Pergamon

SCIENCE DIRECT®

Tetrahedron 59 (2003) 5027–5031

TETRAHEDRON

Stereoselective synthesis of *Z*-3-alkoxy-2-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranones

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Received 5 February 2003; revised 11 April 2003; accepted 8 May 2003

Abstract—Photoreorganisation of 2-alkoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-diones in anhydrous acetone affords exclusively *Z*-3-alkoxy-3-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranones. The products were identified by X-ray crystallography of one of them. © 2003 Elsevier Science Ltd. All rights reserved.

1. Introduction

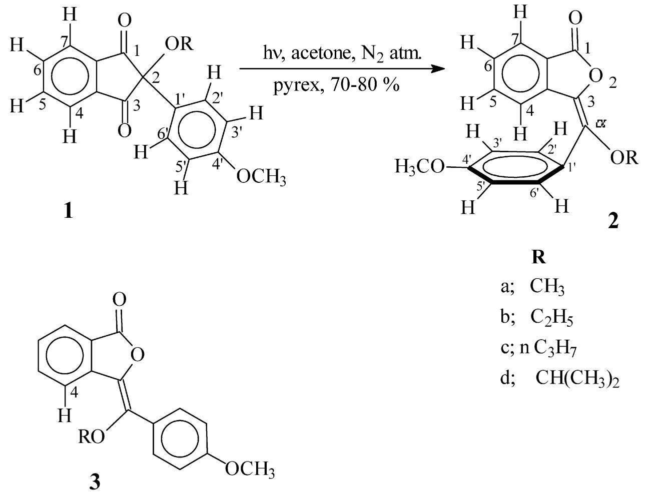
In view of the wide ranging[1–3] biological activities associated with both natural and synthetic 3-ylidene-1(3*H*)-isobenzofuranones and their use as valuable intermediates[4–8] for the syntheses of a variety of heterocyclic and carbocyclic ring systems, a number of methods for their syntheses[9–20] have been reported in the past. All these methods either employ multi-step sequences involving rather harsh conditions or lack in stereoselectivity. Here, we report a new stereoselective photochemical synthesis of some *Z*-3-alkoxy-3-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranones **2** through photoreorganisation of the corresponding 2-alkoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-diones **1**.

2. Results and discussion

2-Alkoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-diones **1** were prepared by refluxing 2-bromo-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-dione[22] in the appropriate alcohol for 1.5–2.0 h. Photoirradiation of an acetone solution of the diones **1** with 450 W Hanovia lamp through a pyrex filter under a N_2 atmosphere followed by chromatographic work up of the resulting photolysate afforded **2** in excellent yields (70–80%). The structures of isobenzofuranones **2** and their precursors **1** were found to be consistent with their elemental analysis and spectral parameters (vide experimental) (Scheme 1).

A priori the photolysis of each 1*H*-indene-1,3(2*H*)-dione **1** can, in principle, furnish two stereoisomeric isobenzofuranones **2** (*Z*) and **3** (*E*), respectively, but in all the four cases studied during the present investigations, the exclusive formation of the *Z*-isobenzofuranones **2** was observed.

Our preference for structure **2** over **3** rests upon the literature reports[17,21] that in 3-alkylidene-1(3*H*)-isobenzofuranones, the four aromatic protons usually appear in the region δ



Scheme 1.

Keywords: stereoselective; photoreorganisation.

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doi:10.1016/S0040-4020(03)00757-9

7.20–8.00. But in isobenzofuranones **2**, C_4–H appeared in the region δ 6.37–6.65.[21] This upfield shift of C_4–H relative to C_5–H, C_6–H and C_7–H is presumably due to its near orthogonal disposition with respect to the 4′-methoxyphenyl group. This observation was confirmed by X-ray structural analysis of **2a** (R=CH_3), which clearly shows that 4′-methoxyphenyl group is held almost perpendicular to the C_4–H of the benzene ring of the isobenzofuranone moiety (Fig. 1).

2.1. Crystal structure of 2a

The single crystal of **2a** was obtained as pale yellow needles by recrystallisation from methanol. The X-ray molecular structure of the compound along with the atomic numbering is given in ORTEP drawing Figure 1 for non-H-atoms.

