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Metal carbenoid mediated ring opening of furan-2-yloxy derivatives. Stereoselective synthesis of 2,4-hexadienedioic acid and 6-aryl-1-oxo-2,4-hexadienoic acid derivatives

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Abstract—2-Methoxy- and 2-trimethylsilyloxyfuran undergo facile ring opening reaction upon treatment with metal carbenoid. Treatment of 2-methoxyfuran with ethyl diazoacetate and aryl-α-diazocarbonyl compounds under metal catalysis afforded (2*Z*,4*E*)-hexadienedioate and 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoates respectively. When 2-trimethylsilyloxyfuran was used, desilylation occurred to give directly the mono-protected (*Z*,*E*)-muconic acid and 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acids. © 2001 Elsevier Science Ltd. All rights reserved.

1. Introduction

Rhodium(II) acetate has been a superior catalyst for the generation of transient electrophilic metal carbenoid from α-diazocarbonyl compounds[1] and these undergo inter- or intramolecular carbenoid addition[2–4] and insertion reaction.[5–8] In recent years the reactions of this transient electrophilic carbenoid with furan and 2-alkylfuran have been recognized as one of the most convenient routes to 1,6-dioxo 2,4-diene derivatives, and are used for the synthesis of a number of natural products[9] and heterocyclic systems.[10] Furthermore, a variation in the products was encountered by varying the nature of the substituents attached to the 2-position of furan upon treatment with the transient electrophilic metal carbenoid generated from rhodium(II) acetate catalyst. One important finding is that electron-withdrawing substituents at the 2-position of the furan ring do not inhibit the cyclopropanation but interfere with the ring opening reaction.[11] Although furan and 2-alkylfuran have been found to give a net ring opening reaction after the initial cycloaddition reaction with the metal carbenoid,[12] far less attention has been given to the study of a 2-substituted heteroatom electron-donating group on furan. In this paper we describe the scope of using metal carbenoid generated from α-diazoester and aryl-α-diazocarbonyl compounds with rhodium(II) acetate for the ring opening reaction of a series of 2-alkoxyfurans. The successful execution of this reaction will represent a new powerful methodology for the direct construction of a remarkable array of 2,4-hexadienedioic acid and 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acid derivatives.

At the onset of this work we were uncertain as to whether the ring opening reaction of furan-2-yloxy derivatives with ethyl diazoacetate and aryl-α-diazocarbonyl compounds in the presence of rhodium(II) catalyst can compete favorably with the cyclopropanation or the insertion reaction (Scheme 1).

2. Results and discussions

Our initial studies focused on the dirhodium(II) tetraacetate catalyzed reaction of 2-methoxyfuran **1a** with ethyl diazoacetate[13] in CH_2Cl_2. In this reaction, the prevalent products obtained were a mixture of the (2*E*,4*Z*)-**2a** and 1-ethyl 6-methyl (2*E*,4*E*)-hexadienedioate **3a** respectively, in a ca. 1:1 ratio (after crude product separation by preparative chromatography on silica gel). From the mechanistic point of view, the stereochemistry of the ring cleavage of 2-methoxyfuran with ethyl diazoacetate under rhodium(II)-catalysis should exclusively gave the (2*E*,4*Z*)-**2a**. This arises from the fact that whereas the newly formed double bond between the 2-methoxyfuran and ethyl diazoacetate α-carbons could assume a *trans* configuration, the double bond created between the former furan β-carbons maintained a *cis* orientation, reflecting the original furan framework.[4,5] The formation of the (*E*,*E*)-**3a** thus implicate an acid induced isomerization of the *cis*-double bond into the more stable *trans*-double bond during silica gel separation of the crude reaction product.[14] The introduction of a 2-methoxy substituent on the furan ring was found to promote the ring opening reaction. Stereochemical

Keywords: diazo compounds; rhodium acetate; furan-2-yloxy derivatives; ring opening reaction.

