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Synthesis and Crystallography of 1-acetoxy-3-ethoxy carbonyl-4-(4-methoxy)phenyl-7-methoxynaphthalene

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Abstract

*1-Acetoxy-3-ethoxy carbonyl-4-(4-methoxy)phenyl-7-methoxynaphthalene (**3**) has been synthesized and characterized by X-ray diffraction method . The compound crystallizes in triclinic space group P-1 with cell parameter a = 7.793 (6) Å, b = 10.7428(16) Å, c = 13.306 (4) Å and Z = 2.*

Keywords: *Phenyl naphthalene, aryl naphthalene, ligran, Benzophenone, diethylsuccinate*

Introduction

Several aryl-1,2-dihydronaphthalene[1] and aryl naphthalenes which are derivatives of lignans exhibit inhibitory activity against viral reverse transcriptase, such as retrojusticidin and phyllamyricin[2] . Also they exhibit hypolipidemic activity[3]. Kamal *et al.*[4] have carried out the preparation of dehydropodophyllotox in an important representative of arylnaphthalene lignans, in the presence of yeast. Owing to their interest as antineoplastic agents[5] and other pharmacological activities,[6] considerable work has been completed on lignans and other derivatives. In view of these significances in this article we report the synthesis of some naphthalene analogues. In retrospect of the above observations, the presentation is directed towards facile synthesis and x-ray crystallographic studies of [1-acetoxy-3-ethoxy carbonyl-4-(4-methoxy)phenyl-7-methoxynaphthalene.

Experimental

In a typical procedure, to freshly prepared potassium tertiary butoxide (1.56 g of potassium in 60 mL of t-butanol) 4,4'-dimethoxybenzophenone **1** was added quickly under nitrogen and refluxed for 1h. To this mixture freshly distilled diethylsuccinate (6.6mL, 0.04mol) was added at once and refluxed for 30 h. The excess of t-butanol was removed by distillation under reduced pressure and the residue was acidified with 5N HCl. The product was extracted into 10% $NaHCO_3$ solution and washed with diethyl ether (3×20 mL). Finally on acidification with 10% HCl, it gave isomers of **2** as yellow pasty mass. **2**.

In a typical procedure, a mixture of **2** (5 g, 0.01mol), fused sodium acetate (1.38 g ,0.016 mol) and acetic anhydride (41.4 mL) was stirred over night at room temperature. It was refluxed at 80 ^oC for 5 h. The crude product was extracted in ether (3×20 mL) and washed with distilled water (3×30 mL) to remove excess of sodium acetate and acetic anhydride. Finally it was washed with 10% $NaHCO_3$ 10% NaOH and distilled water and dried over sodium sulphate. After evaporation the product was recrystallized with ethanol. gave 1-acetoxy-3-ethoxy carbonyl-4-(4-methoxy)phenyl-7-methoxynaphthalene **(3)** in % (g) yield. M.P 95-96 °C.

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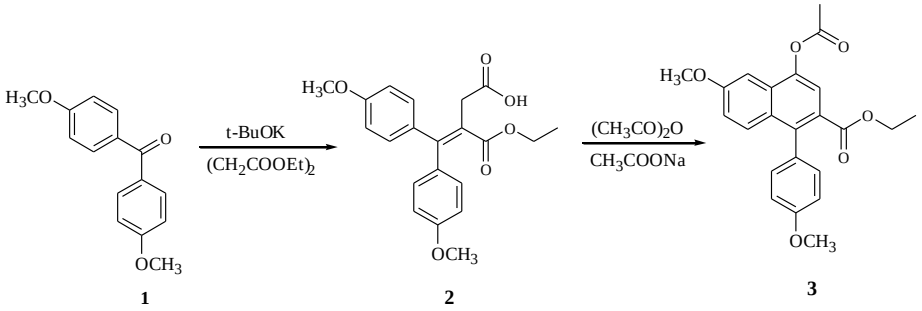
Results and Discussion

IR (Neat) of yellow pasty mass. 2 reveals the following information: 1660 (C=O), 1712 (C=O of ester), 1600 (C=O of acid), 3100-3200 cm^{-1} (OH of carboxylic acid); ^1H NMR ($CDCl_3$): δ 1.02 (t, 3H, CH_3), 1.9-2.3 (m,2H,CH_2) 0.9 (t, J= 6Hz, 3H, CH_3), 3.3 (bs,2H, CH_2-C=O), 3.75 (s, 6H, 2OCH_3) 3.9 (q, 2H, OCH_2), 6.8-7.5 (m, 9H, Ar-H), 10.5 (bs,1H, COOH).

