (1 H, d, 7-H, *J* 8.26), 8.35 (1 H, dd, 6-H, *J* 8.26, 2.0) and 8.79 (1 H, d, 4-H, *J* 2.0); *m/z* 191 ($M^+$, 30%), 149 (60), 123 (23), 69 (95) and 57 (100).

*4,6-Dichloroindan-1,3-dione* **8f**. Yellow crystals, m.p. 222 °C (decomp.) (EtOH) (Found: C, 50.1; H, 2.0; Cl, 32.9. $C_9H_4Cl_2O_2$ requires C, 50.26; H, 1.87; Cl, 32.97%); $\nu_{max}$(KBr)/$cm^{-1}$ 1720 (CO); $\delta_H$(100 MHz, $CDCl_3$) 3.31 (2 H, s, $CH_2$), 7.76 (1 H, d, 5-H, *J* 1.7), 7.85 (1 H, d, 7-H, *J* 1.7); *m/z* 218 ($M^+$ + 4, 60%), 216 ($M^+$ + 2, 11), 214 ($M^+$, 100), 186 (44), 172 (50), 144 (40) and 74 (52).

*Cyclopenta[b]thiophene-4,6-dione* **8h**.[18] Yellow crystals, m.p.

120 °C (decomp.) (EtOH) (Found: C, 55.1; H, 2.5; S, 21.2. $C_7H_4O_2S$ requires C, 55.25; H, 2.65; S, 21.06%); $\nu_{max}$(KBr)/cm$^{-1}$ 1710 (CO); $\delta_H$(100 MHz, $CDCl_3$) 3.49 (2 H, s, $CH_2$), 7.39 (1 H, d, 4-H, *J* 4.88), 7.99 (1 H, d, 5-H, *J* 4.88); *m/z* 152 ($M^+$, 58%), 124 (15), 110 (97) and 96 (100).

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