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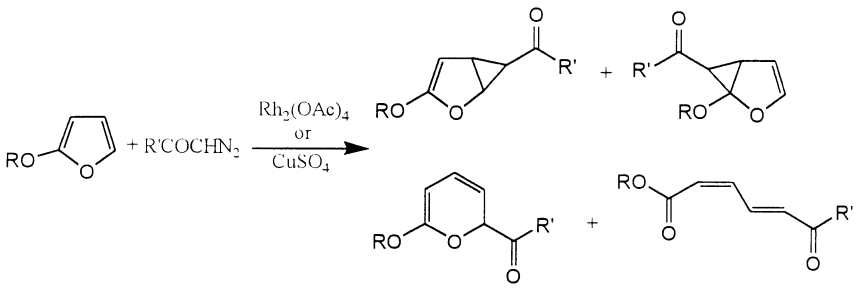


Scheme 1. Possible products from the reaction of furan-2-yloxy derivative with electrophilic metal carbenoid.

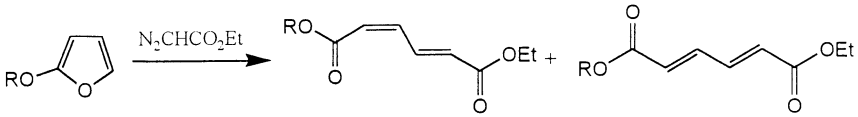
assignments of the products were straightforward upon analyzing of the ^{1}H NMR obtained. The (2*E*,4*Z*)-isomer has a typical downfield chemical shift for the internal *E*-double bond proton at approximately δ 8.40 (dd, J=11 and 15 Hz), similar to that reported for the (*E*,*Z*)-dimethylhexadienedioate.[15] On the other hand, the (*E*,*E*)-isomer internal *Z*-double bond proton has a chemical shift at approximately δ 7.50, similar to that reported for the (*E*,*E*)-dimethylhexadienedioate.[16] We have now added another new repertoire for the preparation of unsymmetrical muconic diester. Several other reported methods for the synthesis of muconic diester include: (i) the oxidative cleavage of catechol[17] to the (*E*,*E*)-isomer and further isomerized into the (*E*,*Z*) form; (ii) the thermolysis of 1,2-cyclobutenedicarboxylic anhydride[18] to the (*E*,*Z*) isomer and (iii) the Wittig[19] or Horner–Emmons[20] reaction with malealdehyde. Each method has its advantages and drawbacks; thus our method provides another alternative route (Scheme 2).

It is reasonable to assume that 6-ethyl 1-trimethylsilyl (2*Z*,4*E*)-hexadienedioate can be prepared from the ring opening reaction of 2-trimethylsilyloxyfuran with ethyl diazoacetate in the presence of rhodium(II)-catalyst. Selective deprotection of the trimethylsily-protecting group should be possible under mild condition to afford monoprotected muconic acid. Monoprotected muconic acid is an important intermediate for the synthesis of macrocyclic trichothecenes.[19–21] Interestingly, the reaction of 2-trimethylsilyloxyfuran **1b** with ethyl diazoacetate in the presence of rhodium(II)-catalyst was found to give directly the (2*E*,4*Z*)-**2b** and (2*E*,4*E*)-**3b** monoprotected muconic acid in a 1:1 ratio (after chromatography of the crude product on silica gel). The trimethylsilyl ester group in the product proved to be extremely labile and undergo selective deprotection to yield the acid. Earlier report for the preparation of monoprotected (*E*,*Z*)-muconic acid generally involved a multi-step sequence via hydrolysis of an ester group in muconic diester that proceed with poor regioselectivity.[18] Our method circumvents the difficulty encounter for the regioselective hydrolysis of an ester group in muconic diester by generating a highly labile trimethylsily-ester precursor that undergoes desilylation in situ.

These results represent the first example using the reactions of furan-2-yloxy derivatives with metal carbenoids for the direct synthesis of unsymmetrical muconic diester and monoprotected muconic acid.

