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# FACILE ONE-POT SYNTHESIS OF TRICYCLIC/POLYCYCLIC LACTONES

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**Abstract** - The reaction of substituted or unsubstituted 2-hydroxybenzaldehydes with 5, 6-dihydropyran-2-one in the presence of DMAP in DMSO afforded tricyclic/polycyclic lactones in good yields.

## INTRODUCTION

Lactone moiety is an important substructure present in many biological active compounds.[1-3] Also the lactone unit constitutes an interesting and important part of many natural molecules, many of which possess antitumor[4-9] and antifungal activities.[10-13] In the literature a number of methods are reported for the preparation of bioactive compounds containing lactone structure unit, and development of novel and efficient methods for the synthesis of these important compounds still continues to be an attractive area in organic chemistry.[14-19] During the course of our drug research we developed a convenient and one pot synthesis of tricyclic/polycyclic lactones under Baylis-Hillman conditions.

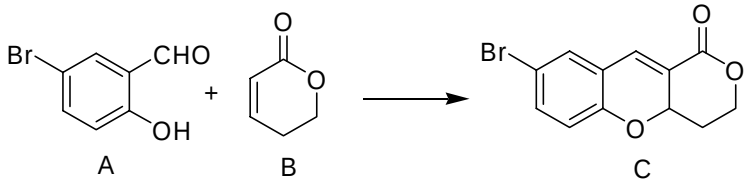
The Baylis-Hillman-type carbon-carbon bond formation has become an active area in synthetic organic chemistry in recent years,[20,21] but the direct carbon-carbon bond formation under Baylis-Hillman conditions to give novel heterocycles has not been well documented.[22-24] Although there are some reports on the reactions of 2-hydroxybenzaldehydes with various activated alkenes[25-29] and 2-hydroxybenzaldehyde derivatives with cyclic enones[30,31] and with chromone derivatives,[32] yet only a few reports described the less reactive activated β-substituted-α, β-unsaturated lactones as Baylis-Hillman substrates.[33]

Recently, we developed a new method for the synthesis of tricyclic/polycyclic lactones via a one-pot tandem reaction of 5, 6-dihydropyran-2-one and 2-hydroxybenzaldehydes in the presence of DMAP in DMSO.

## RESULTS AND DISCUSSION

Initially the reaction was carried out by reacting 5-bromo-salicylaldehyde with 5, 6- dihydropyran-2-one in the presence of different bases such as DMAP, DABCO, DBU, HMT (hexamethylenetetramine), imidazole, quinuclidine and quinine, as catalyst, and dichloromethane or THF as solvent at room temperature for 3 days. Unfortunately, no desired product was observed. Raising the reaction temperature resulted in some improvement, the results were summarized in Table 1.

**Table 1**. Synthesis of 8-bromo-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one in the presence of different bases and different solvents [a]




| Entry | Catalyst | Solvent | Conditions | Yield[b] % ( )[c] ( )[d] |
|---|---|---|---|---|
| 1 | quinuclidine | MeOH | reflux, 2 days | 13 (86) (11) |
| 2 | quinine | THF | reflux, 3 days | 53 (90) (48) |
| 3 | DMAP | $CH_2Cl_2$ | reflux, 3 days | 71 (80) (57) |
| 4 | DMAP | DMSO | 60℃, 3 days | 74 (83) (61) |
| 5 | quinine | DMSO | 60℃, 3 days | 59 (64) (38) |
| 6 | DMAP | DMSO | 60℃, 5 days | 75 (85) (64) |

[a] Reaction conditions: a stirred mixture of **A** (0.5 mmol) and **B** (0.6 mmol) in different solvent (2 mL) in the presence of different catalyst (0.5 mmol) was maintained at reflux or 60 ℃ for 3 days.

[b] values refer to yield based on consumed starting salicylaldehyde.

[c] conversion yield based on salicylaldehyde.