The C–C bond distances in the 4′-methoxyphenyl ring and the benzene ring of the isobenzofuranone moiety vary from 1.365(6) to 1.400(5) Å; some of the atoms have relatively large thermal parameters. The internal angles of the benzene ring shows variation with the minimum value of 117.4° for C(12)–C(11)–C(16) and the maximum value of 122.5(4)° for C(6)–C(1)–C(2). However, the average value of 120.0° of the inter-bond angles in the two rings is the ideal one required for a planar conformation. The O(1), C(9) and C(11) atoms all lie essentially in the plane of the isobenzofuranone moiety, and C(8) and C(9) atoms deviate 0.041(4) and 0.030(5) Å from the least squares plane defined by all the nine atoms of the isobenzofuranone nucleus. The methoxy C(10) atom faces C(11) atom of the 4′-methoxyphenyl ring but is not coplanar with the atoms O(3), C(9) and C(11) [torsion angle C(10)–O(3)–C(9)–C(11), −10.1(6)°]. The methoxy C(17) atom faces C(14) atom of the 4′-methoxyphenyl ring and the C(17) and O(4)atoms lie in the plane of the phenyl ring attached to O(4) atom) [torsion angle C(17)–O(4)–C(14)–C(13), 0.7(7)°].

The bond distance C(9)–O(3), 1.349(5) Å, is considerably shorter than the sum of the covalent radii of C(sp^2) and O[25] (1.468 Å) but is similar to the value for compounds containing C–O bond where charge delocalization occurs. Participation of the O(3) atom lone pairs in the conjugation system is confirmed by the approximately planar configuration of the atoms C(6), C(8), O(2), C(9), O(3) and C(11) [the deviations of the atoms from the r.m.s. plane do not exceed to 0.0036(4) Å and the sum of the angles around the C(9) atoms is exactly 360°] and the essential co planarity of this plane with the isobenzofuranone moiety (dihedral angle 4.8°). The bond length C(10)–O(3), 1.402(5) Å is, however, considerably larger than that of the C(9)–O(3) bond length, 1.349(5) Å, probably due to the sp^3 character of the C(10) atom. The exocyclic C(9)–C(11) bond length, 1.487(5) Å is shorter than a typical C–C single bond of 1.542 Å. The shorter C(7)–O(2) bond length of 1.384(5) Å as compared with the bond length C(8)–O(2) of 1.420 Å implies substantial double bonding due to the participation of non-bonding electrons on the O(2) atom and is consequently responsible for the elongation of the formal C(7)–O(1) double bond to 1.207(5) Å by ~3σ which testifies to participation of the O(1) atom in the conjugation system [the standard bond length of a non-conjugated C(sp^2)=O bond is 1.191 Å].

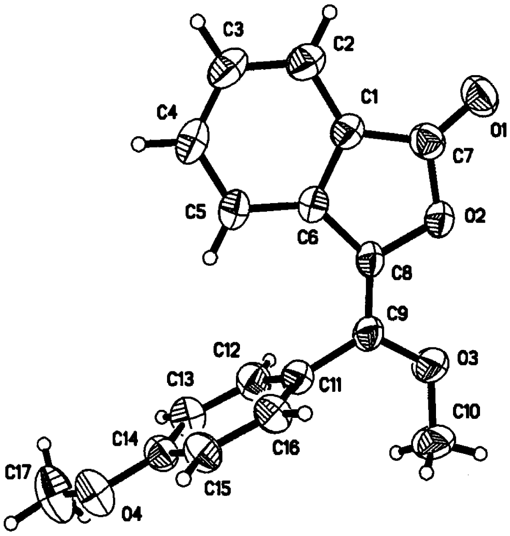


Figure 1. ORTEP drawing and crystallographic numbering scheme of **2a** with probability thermal ellipsoids for non-H-atoms. H-atoms are represented by spheres of arbitrary size.

The plane of the phenyl ring bonded to C(9) atom is almost perpendicular to the C(8)–C(9) double bond and this orientation is described by torsion angles C(8)–C(9)–C(11)–C(16), 83.7(5)° and C(8)–C(9)–C(11)–C(12), −93.8(5)°. The isobenzofuranone plane and the 4′-methoxyphenyl ring enclose a dihedral angle of 90° suggesting the π-electron delocalization is not extended to isobenzofuranone moiety indicated by the normal value of C(8)–C(9) double bond, 1.335(5) Å [the standard length for C(sp^2)=C(sp^2) bond is 1.331 Å] and the shortening of the C(9)–C(11) bond length may be attributed to the inductive effect caused by the release of electrons along the C(11)–C(9) bond by the OCH_3 group of the phenyl ring. Unusual shortening of the angle C(12)–C(11)–C(16), 117.7(4)° in the 4′-methoxyphenyl ring in comparison with the value ~120° in the aromatic compounds without any substituent at C(14) position points to same phenomenon.