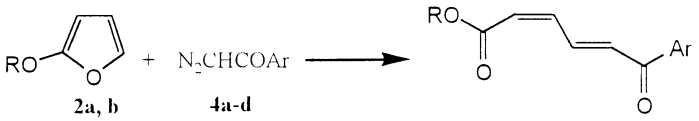
The success achieved by the rhodium(II)-catalyzed ring opening of furan-2-yloxy derivatives with ethyl diazoacetate was extended to a series of aryl diazoketone for the synthesis of 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acid and derivatives. In fact there are two possible routes to 6-aryl-6-oxo-2,4-hexadienoic acid by this method: (i) the reaction of 2-arylfuran with ethyl diazoacetate, and (ii) the reaction of furan-2-yloxy derivatives with aryl-diazoketone in the presence of rhodium(II)-catalyst. The preparation of 2-arylfuran itself is a cumbersome process and this route was deemed unsuitable for the preparation of 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acid and derivatives. The fact that the ring opening reaction of commercially available furan-2-yloxy derivatives with metal carbenoid provides an alternative route for the direct synthesis of 6-aryl-6-oxo-2,4-hexadienoic derivatives is particularly noteworthy. The 6-phenyl-6-oxo-(2*E*,4*E*)-hexadienoate has recently been prepared from phenylglyoxal after two consecutive Wittig reactions with formylmethylenetriphenylphosphorane and (methoxycarbonylmethylene)triphenylphosphorane.[22]

We therefore investigated the reactions of 2-methoxyfuran **1a** with α-diazoacetophenone[23] **4a**, α-diazoacetothiophene[10] **4b**, 4-methoxy-α-diazoacetophenone[23] **4c** and α-diazoaceto-2-naphthone[24] in the presence of rhodium(II)-catalyst. The aryl-α-diazocarbonyl compounds **4a–d** were synthesized from the reaction of the corresponding acid chloride with diazomethane. Treatment of 2-methoxyfuran with α-diazoacetophenone **4a** in the presence of Rh(II)-catalyst afforded the furan ring opening product, methyl 6-oxo-6-phenylhexa-(2*Z*,4*E*)-dienoate **5a**, after purification

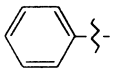
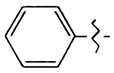
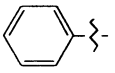
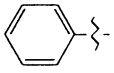
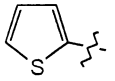
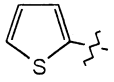
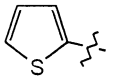
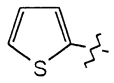
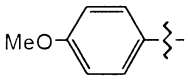
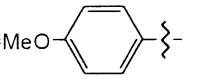
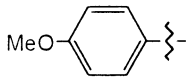
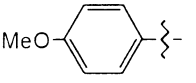
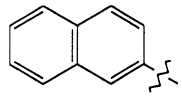
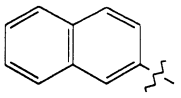
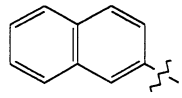
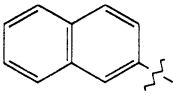


Scheme 2. Synthesis of 2,4-hexadiendioic acid derivatives from 2-methoxy and 2-trimethylsilyloxyfuran and ethyl diazoacetate under Rh(II)-catalyst.

Table 1. Reaction of aryl diazoketone with furan 2-yloxy derivatives



2a, b **4a-d**

1	R=Me	Ar= 	**5a** R=Me	Ar= 	79
	R=TMS	Ar= 	**6a** R=H	Ar= 	79
2	R=Me	Ar= 	**5b** R=Me	Ar= 	53
	R=TMS	Ar= 	**6b** R=H	Ar= 	55
3	R=Me	Ar= 	**5c** R=Me	Ar= 	84
	R=TMS	Ar= 	**6c** R=H	Ar= 	57
4	R=Me	Ar= 	**5d** R=Me	Ar= 	60
	R=TMS	Ar= 	**6d** R=H	Ar= 	55

by chromatography. A point worth noting is that the thermodynamically less stable (*Z*,*E*)-isomer that correspond to an overall retention of configuration of the double bond created between the former furan β-carbons can be obtained without undergoing isomerization during chromatography, and this might be attributed to the increase stability of the *cis*-double bond. By changing the aryl diazocarbonyl compound to α-diazoacetothiophene **4b**, 4-methoxy-α-diazoacetophenone **4c** and α-diazoaceto-2-naphthone **4d**, they were found to react smoothly with 2-methoxyfuran in the presence of rhodium(II) catalysis to give the ring opening product **5b**–**d** respectively, whereby the (*Z*,*E*)-stereochemisty was again retained after purification by chromatography (Table 1). The resistance towards isomerization of the *cis*-double bond in **5a**–**d** is particularly worth noting.

