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Synthesis, Fourier transform infrared, 1D and 2D NMR spectral studies on the conformation of two new cholesteryl 4-alkoxyphenyl-4′ benzoates

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Abstract

Two new cholesteryl 4-alkoxyphenyl-4′-benzoates ($C_nH_{2n+1}OC_6H_4C_6H_4COOCh$), where Ch represents cholesteryl moiety and $n = 10$ and 16) have been synthesized and their molecular orientation at ambient temperature were studied. The structure determination on these compounds was performed in solid state by infrared spectroscopy based on vibrational analysis wherein the cholesteryl–phenyl and phenyl–aliphatic carbon linkages were concluded. Their molecular structures were further ascertained through the ^1H and ^{13}C NMR spectra along with two-dimensional COSY, NOESY, ROESY, $^1H-^{13}C$ HMQC and HMBC. The long-range connectivity as concluded from the NOESY, ROESY and HMBC spectra together with the related data led to a postulation that the title compounds in the liquid state exist in the conformation whereby the cholesteryl moiety was not lying along the entire molecular long axis. The cholesteryl fragment was presumed to be bent at the ester linkage of O=C–O and the phenyl rings located between cholesteryl and alkoxy chain group are not coplanar.

Keywords: Structure determination; Vibrational analysis; ^1H NMR; ^{13}C NMR; 2D NMR; Cholesteryl-4-alkoxyphenyl-4′-benzoates

1. Introduction

Cholesterol is one of the well-known natural products since it is commercially available as an economic natural product and possesses multiple chiral carbon atoms which are prerequisites for chiral recognition [1–4]. Its derivatives exist in several unique aggregates such as liquid crystals, organic gels and monolayers [5–9]. Many of the cholesterol derivatives possessed cholesteric mesophases since the existence of cholesteryl fragment in the molecular structure is one of the requirements towards the formation of mesophase [4,10–12].

In 1888, the Australian botanists Reinitzer had discovered the first thermotropic liquid crystalline material known as cholesteryl benzoates, whereby the molecular structure consists of an aromatic ring linked to the cholesteryl fragment at the ester linkage of COO [13]. The expansion of cholesteryl benzoates known as cholesteryl 4-*n*-alkoxybenzoates ($C_nH_{2n+1}OC_6H_4COOCh$ or ROACh, where A and Ch represent a phenyl ring and cholesteryl moiety, respectively) were made 82 years later by Dave and coworker and their mesomorphic properties had also been studied [14]. Later, Yakubov had performed a systematic study of infrared spectra of ROACh in different phases and found a number of correlations between spectra, structure and mesomorphic characteristic of these compounds [15].

In this paper, we report another expansion of cholesteryl benzoates whereby we synthesized cholesteryl 4-alkoxyphenyl-4′-benzoates ($C_nH_{2n+1}OC_6H_4C_6H_4COOCh$ or ROABCh), a new liquid crystals compound which consists of two phenyl rings (A and B) compared to one ring in ROACh. The conformation of the compounds thus obtained were elucidated by Fourier transformed infrared and NMR techniques including the two-dimensional multinuclear correlation spectroscopy. The partial ^1H and complete ^{13}C NMR assignments were performed for the first time to provide full account of NMR analysis for the conformation of ROABCh in the solution state. Hereafter, 10OABCh and 16OABCh denote ROABCh with the integer representing the number of carbon atom, $n = 10$ and 16, respectively.

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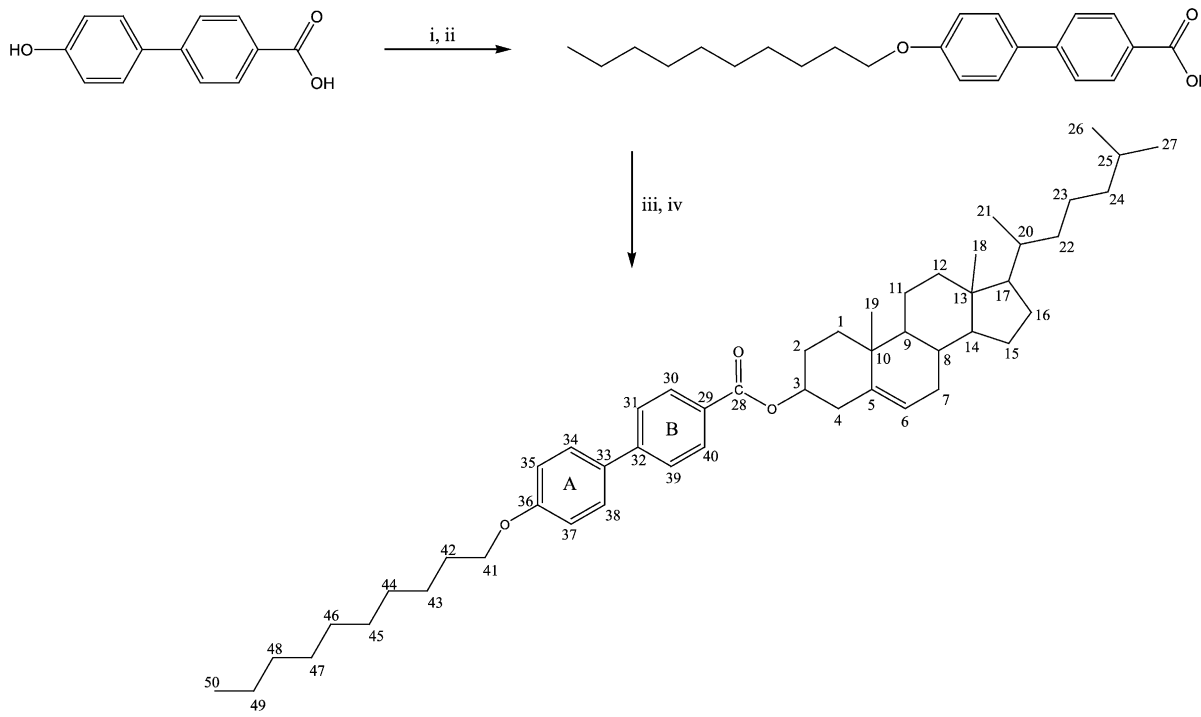