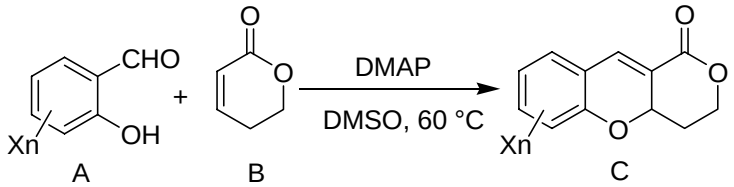
[d] Isolated yield based on salicylaldehyde.

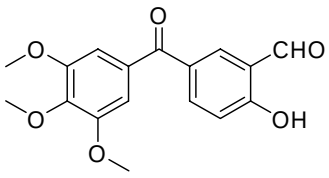
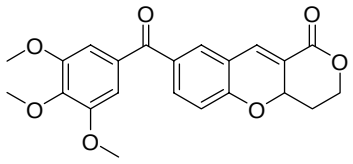
It was reported that a combination of quinuclidine and methanol is the most optimum protocol for the Baylis-Hillman reaction of less reactive activated olefins such as 5,6-dihydropyran-2-one.[33] But in Table 1, we found that quinuclidine and methanol was not a good protocol for this reaction. And we noticed that quinine and DMAP as catalyst in heating condition could afford the product in good yield. We also observed that the use of DMAP was superior to quinine and the use of DMSO as solvent improved the yields a little. When the reaction time was prolonged to 5 days, the conversion yield was increased only 2% compared with that for 3 days (Table 1, entries 4 and 5). The use of DMAP (1 equiv) as catalyst and DMSO

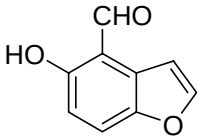
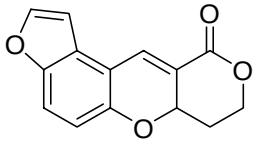
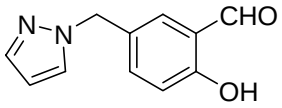
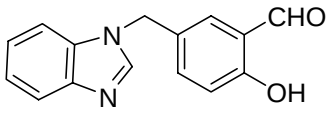
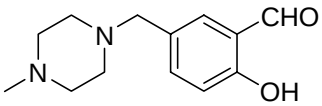
as solvent in 60 ℃ for 3 days gave 74% yield, and this was chosen as the optimized condition (Table 1, entry 4).

The reaction of salicylaldehydes **(A)** and 5,6-dihydropyran-2-one (**B**) in DMSO in the presence of DMAP at 60 ℃ gave 4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one **(C)** in 31 – 74 % yields. The results are summarized in Table 2.

**Table 2**. Synthesis of substituted 4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one in the presence of DMAP and DMSO [a]




| Entry | Salicylaldehydes (A) | Products (C) | Time (day) | Yield(%) [b] ( )[c] |
|---|---|---|---|---|
| 1 | H | 1 | 3 | 63 (95) |
| 2 | 5-Br | 2 | 3 | 74 (83) |
| 3 | 5-Cl | 3 | 3 | 60 (84) |
| 4 | 5-F | 4 | 1 | 52 (100) |
| 5 | 3-F | 5 | 3 | 65 (90) |
| 6 | 5-I | 6 | 3 | 52 (71) |
| 7 | 5-$CH_3$ | 7 | 3 | 54 (79) |
| 8 | 3-$CH_3$ | 8 | 5 | 43 (85) |
| 9 | 5-Ph | 9 | 3 | 72 (95) |
| 10 | 3-Ph | 10 | 3 | 64 (79) |
| 11 | 5-OMe | 11 | 3 | 63 (79) |
| 12 | 4-OMe | 12 | 3 | 57 (60) |
| 13 | 5-Ac | 13 | 3 | 48 (81) |
| 14 | 5-CN | 14 | 3 | 71 (80) |
| 15 | 5-$NO_2$ | 15 | 5 | 31 (52) |
| 16 |  |  | 5 | 51 (58) |

| | | | | |
|---|---|---|---|---|
| 17 |  |  | 3 | 80(38) |
| 18 |  | - | 5 | 0[d] |
| 19 |  | - | 5 | 0[d] |
| 20 |  | - | 5 | 0[d] |

[a] Reaction conditions: a stirred mixture of **A** (0.5 mmol) and **B** (0.6 mmol) in DMSO(2 mL) in the presence of DMAP (0.5 mmol) was maintained at 60 ℃ for 1-5 days.