3. Attempted photoisomerisation of the *Z*-isobenzofuranone 2 to *E*-isobenzofuranone 3

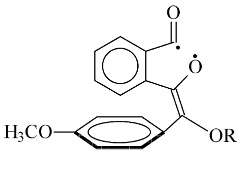
3.1. General procedure

A deoxygenated solution of **2a** (0.5 g) in dry acetone (70 cm^3) was irradiated in a pyrex vessel with light from 450 W Hanovia lamp. TLC on aliquots withdrawn from the reaction mixture at regular intervals of time did not show the formation of any photoproduct even after 10.0 h of irradiation. Solvent was removed under reduced pressure and resulting photolysate was crystallised using methanol to yield **2a**, which was characterised through TLC, superimposable IR and NMR spectra and undepressed mmp with an authentic sample. The solution of other three *Z*-isobenzofuranones **2b–d** in dry acetone were separately irradiated, under conditions similar to those employed for **2a** for 10 h. The formation of the corresponding

E-isobenzofuranones **3b–d** was not observed and *Z*-isobenzofuranones **2b–d** were recovered unchanged.

3.2. Mechanism

Mechanistically, the phototransformation **1**→**2** may be visualised to have occurred through an initial[19,20] Norrish type-I reaction followed by reorganisation to afford biradical **4** which subsequently recloses to furnish isobenzofuranones **2**.



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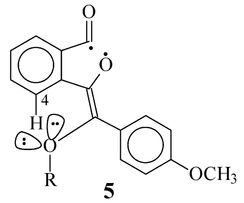

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The alternate geometry **5** for the biradical intermediate which could lead to the formation of *E*-isobenzofuranone **3** is ruled out on the basis of severe steric congestion expected to operate between the lone pairs of electrons on the oxygen atom of the alkoxy group and C_4–H. Such a situation does not occur in biradical **4** where the 4′-methoxyphenyl group, being planar, can easily assume an orthogonal orientation with respect to C_4–H thereby experiencing no steric strain.

4. Conclusion

From the above discussion we conclude that the incorporation of alkoxy substituents at C_2 of the 1*H*-indene-1,3(2*H*)-dione moiety confers stereoselectivity to the phototransformation of 1*H*-indene-1,3(2*H*)-diones **1a–d**, thereby leading to the exclusive formation of the corresponding *Z*-isobenzofuranones **2a–d**.

5. Experimental

Mps were determined in open capillaries using a sulfuric acid bath and are uncorrected. IR spectra were recorded in Nujol mulls on a Perkin–Elmer 842 spectrophotometer and ^{1}H NMR spectra were recorded on 90 MHz Perkin–Elmer R-32 instrument in $CDCl_3$ using Me_4Si as internal standard. Mass spectra were recorded at 70 eV using KRATOS MS-50 instrument and X-ray data were collected at room temperature on Siemens P4 diffractometer using graphite monochromator with Mo K_α radiations.

5.1. General method for the preparation of 2-alkoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-diones 1

2-Bromo-2-(4′-methoxyphenyl)-1*H*-indene-1,3-(2*H*)-dione (1.0 g, 9.06 mmol) was refluxed in an appropriate alkanol (50 cm^3) for 1.5–2.0 h. Thereafter, the solvent was removed under vacuum and the residue thus obtained was either crystallised or purified using column chromatography.

5.1.1. 2-Methoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-dione 1a. 1.5 h, methanol (0.8 g, 94%), light yellow crystalline solid, mp 108–109°C. (Found: C, 72.43; H, 4.87; $C_{17}H_{14}O_4$ requires C, 72.34; H, 4.96%); λ_{max} (MeOH)/nm 228.5 (ε_m 63845) and 355.0 (ε_m 790) nm; ν_{max}/cm^{-1} 1745 and 1700 (s, asym. and sym., C=O str of 1*H*-indene-1,3(2*H*)-dione); δ_H 3.45 (s, 3H, C_2–OCH_3), 3.75 (s, 3H, $C_{4'}$–OCH_3), 6.80 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.40 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.80–8.10 (m, 4H, C_4–*H*, C_5–*H*, C_6–*H*, and C_7–*H*).

5.1.2. 2-Ethoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-dione 1b. 1.0 h, ethanol (0.7 g, 80%), yellow crystalline solid, mp 97–98°C. (Found: C, 72.89; H, 5.43; $C_{18}H_{16}O_4$ requires C, 72.97; H, 5.41%); λ_{max} (MeOH)/nm 228.5 (ε_m 67381) and 381.0 (ε_m 1904); ν_{max}/cm^{-1} 1740 and 1708; δ_H 1.17 (t, 3H, J=7 Hz, C_2–OCH_2CH_3), 3.55 (q, 2H, J=7 Hz, C_2–OCH_2CH_3), 3.67 (s, 3H, $C_{4'}$–OCH_3), 6.76 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.33 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.70–8.05 (m, 4H, C_4–*H*, C_5–*H*, C_6–*H*, and C_7–*H*).