Similarly, 2-trimethylsiloxyfuran **1b** was found to react favorably with all the aryl diazocarbonyl compounds **4a**–**d** in the presence of rhodium(II) catalyst to give the ring opening product, 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acid **6a**–**d** respectively, after purification by silica gel chromatography. The labile trimethylsilyl ester functional group undergoes hydrolysis readily in situ to give the corresponding acid, and the (*Z*,*E*)-double bond was inert towards isomerization during chromatographic purification. We have developed a new and highly efficient direct entry for the synthesis of 6-aryl-6-oxo-(2*Z*,4*E*)-hexadienoic acids and its esters by the ring opening reaction of 2-trimethylsilyloxyfuran and 2-methoxyfuran with a wide array of aryl diazoketones in the presence of rhodium(II) catalyst.

3. Conclusions

In conclusion, this represent the first example of the highly efficiency rhodium(II)-catalyzed ring opening reaction of 2-methoxy- and 2-trimethylsilyloxyfuran with ethyl diazoacetate, α-diazoacetophenone, α-diazoacetothiophene, 4-methoxy-α-diazoacetophenone and α-diazoaceto-2-naphthone for the stereoselective synthesis of 2,4-hexadienedionic acid and 1-aryl-1-oxo-2,4-hexadienoic acid derivatives which will find wide application in organic synthesis. The generality of this transformation is also worth noting.

4. Experimental

^{1}H NMR spectra were recorded using 200 MHz spectrometer in $CDCl_3$, using tetramethylsilane as an internal standard. Melting points were measured on a Fisher–John

apparatus and uncorrected. Unless otherwise noted, all reactions were performed in oven-dried glassware under an atmosphere of nitrogen. Separation of the crude reaction mixture was carried out on preparative silica gel TLC using ethyl acetate–hexane mixture as the eluent. Ethyl diazoacetate,[13] α-diazoacetophenone,[23] α-diazoacetothiophene,[10] 4-methoxy-α-diazoacetophenone[23] and α-diazoaceto-2-naphthone[24] were prepared according to the previously reported methods. Approximately 5 mg of catalyst was used to decompose 0.001 mmol of the diazo compound.

4.1. Rhodium(II)-catalyzed ring opening of 2-methoxy and 2-silyloxyfuran with diazo compounds

To a solution containing diazo compound (0.006 mmol) in dichloromethane (10 mL) was added dropwise to a solution containing 2-methoxyfuran or 2-silyloxyfuran (0.005 mmol) and dirhodium(II) tetraacetate (30 mg) in dichloromethane (15 mL). The reaction mixture was stirred at room temperature over overnight. The reaction mixture was filtered and concentrated under reduced pressure. The resulting crude product was purified on preparative TLC.

4.1.1. 1-Ethyl 6-methyl (2*E*,4*Z*)-2,4-hexadienedioate 2a. Yield: 31.5% (total yield of 63% for *Z*,*E*- and *E*,*E*-isomers) as clear oil. ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.32 (3H, t, *J*=8 Hz), 3.78 (3H, s), 4.25 (2H, q, *J*=8 Hz), 5.98 (1H, d, *J*=11 Hz), 6.12 (1H, d, *J*=16 Hz), 6.66 (1H, dd, both *J*=11 Hz), 8.38 (1H, dd, *J*=11, 16 Hz); MS *m*/*z* 184. (Found: C, 58.83; H, 6.79%. Calcd for $C_9H_{13}O_4$: C, 58.69; H, 6.57%.)