[b] values refer to yield based on consumed starting salicylaldehyde.

[c] conversion yield based on salicylaldehyde.

[d] without DMAP.

Both electron-rich and electron-deficient salicylaldehydes are suitable substrates for this reaction to provide the corresponding 4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one in good yields, among which 5-bromo-2-hydroxybenzaldehyde gave the highest yield (Table 2, entry 2). A variety of functional groups of salicylaldehydes are known to tolerate this reaction condition, which include methoxy, methyl, halide (fluoro, chloro, bromo and iodo), phenyl, carbonyl and cyano groups. But salicylaldehyde with nitro group (Table 2, entry 15) and 5-hydroxybenzofuran-4-carbaldehyde (Table 2, entry 17) showed lower reactivity, and the steric hindrance of the ortho position of carbaldehyde was the possible reason for entry 17. That is very interesting, when we assumed a self-catalyst Baylis-Hillman reaction, no product could be observed. We used the salicylaldehydes with heterocyclic group such as imidazole and piperazine, no new compounds were observed without DMAP. And with DMAP, by TLC we observed a succession of minor points were produced except for the unconsumed salicylaldehydes. We did not get the desired products. (Table 2, entries 18-20).And we examined the reaction of salicylaldehyde and furan-2(5*H*)-one in DMSO in the presence of DMAP at 60 ℃, the corresponding tricyclic lactone was not obtained.

In conclusion, we disclosed a facile synthesis of novel 4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one from the reaction of salicylaldehydes and 5,6-dihydropyran-2-one for the first time in reasonable yields. And the new combination of DMAP and DMSO as one of the most effective systems may find further application in Baylis-Hillman reactions.

## EXPERIMENTAL

Melting points were measured by Buchi 510 melting point apparatus and were uncorrected. The $^1$H-NMR spectra were recorded by GEMINI spectrometer at 300 MHz and $^{13}$C-NMR spectra were recorded by Brucker AM-400 spectrometer at 400MHz. Ms and HRMS spectra were recorded on a MAT-95 spectrometer. Elemental analysis for carbon, hydrogen and nitrogen were performed on a Carlo-Erba-1110 elemental analyzer.

### 8-Bromo-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C2) (General procedure)

A stirred mixture of 5-bromo-2-hydroxybenzaldehyde (101 mg, 0.5 mmol) and 5,6-dihydropyran-2-one (59 mg, 0.6 mmol) in DMSO (2 mL) in the presence of DMAP (61 mg, 0.5 mmol) was maintained at 60 ℃ for 3 days. The workup involved adding $H_2O$ (15 mL) to the reaction mixture followed by extraction with EtOAc. The combined organic phases were dried ($Na_2SO_4$) and concentrated. The residue was purified by silica-gel column chromatography to give 5-bromo-2-hydroxybenzaldehyde 17 mg and the product 8-bromo-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one with 74% yield (value refer to yield based on consumed starting salicylaldehyde) as a white solid, mp 172-174 ℃. $^1$H-NMR(300MHz,$CDCl_3$) δ2.39-2.48(2H, m), δ4.27-4.33(1H, m), δ4.52(1H, m), δ5.03-5.08(1H, m), δ6.80(1H, d, *J*=8.5Hz), δ7.35-7.39(2H, m), δ7.59(1H, d, *J*=2.0Hz). $^{13}$C-NMR(400MHz, $CDCl_3$) δ29.02, δ64.19, δ71.26, δ114.52, δ118.00, δ121.40, δ123.73, δ131.58, δ134.98, δ135.07, δ154.17, δ163.62. MS(EI) *m/z*(%): 282(14), 280($M^+$, 15), 238(18), 236(20), 210 (12), 208(13), 149(100). Anal. Calcd for $C_{12}H_9BrO_3$: C, 51.27; H, 3.23. Found: C, 51.16; H, 3.23.