5.1.3. 2-*n*Propoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-dione 1c. 1.0 h, purified through column chromatography (pet.ether–benzene, 1:1), light yellow thick oil (0.65 g, 70%). (Found: C, 73.61; H, 5.90; $C_{19}H_{18}O_4$ requires C, 73.55; H, 5.80%); λ_{max} (MeOH)/nm 228.5 (ε_m 68200) and 391.0 (ε_m 372); ν_{max}/cm^{-1} 1750 and 1705; δ_H 0.79 (t, 3H, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 1.47 (m, 2H, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 3.35 (t, 2H, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 3.58 (s, 3H, $C_{4'}$–OCH_3), 6.70 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.3 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.60–7.94 (m, 4H, C_4–*H*, C_5–*H*, C_6–*H* and C_7–*H*).

5.1.4. 2-Isopropoxy-2-(4′-methoxyphenyl)-1*H*-indene-1,3(2*H*)-dione 1d. 2.0 h, benzene–hexane (0.61 g, 65%), yellow crystalline solid, mp 90–92°C. (Found: C, 73.75; H, 5.95; $C_{19}H_{18}O_4$ requires C, 73.55; H, 5.81%); λ_{max} (MeOH)/nm 228.5 (ε_m 67828) and 361.5 (ε_m 6448); ν_{max}/cm^{-1} 1755 and 1720; δ_H 1.00–1.10 (2s, 6H, C_2–$OCH(CH_3)_2$), 3.60 (s, 3H, $C_{4'}$–OCH_3), 3.75 (m, 1H, J=7 Hz, C_2–$OCH(CH_3)_2$), 6.70 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–H), 7.30 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.65–7.95 (m, 4H, C_4–*H*, C_5–*H*, C_6–*H* and C_7–*H*).

5.2. Photolysis: general procedure

A deoxygenated solution of **1** (0.5 g) in dry acetone (80 cm^3) was irradiated in a pyrex vessel with light from 450 W Hanovia lamp for 3.0–5.0 h. Solvent was removed and the resulting photolysate was either crystallised or chromatographed over a column of silica gel to afford the corresponding *Z*-3-alkoxy-3-[(4′-methoxyphenyl)methylidene]-1(3*H*) isobenzofuranones **2**.

5.2.1. *Z*-3-Methoxy-2-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranone 2a. 3.0 h, methanol (0.35 g, 70%), light yellow crystalline solid, mp 139–141°C. (Found: C, 72.45; H, 5.126; $C_{17}H_{14}O_4$ requires C, 72.34; H, 4.96%); ν_{max}/cm^{-1} 1750 (s, C=O str. five-membered lactone) and 1670 (m, C=C str.), δ_H 3.63 (s, 3H, >C=C–OCH_3), 3.84 (s, 3H, C_4–OCH_3), 6.37–6.52 (m, 1H, C_4–*H*), 6.96 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.16–7.26 (m, C_5–*H* and C_6–*H*), 7.36 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.69–7.84 (m, 1H, C_7–*H*); *m*/*z* 282 (M^+, 100%), 239 (77),195 (7) and 76 (4.4).

Crystal structure determination of **2a**.

Crystal data. $C_{17}H_{14}O_4$, M=282.28, orthorhombic, a=18.2890(10), b=8.0690(10), c=19.3380(10) Å, V=853.8(4) Å^3 (by least squares refinement on diffractometer angles for 25 automatically centred high angle reflections, λ=0.71073 Å^3, space group *Pbca*, Z=8, $F(000)$=1184, D_x=1.314 mg cm^{-3}, μ(Mo K_α)=0.74 cm^{-1}, $T\sim$293 K.

Data collection and processing. A crystal of size 0.2×0.1×0.1 mm^3 was mounted on Siemens P4 diffractometer operating in the θ–2θ mode with variable scan speed. 1873 independent reflections were measured using graphite monochromated Mo K_α radiation for $2<\theta<22.5°$ (*hkl* range: $0\leq h\leq 19$, $0\leq k\leq 8$, $-20\leq l\leq 0$) out of which 1706 reflections were unique. The data were corrected for Lorentz and polarization effects. Consideration of the crystal shape and transmission factor enabled us to neglect absorption.