Fig. 1. Synthesis and atomic labeling scheme for 10OABCh. Reagents and conditions; (i) $C_{10}H_{21}Br$, KOH, KI, CH_3OH, inert atmospheres, reflux for 12 h (ii) dilute HCl; (iii) $SOCl_2$, (iv) cholesterol, pyridine, CH_2Cl_2, inert atmospheres, reflux for 6 h.

2. Experimental

4-Hydroxybiphenyl-4′-carboxylic acid was obtained from TCI Chemical Company (Japan). Whilst cholesterol was purchased from Acros Organics (USA), 1-bromodecane and 1-bromohexadecane were obtained from Aldrich Chemical Company (USA).

2.1. Synthesis of 4-n-decyloxy-4′-biphenylcarboxylic acid

The compounds were prepared according to the scheme as shown in Fig. 1. 4-*n*-Decyloxy-4′-biphenylcarboxylic acid was prepared by alkylation of 4-hydroxybiphenyl-4′-carboxylic acid. The white solid thus obtained was acidified with dilute hydrochloric acid. 4-*n*-Hexadecyloxy-4′-biphenylcarboxylic acid was synthesized with the similar method with 1-bromohexadecane.

2.2. Synthesis of cholesteryl 4-decyloxyphenyl-4′-benzoates (10OABCh)

Cholesteryl 4-decyloxyphenyl-4′-benzoates was synthesized in the modified reaction described by Gray [16]. 4-*n*-Decyloxy-4′-biphenylcarboxylic acid was converted to

Table 1
Acquisition parameters used in the NMR measurements

Parameters	Types of Bruker programs						
	^{1}H NMR	^{13}C NMR	2D COSY	2D NOESY	2D ROESY	2D HMQC	2D HMBC
SF	400.1 MHz	100.6 MHz	400.1 MHz	400.1 MHz	400.1 MHz	F_1 = 100.6 MHz F_2 = 400.1 MHz	F_1 = 100.6 MHz F_2 = 400.1 MHz
SW	12 ppm	200 ppm	10 ppm	14 ppm	12 ppm	F_1 = 200 ppm F_2 = 12 ppm	F_1 = 200 ppm F_2 = 12 ppm
PW	8.3 μs (30° flip angle)	20.0 μs (90° flip angle)	8.3 μs (90° flip angle)	8.3 μs (90° flip angle)	8.3 μs (90° flip angle)	8.3 μs (90° flip angle)	8.3 μs (90° flip angle)
AQ	4.0 s	1.3 s	0.2 s	0.2 s	0.2 s	0.1 s	0.4 s
D1	1.0 s	2.0 s	2.0 s	1.5 s	1.5 s	1.0 s	1.0 s
NS	16	6699	24	24	24	20	24
TD	65536	65536	F_1 = 256 F_2 = 2048	F_1 = 400 F_2 = 2048	F_1 = 400 F_2 = 2048	F_1 = 512 F_2 = 1024	F_1 = 512 F_2 = 4096

Abbreviations: F_1, ^{13}C channel; F_2, ^{1}H channel (except 2D COSY, NOESY and ROESY, where F_1 and F_2 are ^{1}H channels); SF, spectrometer frequency; SW, spectral width; PW, pulse width; AQ, acquisition time; D1, relaxation delay; NS, number of scan; TD, number of data points.