### 4,4*a*-Dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C1)

White solid in 63% yield, mp 172-174 ℃. $^1$H-NMR(300MHz,$CDCl_3$) δ2.40-2.49(2H, m), δ4.26-4.34(1H, m), δ4.87-4.55(1H, m), δ5.04-5.10(1H, m), δ6.91(1H, d, *J*=8.1Hz), δ7.00(1H, td, *J*=1.1, 7.5Hz), δ7.23-7.33(2H, m), δ7.67(1H, d, *J*=2.3Hz). MS(EI) *m/z*(%): 202($M^+$, 100), 158(92), 149(30), 144(33), 130(84), 115(40), 102(28). HRMS(EI) calcd. For $C_{12}H_{10}O_3$: 202.0630; found: 202.0614.

### 8-Chloro-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C3)

White solid in 60% yield, mp 186-188 ℃. $^1$H-NMR(300MHz,$CDCl_3$) δ2.36-2.49(2H, m), δ4.27-4.33(1H, m), δ4.50-4.55(1H, m), δ5.03-5.08(1H, m), δ6.85(1H, d, *J*=8.8Hz), δ7.21-7.25(2H, m), δ7.59(1H, d, *J*=1.5Hz). $^{13}$C-NMR(400MHz, $CDCl_3$) δ29.03, δ64.21, δ71.30, δ117.60, δ121.47, δ123.20, δ127.36, δ128,65, δ132.09, δ135.20, δ153.67, δ163.68. MS(EI) *m/z*(%): 238(32), 236($M^+$, 86), 194(33), 192(100), 178(35), 164(82), 115(32). Anal. Calcd for $C_{12}H_9ClO_3$: C, 60.90; H, 3.83; found: C, 60.60; H, 3.83.

### 8-Fluoro-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C4)

White solid in 52% yield, mp 193-195 ℃. $^1$H-NMR(300MHz,$CDCl_3$) δ2.38-2.48(2H, m), δ4.27-4.33(1H, m), δ4.26-4.33(1H, m), δ5.01-5.06(1H, m), δ6.86-6.88(1H, m), δ6.94-7.02(2H, m), δ7.60(1H, d,

*J*=2.2Hz). MS(EI) *m/z*(%): 220($M^+$, 94), 176(100), 148(92), 133(50), 120(30). HRMS(EI) calcd. For $C_{12}H_9FO_3$: 220.0536; found 220.0535.

**6-Fluoro-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C5)**

White solid in 65% yield, mp 215-217 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.41-2.57(2H, m), δ4.27-4.36(1H, m), δ4.51-4.57(1H, m), δ5.09-5.15(1H, m), 6.91-6.97(1H, m), δ7.03-7.15(2H, m), δ7.68-7.69(1H, m). MS(EI) *m/z*(%): 220($M^+$, 88), 176(100), 148(82), 133(46), 120(28). HRMS(EI) calcd. For $C_{12}H_9FO_3$: 220.0536; found 202.0536.

**4,4*a*-Dihydro-8-iodopyrano[4,3-*b*]chromen-1(3*H*)-one (C6)**

Yellow solid in 52% yield, mp 176-178 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.39-2.47(2H, m), δ4.25-4.34(1H, m), δ4.48-4.55(1H, m), δ5.03-5.09(1H, m), δ6.67(1H, d, *J*=9.1Hz), δ7.53-7.58(3H, m). MS(EI) *m/z*(%): 328($M^+$, 100), 284(84), 256(58), 149(32). HRMS(EI) calcd. For $C_{12}H_9IO_3$: 327.9596; found 327.9591.