IR(Nujol) 1-acetoxy-3-ethoxy carbonyl-4-(4-methoxy)phenyl-7-methoxynaphthalene **(3)** reveals the following information: 1660 (C=O) , 1765 cm^{-1}(C=O of ester); ^1H NMR ($CDCl_3$): δ 0.9-1.1 (t, 3H, CH_3 of ethyl), 2.5 (s, 3H, CH_3), 3.8 (s, 6H, 2 OCH_3), 3.9-4.15(q, 2H, CH_2), 6.8-7.7 (m ,8H, Ar-H).

A view of (**3**), showing the atom-numbering scheme, is given in Fig. 1 and selected geometric parameters are given in Table 1.



1 **2** **3**

Scheme 1

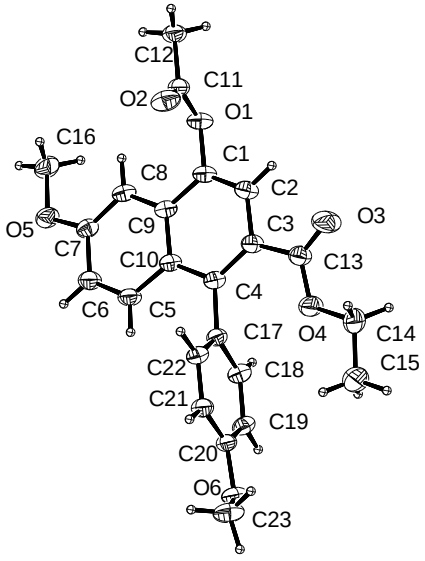


Figure 1**: Triclinic Crystal of compound **3

Table 1: *Geometric parameters*

Formula	$C_{23}H_{22}O_6$
Formula weight	394.41
Crystal system	triclinic
Space group	P_-1, Z = 2
a	7.793 (6) Å
b	10.7428(16) Å
c	13.306 (4) Å
α	71.097 (18)
β	74.94 (3)^0
ν	88.43 (3)Å^3
D_x	1.290 Mg/m^3
$2\theta_{max}$	49.44^0 with MoKa
No. of reflections used (I>2σ(I)	2422
R	0.0447
$(\Delta/\sigma)_{max}$	0.000
$(\Delta\rho)_{max}$	0.996 e Å
$(\Delta\rho)_{min}$	-0.189 e Å
Measurement	Enraf-Nonius MACH3 diffractometer
Program system	SHELXS-97
Refinement fullmatrix:	SHELXS-97