4.1.2. 1-Ethyl 6-methyl (2*E*,*E*)-2,4-hexadienedioate 3a. Yield: 31.5% as clear oil. ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.32 (3H, t, *J*=8 Hz), 3.75 (3H, s), 4.24 (2H, q, *J*=8 Hz), 5.98 (2H, d, *J*=14 Hz), 7.89 (2H, d, *J*=14 Hz).

4.1.3. 5-Ethoxycarbonyl-(2*Z*,4*E*)-2,4-pentadienoic acid 2b. Yield: 20.5% (total yield of 41% for *Z*,*E*- and *E*,*E*-isomers), as colorless solid, mp 89–91°C (lit.[25] 90–92°C). ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.30 (t, 3H, *J*=8.0 Hz), 4.22 (2H, q, *J*=8.0 Hz), 6.07 (1H, d, *J*=11 Hz), 6.20 (1H, d, *J*=15 Hz), 6.73 (1H, *dd*, both *J*=11 Hz), 8.20 (1H, dd, *J*=11, 15 Hz), 11.04 (1H, br. s), MS *m*/*z* 169.

4.1.4. 5-Ethoxycarbonyl-(2*E*,4*E*)-2,4-pentadienoic acid 3b. Yield: 20.5% as colorless solid, mp 143–145°C (lit.[26] 145–146°C). ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.31 (3H, t, *J*=8 Hz), 4.20 (2H, q, *J*=8 Hz), 6.00 (1H, d, *J*=14 Hz), 7.85 (1H, d, *J*=14 Hz), 7.94 (1H, dd, both *J*=14 Hz), 8.01 (1H, dd, both *J*=14 Hz), 11.24 (1H, br. s), MS *m*/*z* 169.

4.1.5. Methyl 6-oxo-6-phenyl-(2*Z*,4*E*)-2,4-hexadienoate 5a. Yield: 79% as yellowish solid, mp 90–92°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.79 (3H, s), 6.05 (1H, d, *J*=11 Hz), 6.78 (1H, dd, both *J*=11 Hz), 7.09 (1H, d, *J*=15 Hz), 7.53 (3H, m), 7.95 (2H, d, *J*=8 Hz), 8.41 (1H, dd, *J*=11, 15 Hz); ^{13}C NMR (500 MHz) δ 190.969, 165.78, 141.16, 138.44, 137.50, 133.11, 133.03, 128.69, 128.67, 125.01, 51.74; MS *m*/*z* 216. (Found: C, 71.94; H, 5.67%. Calcd for $C_{13}H_{12}O_3$: C, 72.21; H, 5.59%.)

4.1.6. Methyl 6-oxo-6-thienyl-(2*Z*,4*E*)-2,4-hexadienoate 5b. Yield: 53% as yellowish solid, mp 95–96°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.80 (3H, s), 6.05 (1H, d, *J*=11 Hz), 6.76 (1H, dd, both *J*=11 Hz), 7.00 (1H, d, *J*=15 Hz), 7.10 (1H, dd, *J*=5, 6 Hz), 7.70 (1H, d, *J*=6 Hz), 7.81 (1H, d, *J*=5 Hz), 8.48 (1H, dd, *J*=11, 15 Hz); ^{13}C NMR (500 MHz) δ 182.28, 165.75, 164.85, 140.87, 137.63, 134.35, 132.58, 132.38, 128.36, 125.16, 51.74; MS *m*/*z* 222. (Found: C, 59.36; H, 4.52%. Calcd for $C_{11}H_{10}SO_3$: C, 59.44; H, 4.53%.)

4.1.7. Methyl 6-oxo-6-(4-methoxyphenyl)-(2*Z*,4*E*)-2,4-hexadienoate 5c. Yield: 84% as yellowish solid, mp 125–126°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.79 (3H, s), 3.89 (3H, s), 6.03 (1H, d, *J*=11 Hz), 6.77 (1H, dd, both *J*=11 Hz), 6.97 (2H, d, *J*=8 Hz), 7.10 (1H, d, *J*=15 Hz), 7.97 (2H, d, *J*=8 Hz), 8.40 (1H, dd, *J*=11, 15 Hz); ^{13}C NMR (500 MHz) δ 189.10, 165.86, 163,67, 141.34, 137.61, 133.07, 130.03, 130.46, 124.55, 113.93, 55.52, 51.71; MS *m*/*z* 246. (Found: C, 68.19; H, 5.54%. Calcd for $C_{14}H_{14}O_4$: C, 68.28; H, 5.73%.)