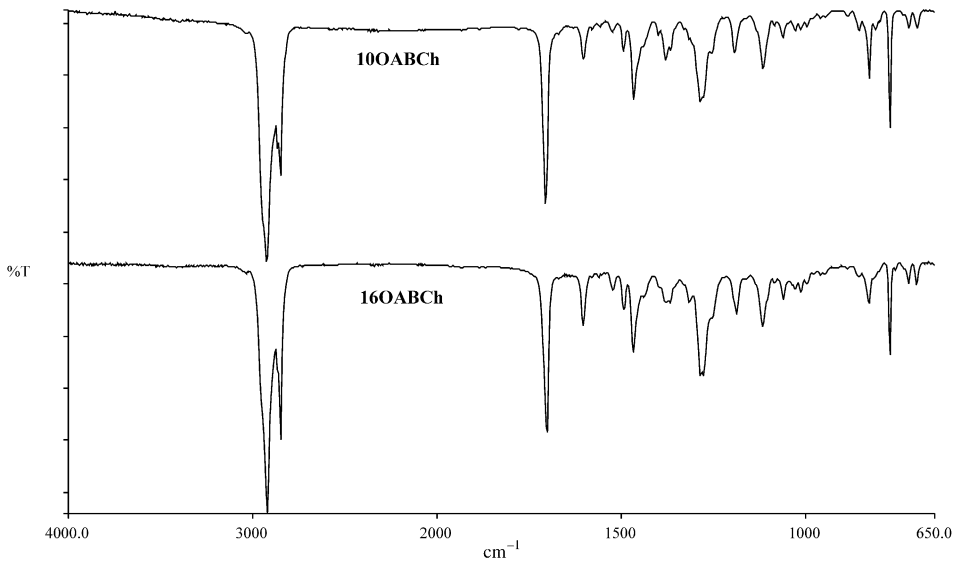

Fig. 2. FTIR spectra of 10OABCh and 16OABCh sandwiched between zinc selenide windows and measured within frequency range 4000–650 cm^{-1}.

its acid chloride using excess thionyl chloride. The acid chloride produced was then esterified with cholesterol in the presence of pyridine as the base. The esterification reaction was performed under inert atmospheres. Cholesteryl 4-hexadecyloxyphenyl-4′-benzoates (16OABCh) was prepared with the similar method. The labeling of atomic positions in 10OABCh (Fig. 1) was numbered in a sequential manner for discussion purpose in the later part of spectroscopic techniques.

The purification of both compounds was carried out by using column chromatography over silica gel 60 with a mixture of chloroform: *n*-hexane (1:1) as the eluent.

2.3. FT-IR measurements

The FT-IR spectra of 10OABCh and 16OABCh were recorded by using Perkin Elmer 2000-FTIR spectrophotometer in the frequency range 4000–650 cm^{-1}. All solid samples were sandwiched between two zinc selenide windows prior to measurement at ambient temperature.

2.4. NMR measurements

NMR spectra were recorded in $CDCl_3$ at 298 K on a Bruker 400 MHz Ultrashield™ equipped with a 5 mm BBI inverse gradient probe. Chemical shifts were referenced to internal TMS. The concentration of solute molecules was 25 mg in 0.8 ml $CDCl_3$. Standard Bruker pulse programs [17] were used throughout the entire experiment. The spectroscopic details were summarized in Table 1.

3. Results and discussion

The molecular structure associated with the title compounds 10OABCh and 16OABCh was investigated first in the solid state by using the infrared spectral analysis which was further studied by advanced NMR techniques wherein the compounds were present in solution.

3.1. IR spectroscopy

The IR spectra of the title compounds and the selected data were shown in Fig. 2 and Table 2, respectively. It is clearly shown from the figure and data that the title compounds were well isolated wherein the terminal phenolic hydroxy group (OH) attached at one end of the biphenyl moiety was substituted by OR, where R=C_nH_{2n+1} and $n = 10$ and 16. The presence of these alkyl groups can be supported by the appearance of diagnostic bands within the frequency range 2926–2852 cm^{-1} as reported by Wiberley and his coworkers [18]. The bands with strong intensities observed at 1706 and 1703 cm^{-1} in the IR spectra of 10OABCh and 16OABCh, respectively, can be ascribed to the carbonyl C=O as that present in the ester

Table 2
Frequencies (cm^{-1}) and relative intensities of the assigned bands of 10OABCh and 16OABCh

Assignments	Compound	
	10OABCh	16OABCh
$\nu_{C-H\ stretching}$	2926 vs	2923 vs
	2852 s	2852 s
$\nu_{C=O}$	1706 s	1703 s
ν_{CH2} [a]	1467 m	1469 m
$\nu_{C29-C28-O}$	1286 m	1287 m
ν_{C36-O}	1256 w	1257 w
$\nu_{arom(1,4)}$ [b]	828 m	830 w

Abbreviations: v, very; s, strong; m, medium; w, weak.
[a] Methylene groups in cholesteryl fragment.
[b] 1,4-Disubstituted aromatic ring.