Structure analysis and refinement. The non-hydrogen atoms were located by routine methods using the program system SHELXTL PC with atomic scattering factor incorporated in the program from International Tables for X-ray crystallography and refined anisotropically by full matrix least squares based on F^2 on IBM PC. The H-atoms were located on the basis of geometrical considerations and during refinement floated on the respective non-hydrogen atoms. The 200 reflection was very intense but outside the range of instrument and was suppressed during the refinement. The weighting scheme $W=1/[\sigma^2(F_0^2)+(0.2476P)^2+1.4388P]$ where $P=(F_0^2+2F_c^2)/3$ with $\sigma(F_0)$ from counting statistics gave satisfactory agreement analysis. Final R and wR values for 190 parameters using 1706 reflections [$I>2\sigma(I)$], were 0.0468 and 0.1651, respectively and goodness of fit on F^2 was S=0.728. A final Fourier difference map was featureless, $\Delta\rho_{max}$=0.234 and $\Delta\rho_{min}$=−0.199 e Å^{-3}.

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC.207959. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK [fax: +44(0)-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk].

5.2.2. *Z*-3-Ethoxy-2-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranone 2b. 5.0 h, isolated through column chromatography (pet. ether–benzene, 1:1) (0.42 g, 84%), white crystalline solid, mp 143–144°C. (Found: C, 72.90; H, 5.51; $C_{18}H_{16}O_4$ requires C, 72.97; H, 5.40%); ν_{max}/cm^{-1} 1765 and 1655; δ_H 1.27 (t, 3H, J=7 Hz, C_2–OCH_2CH_3), 3.82 (s, 3H, $C_{4'}$–OCH_3), 3.90 (q, 2H, J=7 Hz, C_2–OCH_2CH_3), 6.41–6.56 (m, 1H, C_4–*H*), 6.97 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.17–7.27 (m, 2H, C_5–*H* and C_6–*H*), 7.38 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.68–7.83 (m, 1H, C_7–*H*); *m*/*z* 296 (M^+, 94%), 268 (31), 240 (38), 239 (100), 135 (54), 104 (15) and 76 (16).

5.2.3. *Z*-3-*n*Propoxy-2-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranone 2c. 3.5 h, isolated through column chromatography (pet. ether–benzene, 1:1) (0.33 g, 65%), white crystalline solid, mp 79–80°C. (Found: C, 73.68; H, 5.92; $C_{19}H_{18}O_4$ requires C, 73.55; H, 5.80%); ν_{max}/cm^{-1} 1760 and 1660; δ_H 0.95 (t, 2H, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 1.75 (m, 2H, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 3.80 (t, J=7 Hz, C_2–$OCH_2CH_2CH_3$), 3.85 (s, 3H, $C_{4'}$–OCH_3), 6.50–6.65 (m, 1H, C_4–*H*), 7.05 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.20–7.35 (m, 2H, C_5–*H* and C_6–*H*), 7.40 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*), 7.75–7.90 (m, 1H, C_7–*H*); *m*/*z* 310 (M^+, 77%), 268 (100), 240 (32), 239 (29), 135 (45), 104 (14) and 76 (10).

5.2.4. *Z*-3-Isopropoxy-2-[(4′-methoxyphenyl)methylidene]-1(3*H*)-isobenzofuranone 2d. 3.0 h, isolated through column chromatography (pet. ether–benzene,1:1) (0.27 g, 54%), light yellow crystalline solid, mp 162–163°C. (Found: C, 73.63; H, 5.92; $C_{19}H_{18}O_4$ requires C, 73.55; H, 5.80%); ν_{max}/cm^{-1} 1760 and 1670; δ_H 1.07–1.30 (2s, 6H, C_2–$OCH(CH_3)_2$), 3.81 (s, 3H, $C_{4'}$–OCH_3), 3.95–4.30 (m, 1H, C_2–$OCH(CH_3)_2$), 6.43–6.64 (m, 1H, C_4–*H*), 6.90 (d, 2H, J=8 Hz, $C_{3'}$–*H* and $C_{5'}$–*H*), 7.40 (d, 2H, J=8 Hz, C_5–*H* and C_6–*H*), 7.35–7.45 (d, 2H, J=8 Hz, $C_{2'}$–*H* and $C_{6'}$–*H*),7.65–7.75 (m, 1H, C_7–*H*);. *m*/*z* 310 (M^+, 17%), 268 (100), 240 (29), 239 (15), 132 (8), 104 (11) and 76 (5).

Acknowledgements

Mona Kapoor thanks UGC and Satbir Mor thanks CSIR, New Delhi for generous financial grants.

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