**4,4*a*-Dihydro-8-methylpyrano[4,3-*b*]chromen-1(3*H*)-one (C7)**

Yellowish solid in 54% yield, mp 182-184 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.30(3H, s), δ2.39-2.47(2H, m), δ4.25-4.33(1H, m), δ4.48-4.54(1H,m), δ4.99-5.05(1H, m), δ6.81(1H, d, *J*=8.2Hz), δ7.04-7.12(2H, m), δ7.67(1H, d, *J*=2.2Hz). MS(EI) *m/z*(%): 216($M^+$, 92), 172(88), 149(64), 144(100), 115(42). HRMS(EI) calcd. For $C_{13}H_{12}O_3$: 216.0786; found 216.0783.

**4,4*a*-Dihydro-6-methylpyrano[4,3-*b*]chromen-1(3*H*)-one (C8)**

Yellowish solid in 43% yield, mp 150-152 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.22(3H, s), δ2.42-2.51(2H, m), δ4.26-4.35(1H, m), δ4.49-4.45(1H, m), δ5.01-5.07(1H, m), δ6.88-6.92(1H, m), δ7.08-7.26(2H, m), δ7.67(1H, d, *J*=2.1Hz). MS(EI) *m/z*(%): 216($M^+$, 100), 172(70), 144(90), 115(38). HRMS(EI) calcd. For $C_{13}H_{12}O_3$: 216.0786; found 216.0779.

**4,4*a*-Dihydro-8-phenylpyrano[4,3-*b*]chromen-1(3*H*)-one (C9)**

White solid in 72% yield, mp 170-172 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.42-2.51(2H, m), δ4.27-4.36(1H, m), δ4.50-4.57(1H, m), δ5.07-5.13(1H, m), δ6.99(1H, d, *J*=8.6), δ7.32-7.37(1H, m), δ7.41-7.46(3H, m), δ7.51-7.55(3H, m), δ7.74(1H, d, *J*=2.3Hz). MS(EI) *m/z*(%): 278($M^+$, 100), 234(60), 206(92), 149(30). HRMS(EI) calcd. For $C_{18}H_{14}O_3$: 278.0943; found 278.0935.

**4,4*a*-Dihydro-6-phenylpyrano[4,3-*b*]chromen-1(3*H*)-one (C10)**

White solid in 64% yield, mp 150-152 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.37-2.45(2H, m), δ4.23-4.32(1H, m), δ4.47-4.53(1H, m), δ5.02-5.08(1H, m), δ7.06-7.11(1H, m), δ7.25-7.27(1H, m), δ7.36-7.46(4H, m), δ7.52-7.55(2H, m), δ7.73(1H, d, *J*=2.4Hz). MS(EI) *m/z*(%): 278($M^+$, 100), 234(88), 206(72), 149(80). HRMS(EI) calcd. For $C_{18}H_{14}O_3$: 278.0943; found 278.0940.

**4,4*a*-Dihydro-8-methoxypyrano[4,3-*b*]chromen-1(3*H*)-one (C11)**

White solid in 63% yield, mp 150-152 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.33-2.50(2H, m), δ3.79(3H,s), δ4.24-4.33(1H, m), δ4.48-4.54(1H, m), δ4.96-5.02(1H, m), δ6.76 (1H, d, *J*=1.8Hz), δ6.86-6.87(2H, m), δ7.64(1H, d, *J*=2.1Hz). MS(EI) *m/z*(%): 232($M^+$, 100), 188(60), 160(96), 99(30). HRMS(EI) calcd. For $C_{13}H_{12}O_4$: 232.0736; found 232.0730.

**4,4*a*-Dihydro-7-methoxypyrano[4,3-*b*]chromen-1(3*H*)-one (C12)**

White solid in 57% yield, mp 132-134 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.38-2.47(2H, m), δ3.81(3H, s), δ4.23-4.32(1H, m), δ4.46-4.53(1H, m), δ5.00-5.06(1H, m), δ6.46(1H, d, *J*=2.3Hz), δ6.56(1H, dd, *J*=2.4, 8.5Hz), δ7.17(1H, d, *J*=8.4Hz), δ7.65(1H, d, *J*=2.2Hz). MS(EI) *m/z*(%): 232($M^+$, 95), 188(48), 160(100), 149(64). HRMS(EI) calcd. For $C_{13}H_{12}O_4$: 232.0736; found 232.0733.