Table 3
^{1}H NMR chemical shifts (ppm) and coupling constants (Hz) of 10OABCh and 16OABCh in $CDCl_3$

Atom	Chemical shift (ppm)		
	Cholesterol[a]	10OABCh	16OABCh
H4	2.19–2.33, m	2.49–2.51, d, $J = 7.7$	2.49–2.51, d, $J = 7.7$
H3	3.47–3.57, m	4.88–4.91, m	4.86–4.94, m
H6	5.36, d	5.45–5.46, d, $J = 3.8$	5.45–5.46, d, $J = 4.0$
H41	–	4.01–4.04, t, $J = 6.6$	4.01–4.04, t, $J = 6.6$
H35&H37	–	6.99–7.03, dd, $J = 4.5, 8.7$	6.99–7.02, dd, $J = 3.6, 8.7$
H34&H38[b]	–	7.57–7.59, d, $J = 8.6$	7.56–7.58, d, $J = 8.6$
H31&H39[b]	–	7.62–7.64, d, $J = 8.6$	7.62–7.64, d, $J = 8.8$
H30&H40	–	8.09–8.11, dd, $J = 2.6, 8.7$	8.08–8.11, dd, $J = 2.5, 8.5$

TMS as internal standard; d, doublet; dd, double doublets; t, triplet; m, multiplet.
[a] Reported by Bhacca et al. [26].
[b] The peaks ascribed to the pairs of (i) H31&H39 and (ii) H34&H38 are doublets wherein only one J coupling constant was observable in each (i) and (ii).

bond between the biphenyl moiety and cholesterol fragment (Fig. 1). Another band with medium intensities observed for the title compounds at 1467–1469 cm^{-1} can be due to the methylene groups of cholesterol fragment [19]. A strong band occurred at 1286 and 1287 cm^{-1} in the IR spectra of 10OABCh and 16OABCh, respectively, can be attributed to the stretching of C29–C28–O. This observation resembles that reported by Smith et al. [20]. In addition to this band, there is another band occurring at 1256–1257 cm^{-1} with weak intensities indicating the stretching of C36–O in ether bond [20,21]. The 1,4-disubstitued aromatic ring attached to the cholesteryl fragment can also be assigned from the diagnostic band observed at 828 and 830 cm^{-1} in the respective IR spectra of 10OABCh and 16OABCh. These bands fall within the frequency range as reported by Landgrebe [22].

The infrared spectral investigation with the understanding associated with the chemical stabilities exhibited by the cholesterol fragment and the alkoxy bonding in the biphenyl group in ambient temperature have led to a conclusion that the alkoxylated biphenyl which carried the long alkyl carbon chain of carbon number 10 and 16 have resulted in the formation of elongated cholesteryl 4-alkoxyphenyl-4′-benzoates. Their molecular structure can best be represented as that shown in Fig. 1.

3.2. NMR spectroscopy

In order to further analyze the conformation in the solution state, the conventional 1D ^{1}H NMR, ^{13}C NMR, DEPT along with 2D COSY, NOESY, ROESY, HMQC and HMBC were used to further substantiate the molecular structures of the title compounds. Whilst the selected 1D ^{1}H NMR data and the relevant coupling constants for cholesterol, 10OABCh and 16OABCh are shown in Table 3, the ^{13}C chemical shift are summarized in Table 4. ^{1}H–^{1}H COSY, ^{1}H–^{1}H NOESY and ^{1}H–^{1}H ROESY maps used to establish the relative disposition of the protons and the observed homonuclear cross peaks are tabulated in Table 5. Whilst the type of ^{13}C nuclei was determined by DEPT, the ^{13}C assignments were deduced from HMQC experiments. The remaining signals owing to the quaternary carbons were derived from HMBC correlations (Table 6). HMBC experiment was also used to determine the multiple bonds ^{1}H–^{13}C correlations.

3.2.1. ^{1}H spectral assignment

The signal corresponding to the proton attached with C3 in cholesterol was shifted to lower field ($\delta = 4.88$–4.91 ppm) upon replacement of OH by OCOR′ in 10OABCh where R′ is $C_6H_4C_6H_4C_{10}H_{21}$ (Table 3). The COSY, NOESY and ROESY experiments were subsequently used to determine the positions of the protons at C4 and C6 wherein the proton at C3 was correlated with the protons at C4 but not with the protons at C6. A typical COSY spectrum resulted from the observed ^{1}H–^{1}H connectivities in 10OABCh is shown in Fig. 3. It is known that the protons at C-4 were far from H6 but surprisingly the information from all 2D homonuclear experiments (Table 5) seemed to indicate these protons (H4 and H6) were coupled with each other. The long-range coupling ^{4}J of both protons showed $J = 3.8$ Hz.

The peaks which appeared within $\delta = 6.99$–8.11 ppm in the ^{1}H NMR spectrum of 10OABCh indicate the presence of aryl group. The protons associated with the aryl group were further identified by the NOESY and ROESY experiments. Through these techniques, the H35 atom and its equivalent H37 atom were distinguished from both H30 and H40 atoms. Similarly, the methylene protons at C41 were found to be correlated with H35 (or H37) but not with H30 (or H40) (Table 5). This observation was further supported by the presence of double doublets (dd) shown by H35 (or H37) in the ^{1}H NMR (Table 3) which can be ascribed to the coupling with H34 (or H38) and the methylene protons at C41 (Table 5). After distinguishing the H35 (or H37) from H30 (or H40), H31 (or H39) could easily be assigned since it only showed correlation with H30, which is identical with

H40. Fig. 4 shows the selected NOE and ROE relationships as observed from the NOESY and ROESY spectra. Similar characteristics shown by 16OABCh are also favourable for the assignments of H and C atoms.