Table 2: *Atomic coordinates and equivalent temperature factors (Å^2)*

Atom	x	y	z	U_{eq}
O1	0.41070(19)	0.88485(12)	0.22273(13)	0.0633(4)
O2	0.2812(2)	0.97064(15)	0.08538(14)	0.0792(5)
O3	-0.1818(2)	0.64003(17)	0.39058(19)	0.1019(7)
O4	-0.13711(19)	0.43258(14)	0.40573(14)	0.0746(5)
O5	0.93619(18)	0.65860(14)	0.05142(13)	0.0667(4)
O6	-0.0388(2)	-0.01032(12)	0.33408(13)	0.0688(4)
C1	0.3457(3)	0.75738(18)	0.23785(18)	0.0559(5)
C2	0.1755(3)	0.71636(19)	0.29434(19)	0.0607(5)
C3	0.1089(3)	0.58774(18)	0.31207(17)	0.0556(5)
C4	0.2189(3)	0.50278(18)	0.27015(16)	0.0520(5)
C5	0.5197(3)	0.46689(19)	0.16249(18)	0.0603(6)
C6	0.6918(3)	0.5081(2)	0.10970(19)	0.0632(6)
C7	0.7585(3)	0.63211(19)	0.10108(17)	0.0561(5)
C8	0.6472(3)	0.71547(18)	0.14130(17)	0.0570(5)
C9	0.4668(3)	0.67510(18)	0.19560(17)	0.0527(5)
C10	0.4001(3)	0.54687(17)	0.21029(17)	0.0526(5)
C11	0.3708(3)	0.98600(19)	0.14087(18)	0.0559(5)
C12	0.4545(3)	1.11272(19)	0.1320(2)	0.0662(6)
C13	-0.0836(3)	0.5572(2)	0.37303(19)	0.0611(5)
C14	-0.3256(3)	0.3980(2)	0.4579(2)	0.0756(7)
C15	-0.3574(4)	0.2558(3)	0.4775(3)	0.0986(9)
C16	1.0147(3)	0.7770(2)	0.0510(2)	0.0772(7)
C17	0.1573(2)	0.36706(17)	0.28193(16)	0.0499(5)
C18	0.1861(3)	0.25812(19)	0.36551(17)	0.0573(5)
C19	0.1224(3)	0.13414(19)	0.37892(18)	0.0578(5)
C20	0.0255(3)	0.11647(17)	0.31100(17)	0.0524(5)
C21	0.0002(3)	0.22260(18)	0.22618(17)	0.0568(5)
C22	0.0679(3)	0.34659(18)	0.21198(17)	0.0551(5).
C23	-0.1701(4)	-0.0284(2)	0.2831(2)	0.0863(8)

$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij}(a_i^* a_j^*)(a_i a_j)$

Table 3 *Selected bond lengths (Å) and bond angles (°)*

O1-C11	1.362(3)	C11-O1-C1	117.07(16)
O1-C1	1.407(2)	C13-O4-C14	117.38(18)
O2-C11	1.189(3)	C7-O5-C16	117.15(18)
O3-C13	1.198(3)	C20-O6-C23	117.48(17)
O4-C13	1.310(2)	C2-C1-O1	120.12(18)
O4-C14	1.453(3)	C2-C1-C9	122.48(18)
O5-C7	1.364(3)	O1-C1-C9	117.36(18)
O5-C16	1.424(2)	C1-C2-C3	120.9(2)
O6-C20	1.374(2)	C4-C3-C2	119.81(19)
O6-C23	1.415(3)	C4-C3-C13	125.12(18)
C1-C9	1.410(3)	C2-C3-C13	115.03(18)
C2-C3	1.414(3)	C3-C4-C10	119.07(17)
C3-C4	1.385(3)	C3-C4-C17	123.16(18)
C3-C13	1.497(3)	C10-C4-C17	117.76(17)
C4-C10	1.435(3)	O5-C7-C8	125.18(18)
C4-C17	1.494(3)	O5-C7-C6	114.86(19)
C6-C7	1.402(3)	C8-C7-C6	119.96(19)
C9-C10	1.421(3)	C1-C9-C8	122.62(18)
C11-C12	1.483(3)	C1-C9-C10	117.11(19)
C14-C15	1.480(3)	C5-C10-C4	122.44(17)
C17-C22	1.370(3)	C9-C10-C4	120.60(18)
C19-C20	1.377(3)	O2-C11-O1	122.86(18)
C20-C21	1.372(3)	O2-C11-C12	126.4(2)
		O1-C11-C12	110.74(19)
		O3-C13-O4	122.4(2)
		O3-C13-C3	122.8(2)
		O4-C13-C3	114.76(18)
		O4-C14-C15	107.8(2)
		C22-C17-C18	118.04(17)
		C22-C17-C4	121.05(17)
		C21-C20-O6	124.45(19)
		C21-C20-C19	119.60(17)
		O6-C20-C19	115.95(17)

Acknowledgements

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