4.1.8. Methyl 6-oxo-6-(2-naphthyl)-(2*Z*,4*E*)-2,4-hexadienoate 5d. Yield: 60% as yellowish solid, mp 111–112°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.79 (3H, s), 6.08 (1H, d, *J*=11 Hz), 6.84 (1H, dd, both *J*=11 Hz), 6.90(1H, d, *J*=15 Hz), 7.60 (3H, m) 7.90 (4H, m), 8.33 (1 dd, *J*=11, 15 Hz); ^{13}C NMR (500 MHz) 190.53, 165.79, 141.17, 138.33, 135.59, 134.85, 132.93, 132.49, 130.34, 129.58, 128.68, 128.59, 127.85, 126.87, 125.01, 124.37, 51.73; MS *m*/*z* 266. (Found: C, 77.01; H, 5.14%. Calcd for $C_{17}H_{14}O_4$: C, 76.68; H, 5.30%.)

4.1.9. 6-Oxo-6-phenyl-(2*Z*,4*E*)-2,4-hexadienoic acid 6a. Yield: 79% as yellowish solid, mp 82–83°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.39 (1H, *J*=11 Hz), 6.61 (1H, dd, both *J*=11 Hz), 7.07 (1H, d, *J*=15 Hz), 7.55 (3H, m), 7.93 (2H, d, *J*=9 Hz), 8.25 (1H, dd, *J*=11, 15 Hz), 11.40 (1H, br. s); MS *m*/*z* 201. (Found: 200.0837. HRMS calcd for $C_{12}H_{10}O_3$: 200.0841.)

4.1.10. 6-Oxo-6-thienyl-(2*Z*,4*E*)-2,4-hexadienoic acid 6b. Yield: 55% as yellowish solid, mp 102–103°C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.08 (1H, d, *J*=11 Hz), 6.86 (1H, dd, both *J*=11 Hz), 7.05 (1H, d, *J*=15 Hz), 7.20 (1H, dd, *J*=5, 6 Hz), 7.76 (1H, d, *J*=6 Hz), 7.98 (1H, d, *J*=5 Hz), 8.50 (1H, dd, *J*=11, 15 Hz), 11.30 (1H, br. s); MS *m*/*z* 208. (Found: 208.0194. HRMS calcd for $C_{10}H_8O_3S$: 208.0194.)

4.1.11. 6-Oxo-6-(4-methoxyphenyl)-(2*Z*,4*E*)-2,4-hexadienoic acid 6c. Yield: 57% as yellowish solid, mp 126–127°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.89 (3H, s), 6.05 (1H, d, *J*=11 Hz), 6.84 (dd, 1H, both *J*=11 Hz), 6.90 (2H, d, *J*=9 Hz), 7.15 (1H, d, *J*=15 Hz), 7.92 (1H, d, *J*=9 Hz), 8.42 (1H, dd, *J*=11, 15 Hz), 11.34 (1H, br. s); MS *m*/*z* 232. Found: 232.0735. HRMS calcd for $C_{13}H_{12}O_4$: 2320745.)

4.1.12. 6-Oxo-6-(2-naphthyl)-(2*Z*,4*E*)-2,4-hexadienoic acid 6d. Yield: 55% as yellowish solid, mp 87–88°C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.07 (1H, d, *J*=11 Hz), 6.78 (1H, dd, both *J*=11 Hz), 7.13 (1H, d, *J*=15 Hz), 7.65 (3H, m), 7.90 (4H, m), 8.43 (1H, dd, *J*=11, 15 Hz), 11.64 (1H, br. s); MS

m/*z* 252. Found: 252.0786. HRMS calcd for $C_{16}H_{12}O_3$: 252.0787.

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