3.2.2. ^{13}C spectral assignment

The ^{13}C NMR signals were identified by DEPT before being assigned by the analysis of the HMQC spectrum for

Table 4
^{13}C NMR chemical shifts (ppm) of cholesterol, 10OABCh and 16OABCh in $CDCl_3$

Carbon no.	Chemical shift (ppm)		
	Cholesterol	10OABCh	16OABCh
1	37.67	37.47	37.47
2	32.02	29.97	29.96
3	72.13	74.92	74.92
4	42.68	38.67	38.67
5	141.17	140.13	140.12
6	122.07	123.16	123.16
7	32.30	32.35	32.35
8	32.30	32.31	32.31
9	50.54	50.47	50.47
10	36.89	37.07	37.08
11	21.49	21.46	21.46
12	40.19	40.16	40.16
13	42.72	42.74	42.74
14	57.17	57.12	57.11
15	24.69	24.71	24.71
16	28.64	28.65	28.65
17	56.57	56.56	56.56
18	12.26	12.27	12.28
19	19.80	19.80	19.80
20	36.20	36.21	36.22
21	19.13	19.13	19.14
22	36.60	36.60	36.60
23	24.25	24.25	24.25
24	39.92	39.93	39.93
25	28.41	28.43	28.43
26	22.97	22.97	22.97
27	23.22	23.23	23.23
28		166.38	166.22
29		129.33	129.32
32		132.66	132.65
33		145.52	145.51
36		159.81	159.81
30, 40		130.46	130.46
34, 38		128.71	128.70
31, 39		126.75	126.75
35, 37		115.33	115.33
41		68.56	68.55
X[a]		23.08, 26.35, 26.45, 29.66, 29.72, 29.74, 29.81, 29.99	23.10, 26.35, 26.45, 28.33, 29.48, 29.67, 29.74, 29.78, 29.81, 29.99, 30.01, 30.07, 30.11, 32.34
Y[b]		14.52	14.53

TMS used as internal standard.
[a] 10OABCh: $X = C42–C49$; 16OABCh: $X = C42–C55$.
[b] 10OABCh: $Y = C50$; 16OABCh: $Y = C56$.

Table 5
$^{1}H–^{1}H$ correlations from 2D COSY, NOESY and ROESY on 10OABCh and 16OABCh

Compounds	Atom H	COSY	NOESY	ROESY
10OABCh	4	3, 6	3, 6	3, 6
	41	–	35 or 37	35 or 37
	3	4	4	4
	6	4	4	4
	35 or 37	34 or 38	34 or 38, 41	34 or 38, 41
	34 or 38	35 or 37	35 or 37	35 or 37
	31 or 39	30 or 40	30 or 40	30 or 40
	30 or 40	31 or 39	31 or 39	31 or 39
16OABCh	4	3, 6	3	6
	41	–	35 or 37	35 or 37
	3	4	4	–
	6	4	–	4
	35 or 37	34 or 38	34 or 38, 41	34 or 38, 41
	34 or 38	35 or 37	35 or 37	35 or 37
	31 or 39	30 or 40	30 or 40	30 or 40
	30 or 40	31 or 39	31 or 39	31 or 39

Table 6
2D $^{1}H–^{13}C$ HMQC and HMBC correlations for 10OABCh and 16OABCh

Compounds	Atom	HMQC	HMBC [J(C,H)]			
		^{1}J	^{2}J	^{3}J	^{4}J	J^{a}
10OABCh	H4	C4	C3, C5	C2, C6, C10	C1	–
	H41[b]	C41	–	C36	–	–
	H3	C3	–	–	–	–
	H6	C6	C7	C8, C10	C1	C36
	H35	C35	C36	–	C32	C6
	H37	C37	C36	–	C32	C6
	H34	C34	C33	C36	C31 or C39	–
	H38	C38	C33	C36	C31 or C39	–
	H31	C31	C32	C33	C34 or C38	–
	H39	C39	C32	C33	C34 or C38	–
	H30	C30	–	C28	C33	–
	H40	C40	–	C28	C33	–
16OABCh	H4	C4	C3, C5	C2, C6, C10	C1	–
	H41[a]	C41	–	C36	–	–
	H3	C3	–	–	–	–
	H6	C6	C7	C8, C10	C1	–
	H35	C35	C36	–	C32	C6
	H37	C37	C36	–	C32	C6
	H34	C34	C33	C36	C31 or C39	–
	H38	C38	C33	C36	C31 or C39	–
	H31	C31	C32	C29	C34 or C38	–
	H39	C39	C32	C29	C34 or C38	–
	H30	C30	–	C28	C33	–
	H40	C40	–	C28	C33	–

[a] Intramolecular interaction.
[b] Correlate with methylene in the alkyl chain but cannot determine their real positions because ^{13}C chemical shift for the methylene chain cannot be distinguished.