**8-Acetyl-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C13)**

White solid in 48% yield, mp 202-204 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.43-2.53(2H, m), δ2.57(3H, s), δ4.10-4.36(1H, m), δ4.51-4.57(1H, m), δ5.12-5.18(1H, m), δ6.96(1H, d, *J*=8.5Hz), δ7.70(1H, d, *J*=2.4Hz), δ7.86(1H, d, *J*=2.3Hz), δ7.93(1H, dd, *J*=2.1, 8.4Hz). MS(EI) *m/z*(%): 244($M^+$, 100), 229(30), 200(80), 172(88), 157(30), 149(40), 57(28). HRMS(EI) calcd. For $C_{14}H_{12}O_4$: 244.0736; found 244.0741.

**1,3,4,4*a*-Tetrahydro-1-oxopyrano[4,3-*b*]chromene-8-carbonitrile (C14)**

White solid in 71% yield, mp 262-264 °C. $^1$H-NMR(300MHz,CDCl3) δ2.44-2.56(2H, m), δ4.29-4.36(1H, m), δ4.52-4.57(1H, m), δ5.15-5.20(1H, m), δ6.98(1H, d, *J*=8.3Hz), δ7,54-7.58(2H, m), δ7.61(1H, d, *J*=2.2Hz). MS(EI) *m/z*(%): 227($M^+$, 68), 183(100), 155(80), 140(28), 127(36). HRMS(EI) calcd. For $C_{13}H_9NO_3$: 227.0582; found 227.0584.

**4,4*a*-Dihydro-8-nitropyrano[4,3-*b*]chromen-1(3*H*)-one (C15)**

White solid in 31% yield, mp 213-215 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.40-2.56(2H, m), δ4.29-4.38(1H, m), δ4.52-4.59(1H, m), δ5.19-5.25(1H, m), δ7.01(1H, d, *J*=8.8Hz), δ7.68(1H, d, *J*=2.5Hz), δ8.15-8.21(2H, m). MS(EI) *m/z*(%): 247($M^+$, 50), 203(100), 175(58). HRMS(EI) calcd. For $C_{12}H_9NO_5$: 247.0481; found 247.0484.

**8-(3,4,5-Trimethoxybenzoyl)-4,4*a*-dihydropyrano[4,3-*b*]chromen-1(3*H*)-one (C16)**

Yellowish solid in 51% yield, mp 224-226 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.44-2.57(2H, m), δ3.88(6H, s), δ3.94(3H, s), δ4.28-4.37(1H, m), δ4.51-4.58(1H, m), δ5.16-5.22(1H, m), δ6.99-7.01(3H, m), δ7.69(1H, d, *J*=1.8Hz), δ7.74(1H, d, *J*=2.0Hz), δ7.80(1H, dd, *J*=2.0, 8.5Hz). MS(EI) *m/z*(%): 396($M^+$,100), 324(30), 195(18). HRMS(EI) calcd. For $C_{22}H_{20}O_7$: 396.1209; found 396.1187.

**7,8-Dihydrofuro[3,2-*f*]pyrano[4,3-*b*]chromen-10(6a*H*)-one (17)**

Yellowish solid in 80% yield, mp 150-152 °C. $^1$H-NMR(300MHz,$CDCl_3$) δ2.44-2.52(2H, m), δ4.10-4.36(1H, m), δ4.51-4.58(1H, m), δ5.04-5.11(1H, m), δ6.86-6.89(2H, m), δ7.42(1H, dd, *J*=1.0, 8.9Hz), δ7.70(1H, d, *J*=2.2Hz), δ7.95(1H, d, *J*=2.2Hz). MS(EI) *m/z*(%): 242($M^+$,100), 198(76), 170(90),

149(26), 57(20). HRMS(EI) calcd. For $C_{14}H_{10}O_4$: 242.0579.1209; found 242.0583.

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