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Gossypiline, a New Lignan from *Jatropha gossypifolia*

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Abstract – A new lignan, gossypiline (**1**) has been isolated from the aerial parts of *Jatropha gossypifolia*. The structure of **1** was established from its spectral data. The conversion of isogadain, a known lignan, to **1** confirmed the structure as well as the stereochemistry of **1**.

Key words – *Jatropha gossypifolia, Euphorbeaceae,* lignan, gossypiline.

Introduction

Jatropha gossypifolia Linn (Euphorbeaceae), a small shrub, possesses significant anti-cancer activity (Hartwell, 1969). The plant also exhibits pesticidal properties (Chatterjee *et al.*, 1980). In continuation of our work (Chatterjee *et al.*, 1988; Das and Das, 1995; Das *et al.*, 1996a; Das *et al.*, 1996b) on the chemical constituents of different parts of the plant we have recently isolated a new lignan, **1** from the aerial parts. Here we report the isolation and structure elucidation of **1**.

Experimental

Spectra were recorded with the following instruments: IR, Nicolet 740 FTIR spectrophotometer; ^{1}H- and ^{13}C-NMR, Varian Gemini 200 MHz; MS, VG Micromass 7070H (70 eV). Optical rotations were determined with a Jasco DIP 360 digital polarimeter. Column chromatography was performed on silica gel (BDH, 100-200 mesh) and TLC with silica gel G.

Plant material – The aerial parts of *J. gossypifolia* were collected from West Bengal. The voucher specimen (No. JG-A) is deposited in our laboratory.

Isolation of 1 – The air dried aerial parts (5 kg) of *J. gossypifolia* were powdered and extracted with CH_2Cl_2-MeOH (1 : 1) at room temperature for 120 hr. The concentrated extract was chromatographed over silica gel, the column being eluted with hexane and hexane-EtOAc mixture. Gossypiline (**1**) was isolated from the EtOAc eluent as a viscous mass (17 mg), $[\alpha]_D^{25}$ +31° (c 0.1, $CHCl_3$), found in dry sample: C, 65.82 and H, 5.27% (calculated for $C_{24}H_{24}O_8$: C, 65.45 and H, 5.45%).

Conversion of 2 to 1 – A solution of **2** (10 mg) in dry THF was added dropwise to a slurry of $LiAlH_4$ (50 mg) in THF (20 ml). The mixture was stirred for 6 hr. The excess reagent was decomposed with EtOAc and cold water. The mixture was filtered and the filtrate was concentrated. The solid residue was subsequently treated with acetic anhydride (3 ml) and pyridine (0.5 ml) and kept at room temperature overnight with continuous stirring. The reaction mixture was poured on crushed ice and extracted with CH_2Cl_2 (3×20 ml). The extract was washed with water (3×50 ml), concentrated and purified by column chromatography over silica gel. The fractions eluted with C_6H_6

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afforded a viscous mass (7 mg), $[\alpha]_D^{25}$ +30° (c 0.1, $CHCl_3$), which was identical to **1** in all respects ($[\alpha]_D$, IR, ^{1}H- and ^{13}C-NMR and mass spectra).

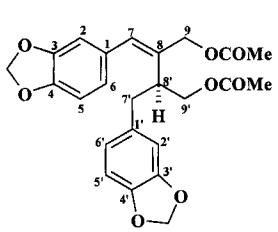
Results and Discussion

Gossypiline, a new lignan, was isolated as a viscous mass from the CH_2Cl_2-MeOH (1 : 1) extract of the aerial parts of *J. gossypifolia*. The IR spectrum of the compound showed the significant peaks at 1735, 1730 (ester carbonyls), 1610 (>C=C<), 1580, 1475 (aromatic residue) and 920 cm^{-1} (-OCH_2O-). The ^{1}H-NMR spectral data of **1** (Table 1) indicated that its structure is very similar to that of the known lignan, prasanthaline (**3**) (Chatterjee *et al.*, 1988). The only difference is that **3** contains one methylenedioxy and two methoxy groups while **1** contains two methylenedioxy groups. The assignments of the signals of the ^{1}H-NMR spectrum of the new lignan were made from a study of its ^{1}H, ^{1}H-COSY spectrum. The assignments of the signals of the ^{13}C-NMR spectrum were made from both a study of the DEPT spectrum and comparison of the data with those of **3** (Table 1) (Chatterjee *et al.*, 1988). The structure of **1** was supported from its mass spectrum which showed the molecular ion peak at m/z 440 (7%) (corresponding to $C_{24}H_{24}O_8$) along with two intense peaks at m/z 305 (56%) and 135 (100%) resulting from the benzylic cleavage of the molecule at C-7'-C-8'.

Table 1. ^{1}H-NMR (200 MHz, $CDCl_3$) and ^{13}C-NMR (50 MHz, $CDCl_3$) spectral data of **1**

C	^{1}H-NMR chemical shift (ppm)	^{13}C-NMR chemical shift (ppm)
1	-	121.7
2	6.55 (1H, d, *J*=1.5 Hz)	108.5
3	-	134.2
4	-	135.6
5	6.58 (1H, d, *J*=8.0 Hz)	110.7
6	6.72 (1H, dd, *J*=8.0&1.5Hz)	121.6
7	6.53 (1H, brs)	112.5
8	-	106.3
9	4.74 (1H, d, *J*=12.0 Hz)	64.8
	4.65 (1H, d, *J*=12.0 Hz)	
1'	-	120.1
2'	6.48 (1H, d, *J*=1.5 Hz)	106.4
3'	-	132.4
4'	-	132.8
5'	6.50 (1H, d, *J*=8.0 Hz)	109.0
6'	6.69 (1H, dd, *J*=8.0&1.5Hz)	120.2
7'	2.72 (1H, dd, *J*=14.0&6.0Hz)	36.4
	2.63 (1H, dd, *J*=14.0&7.0Hz)	
8'	3.57 (1H, m)	40.1
9'	4.13 (2H, m)	64.7
-OCH_2O-	5.92 (2H, s)	100.6
	5.90 (2H, s)	100.2
-$OCOCH_3$	2.08 (3H, s)	170.8 & 170.2[a]
	2.05 (3H, s)	22.1 & 21.5[b]

[a] ^{13}C-NMR data for carbonyl; [b] ^{13}C-NMR data for methyl.



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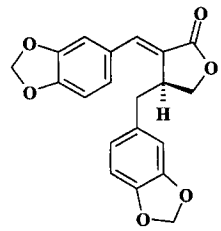

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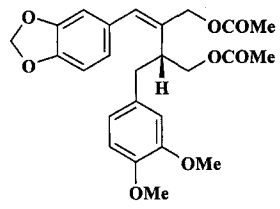

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Finally the structure as well as the stereochemistry of gossypiline was confirmed from preparation of **1** from **2**, a known lignan previously isolated by us from the title plant (Das *et al.*, 1996b). Isogadain (**2**) on reduction with lithium aluminium hydride in THF followed by acetylation with acetic anhydride and pyridine afforded a product as a viscous mass, $[\alpha]_D^{25}$ +30° (c 0.1, $CHCl_3$), which was found to be identical to gossypiline (**1**) in physical and spectral properties ($[\alpha]_D$, IR, ^{1}H- and ^{13}C-NMR and mass spectra). The stereochemistry of gossypiline at C-3 was thus established as S.

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