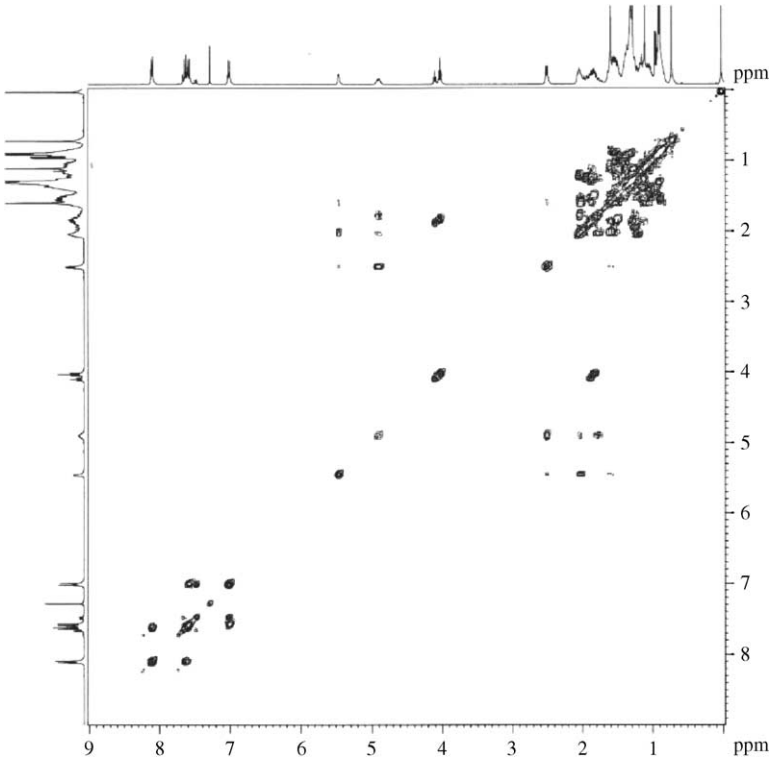

Fig. 3. $^{1}H-^{1}H$ connectivities in the COSY spectrum of 10OABCh.

the protonated carbons on the basis of the additivity rules and substituent effects [23,24]. A HMQC spectrum for 10OABCh is shown in Fig. 5. The assignments for these carbon nuclei were supported by HMBC cross peaks as that exhibited in the HMBC spectrum of 10OABCh (Fig. 6). In order to substantiate the carbon assignments on the cholesterol fragment, efforts were also made through a comparison with the signals inferred from the NMR spectrum of unreacted cholesterol [25].

Inspection from ^{13}C NMR data (Table 4) showed that the signals corresponding to C2, C3 and C4 in cholesterol were shifted upon replacement of OH by OCOR′ in 10OABCh. This phenomenon resembles that observed for the H3 atom in the ^{1}H NMR. Whilst

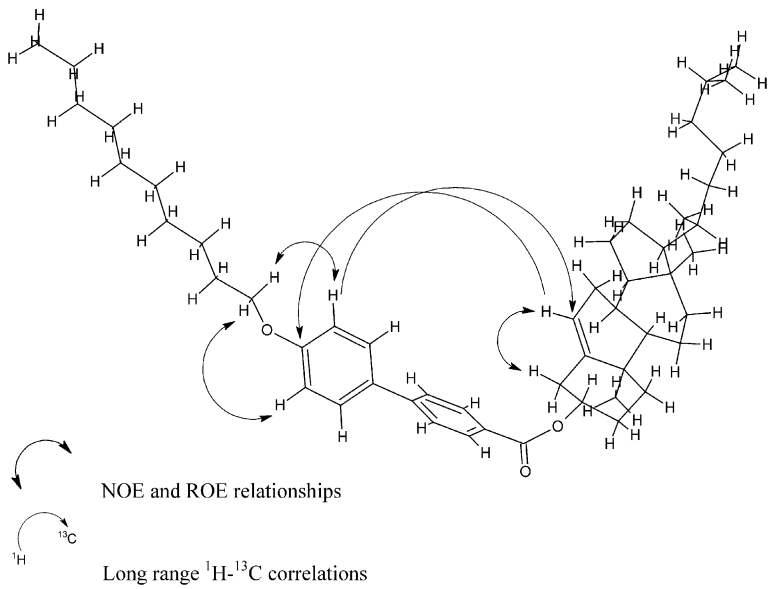

Fig. 4. Selected NOE and ROE relationships and intramolecular interaction via HMBC for 10OABCh.

