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NOTE

Undecyl 2,3-Dimethoxycinnamate from *Daphne oleoides*

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Undecyl 2,3-dimethoxycinnamate, a new natural product, was isolated from the chloroform soluble fraction of the whole plant of *Daphne oleoides*. The structure of the new compound was established by modern spectroscopic methods.

Key Words: Undecyl 2,3-dimethoxycinnamate, *Daphne oleoides*, Thymelaeaceae.

Daphne oleoides, belonging to the family of Thymelaeaceae, is a small multi-branched shrub found in the northern hilly areas of Pakistan and many other Asian, African and European countries. It finds a significant medicinal importance in a variety of uses as a traditional folk medicine[1]. Previous studies on this species have resulted in the isolation of coumarins[2,3], coumarin lignanoids[4], lignans[5-7] and other aromatic compounds[8,9]. This paper deals in the isolation and structure elucidation of a new undecyl 2,3-dimethoxycinnamate from the same species.

Methanolic extract of the whole plant of *Daphne oleoides* was divided into hexane, chloroform, ethyl acetate and *n*-butanol soluble fractions. The chloroform soluble fraction was partitioned, by means of vacuum liquid chromatography (VLC) eluting with gradient system ($CHCl_3$-CH_3OH) of increasing polarity, into sub-fractions A, B, C, D, E and F, respectively. Repeated chromatographic purification of fraction A has led us to the isolation of compound **1** as light orange thick oil. The EI mass spectrum of **1** showed molecular ion peak at m/z 348 whereas the high-resolution MS measurement established the molecular formula as $C_{21}H_{32}O_4$, indicating six double bond equivalents. The IR spectrum (KBr) showed absorptions for an ester carbonyl at 1720 cm^{-1}, for an aromatic moiety at 1637 and 1520 cm^{-1}. The ^1H NMR of **1** showed the presence of a *trans*-vinylic moiety, displaying resonances at δ 7.99 (1H, d, J = 16.2 Hz) and 6.50 (1H, d, J = 16.2 Hz), three aromatic protons at δ 7.15 (1H, dd, J = 8.0, 1.5 Hz), 7.05 (1H, t, J = 8.0 Hz) and 6.93 (1H, dd, J = 8.0, 1.5 Hz). The spectrum further exhibited a triplet at δ 4.20 (t, 2H, J = 6.7 Hz), assigned to ester methylene group and two singlets displayed at δ 3.88 (s, 3H) and 3.86 (s, 3H), which were subsequently assigned to two methoxy groups. In addition, peaks for an aliphatic chain appeared as a triplet at δ 0.87 (t, 3H), assigned to the terminal methyl of the aliphatic chain and a broad multiplet between δ 1.39 to 1.26 (m, 16H).

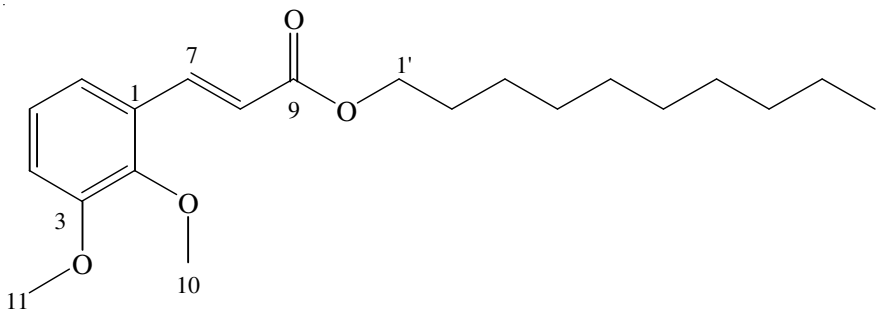

Fig. 1. Undecyl 2,3-dimethoxycinnamate

TABLE-1
^{1}H AND ^{13}C NMR DATA OF COMPOUND **1** (400 AND 125 MHz, $CDCl_3$, δ in ppm)

#H	$^{\delta}H$ (*J* in Hz)	#C	DEPT	$^{\delta}C$
-		1	C	128.63
-		2	C	153.07
-		3	C	147.10
4	6.93 (1 H, dd, *J* = 8.0, 1.5)	4	CH	119.16
5	7.05 (1 H, t, *J* = 8.0)	5	CH	119.55
6	7.15 (1 H, dd, *J* = 8.0)	6	CH	124.11
7	7.99 (1 H, d, *J* = 16.2)	7	CH	139.21
8	6.50 (1 H, d, *J* = 16.2)	8	CH	113.80
-		9	C	167.21
10	3.88 (3 H, s)	10	CH_3	61.26
11	3.86 (3 H, s)	11	CH_3	55.80
1'	4.20 (2 H, t, *J* = 6.7)	1'	CH_3	64.64
2'	1.70 (2 H, m)	2'	CH_2	31.84
3'	1.39-1.27 (16 H, br, m)	3'	[a]	[a]
4'	1.39-1.27 (16 H, br, m)	4'	[a]	[a]
5'	1.39-1.27 (16 H, br, m)	5'	[a]	[a]
6'	1.39-1.27 (16 H, br, m)	6'	[a]	[a]
7'	1.39-1.27 (16 H, br, m)	7'	[a]	[a]
8'	1.39-1.27 (16 H, br, m)	8'	[a]	[a]
9'	1.39-1.27 (16 H, br, m)	9'	[a]	[a]
10'	0.87 (3 H, t, *J* = 7.0)	10'	CH_3	14.07

[a]The resonances for C-3' to C-9' appeared in a complex pattern at δ 29.50 (2 × CH_2), 29.26 (2 × CH_2), 28.69, 25.94 and 22.63.

The ^{13}C NMR and DEPT experiments revealed the presence of five methine, nine methylene and three methyl carbon atoms. The quaternary carbon atoms were deduced by subtracting these from the broad band spectrum. The spectrum showed resonances at δ 139.21 and 113.80, assigned to vinylic carbons and a downfield peak at 167.12 assigned to the carbonyl carbon atom. The substitution pattern of the aromatic ring was established by the analysis of its ^{1}H NMR and NOESY spectra, which was confirmed by ^{1}H-^{1}H COSY, ^{1}H-^{13}C HMQC and HMBC experiments. NOE interaction was observed between protonsd 7.99 (7-H) and 7.16 (6-H).

In the HMBC experiments cross peaks were observed between proton δ 7.16 (6-H) and carbons δ 139.12 (7-C), 153.07 (2-C) and 119.55 (C-5). Likewise, proton at δ 6.93 (4-H) showed interactions with carbons at δ 153.07 (2-C) and 128.63 (C-6). The above evidences led to the structure of **1** as undecyl 2,3-dimethoxycinnamate.

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