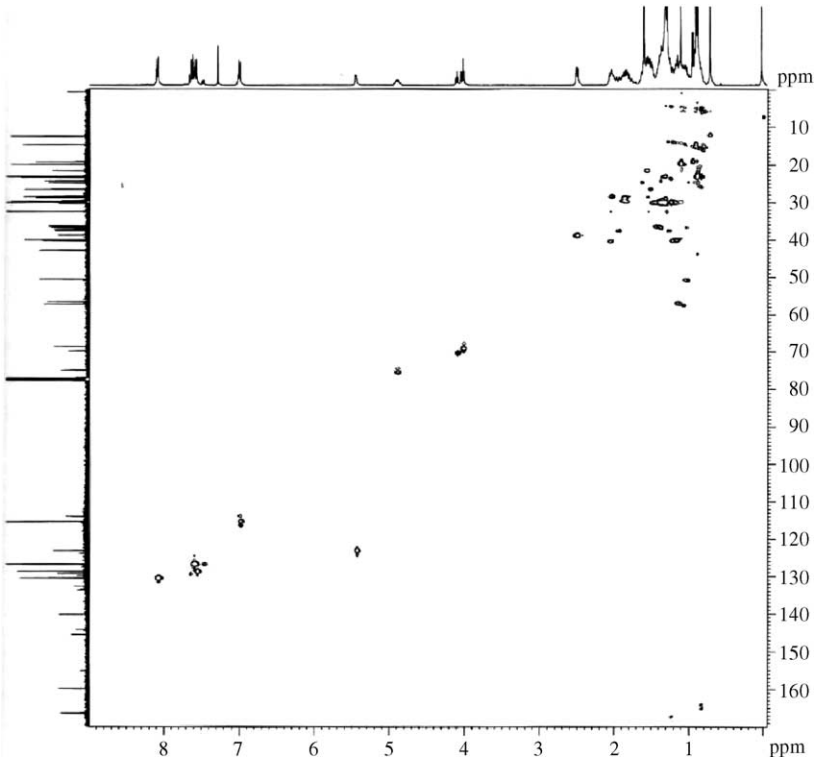

Fig. 5. One bond CH correlations in the HMQC spectrum of 10OABCh.

the chemical shifts for C2 and C4 were observed at the high field, C-3 signal was shifted to opposite field. Only these carbons were shifted drastically because they are located near the OH group in cholesterol. 16OABCh shows similar shift pattern as those discussed for 10OABCh.

All the ^{13}C chemical shifts sequence for both title compounds was unchanged in $CDCl_3$ under ambient

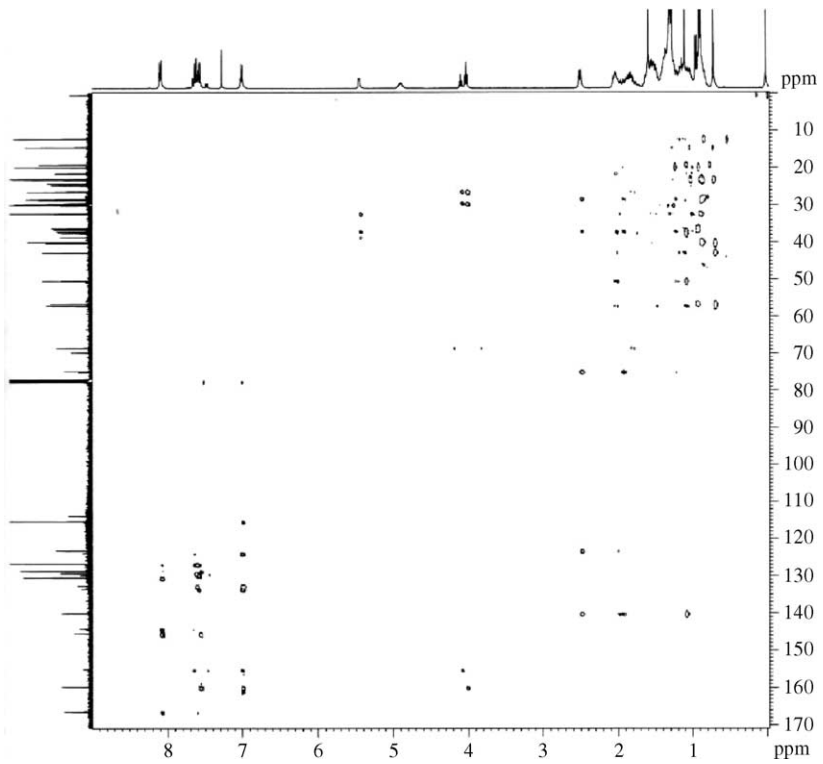

Fig. 6. Long-range CH correlations in the HMBC spectrum of 10OABCh.

temperature. The signals due to the carbonyl, aromatic and aliphatic carbons were observed in these spectra. The difference between these two compounds was that there are six more signals in the ^{13}C spectrum of 16OABCh in comparison with that in 10OABCh.

As for 10OABCh, the HMQC spectrum (Fig. 5) confirmed the attachments of different aromatic hydrogens onto the respective carbons as that discussed in the earlier part on the $^{1}H-^{1}H$ homonuclear correlations. From this figure, the carbon atoms occurred at different chemical shifts can be assigned. The signals owing to the C35 (or C37), C34 (or C38), C31 (or C39) and C30 (or C40) atoms were observed at $\delta = 115.33$, 128.71, 126.75 and 130.46 ppm, respectively. A cross peak due to the coupling between the proton at C4 and a carbon C5 located at $\delta = 140.13$ ppm can be observed. The one-bond $^{13}C-^{1}H$ connectivities were well observed for C4, C41, C3 and C6 which cross peaks appeared at $\delta = 38.67$, 68.56, 74.92 and 123.16 ppm, respectively.

The aromatic quaternary carbons in 10OABCh were established throughout the connectivities between the carbon and its neighbouring proton by using a long-range correlations HMBC experiment (Fig. 6). Thus, the long-range cross peaks of H30 (or H40) with C28 ($\delta = 166.38$ ppm), H31 (or H39) with C29 ($\delta = 129.33$ ppm) and the methylene protons at C41 with C36 ($\delta = 159.81$ ppm) suggest strongly the positions of these atoms. The C32 and C33 carbons were assigned via correlations with H31 (or H39) and H34 (or H38), respectively. 16OABCh shows similar characteristics as those discussed for 10OABCh.

The signal attributed to C36 correlated to H6 proton. On the basis of this analysis, the long-range connectivity through intramolecular interaction between C6 and H37 proton was established even though the C6 atom is far away from H37. One of the probabilities of having this kind of interaction is to bring together the atoms into close proximity. As shown in Fig. 6, the C37, C38, C39 and C40 are likely to be brought near each other by folding up at the O=C–O linkage. The observation of non-interaction between H31 (or H39) and H34 (or H38) as inferred from 2D homonuclear experiments (Table 5) indicates that biphenyl moeities are not coplanar and this phenomenon has further supported the molecular structure of 10OABCh as elucidated from the NOE and ROE relationships and intramolecular interaction via HMBC for this compound (Fig. 4). Similar feature can also be inferred from the spectroscopic data for 16OABCh which is present in solution at ambient temperature.

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References

[1] E.B. Kyba, K. Koga, L.R. Sousa, M.G. Siegel, D.J. Cram, J. Am. Chem. Soc. 95 (1973) 2692.
[2] M. Newcomb, J.L. Toner, R.C. Helgeson, D.J. Cram, J. Am. Chem. Soc. 101 (1979) 4941.
[3] J.M. Lehn, C. Sirlin, J. Chem. Soc., Chem. Commun. (1978) 143.
[4] P.J. Collings, M. Hird, Introduction to Liquid Crystals and Physics, Taylor and Francis, USA, 1998, pp. 10–112.
[5] W. Elser, R.D. Ennulat, J. Phys. Chem. 74 (1970) 1545.
[6] J.M. Pochan, H.W. Gibson, J. Am. Chem. Soc. 94 (1973) 5573.
[7] H.W. Gibson, J.M. Pochan, J. Phys. Chem. 77 (1973) 837.
[8] M. Koden, S. Takenaka, S. Kusabayashi, Bull. Chem. Soc. Jpn. 55 (1982) 2460.
[9] S. Shinkai, K. Murata, J. Mater. Chem. 8 (1998) 485.
[10] R.D. Ennulat, Mol. Cryst. Liq. Cryst. 8 (1969) 247.
[11] J.L.W. Pohlmann, W. Elser, Mol. Cryst. Liq. Cryst. 8 (1969) 427.
[12] F.F. Knapp, H.J. Nicholas, in: J.F. Johnson, R.S. Porter (Eds.), Liquid Crystals and Ordered Fluids, Plenum Press, New York, 1970, p. 147.
[13] F. Reinitzer, Monatsch Chem. 9 (1888) 421.
[14] J.S. Dave, R.A. Vora, in: J.F. Johnson, R.S. Porter (Eds.), Liquid Crystals and Ordered Fluids, Plenum Press, New York, 1970, p. 477.
[15] A.A. Yakubov, J. Mol. Struct. 484 (1999) 11.
[16] G.W. Gray, J.W. Goodby, Mol. Cryst. Liq. Cryst. 37 (1976) 157.
[17] Bruker program 1D WIN-NMR (release 6. 0) and 2D WIN-NMR (release 6.1)
[18] S.E. Wiberley, S.C. Bunce, W.H. Bauer, Anal. Chem. 32 (1960) 217.
[19] F.S. Parker, Applications of Infrared Spectroscopy in Biochemistry, Biology and Medicine, Plenum Press, New York, 1971, p. 322.
[20] B. Smith, Infrared Spectral Interpretation, CRC Press, New York, 1999, p. 75.
[21] S. Sudhakar, T. Narasimhaswamy, K.S.V. Srinivasan, Liq. Cryst. 27 (2000) 1525.
[22] J.A. Landgrebe, Theory and Practice in the Organic Laboratory, Brooks/Cole Publishing Company, Pacific Drive, CA, 1993, p. 255.
[23] S.R. Salman, S.J. Titinchi, H.S. Abbo, A.A.H. Saeed, Spectrosc. Lett. 23 (1990) 447.
[24] S.R. Salman, R.D. Farrant, J.C. Lindon, Spectrosc. Lett. 24 (1991) 1071.
[25] J.W. Blunt, J.B. Stothers, Org. Magn. Res. 9 (1977) 439.
[26] N.S. Bhacca, L.F. Johnson, J.N. Shoolery, High Resolution NMR Spectra Catalog, First ed., Varian Associates, USA, 1962.