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SPECTRAL DIFFERENTIATION OF 3,3'-DI-O-METHYLELLAGIC ACID FROM 4,4'-DI-O-METHYLELLAGIC ACID

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Key Word Index—*Lagerstroemia fauriei*; Lythraceae; 3,3'-di-O-methylellagic acid; 4,4'-di-O-methylellagic acid; NMR spectral differentiation.

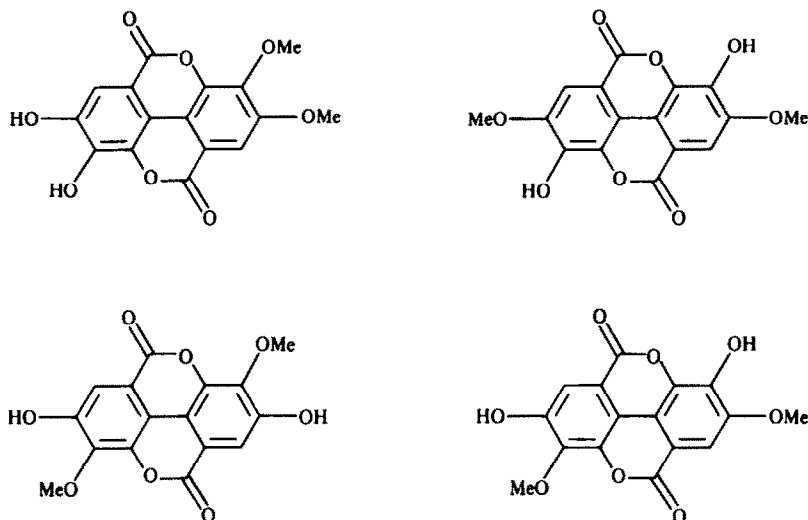
Abstract—3,3'- and 4,4'-di-O-methylellagic acid were synthesized, and their spectra (IR, UV, ^{13}C FT-NMR) were compared with each other. UV and ^{13}C FT-NMR spectra were the most useful for distinguishing them.

INTRODUCTION

The isolation of ellagic acid, and the presence of di-O-methylellagic acids has been reported from *Lagerstroemia fauriei* Koehne [1]. In the case of natural di-O-methylellagic acid, four structural isomers are possible (Scheme 1), but nothing is known of their spectral properties. So 3,3'- and 4,4'-di-O-methylellagic acid were synthesized and characterized by their UV, IR, ^1H and ^{13}C FT-NMR spectra.

RESULTS AND DISCUSSION

3,3'- and 4,4'-di-O-methylellagic acid are difficult to differentiate by using PC, TLC and ^1H NMR. The IR spectra show only small differences in the fingerprint region, which are insufficient for separating them. But the UV spectra are very different from each other (Table 1). The addition of alkali or AlCl_3 cause large bathochromic shifts in the long wavelength band, and change absorption patterns. The ^1H FT-NMR spectra were very similar,



Scheme 1.

Table 1. UV spectral data of 3,3'-di-*O*-methylellagic acid and 4,4'-di-*O*-methylellagic acid

Compound*	λ_{\max} (nm) (log ϵ)			
	MeOH	MeOH-NaOAc (1 mg)	MeOH-10%AlCl ₃ (5 gtt)	MeOH-28%NaOMe (5 gtt)
3,3'-di- <i>O</i> -Me	246(4.620)	254(4.504)	245(4.689)	246(sh) (4.388)
	287(sh†) (4.068)	305(sh) (3.981)	288(sh) (4.165)	272(4.545)
	354(4.068)	365(3.902)	357(4.231)	313(sh) (4.173)
	379(4.106)	410(3.930)	374(4.231)	436(4.115)
4,4'-di- <i>O</i> -Me	246(sh) (4.433)	245(sh) (4.504)	241(4.645)	245(sh) (4.458)
	255(4.450)	255(4.580)	315(4.354)	255(4.535)
	360(4.188)	279(4.661)	363(4.397)	279(4.655)
		353(4.217)		353(4.231)

*Sample (6.2×10^{-3} mg/ml) in MeOH.

†(sh): shoulder.

Table 2. ¹³CFT-NMR data* of 3,3'-di-*O*-methylellagic acid and 4,4'-di-*O*-methylellagic acid

Carbon no.	3,3'-di- <i>O</i> -Me	4,4'-di- <i>O</i> -Me
1(1')	111.68	107.32
2(2')	141.18	140.92
3(3')	140.30	136.32
4(4')	152.13	150.27
5(5')	111.52	107.07
6(6')	112.06	113.55
7(7')	158.37	158.96
-OMe	60.91	56.65

*Chemical shifts in δ values from TMS.

EXPERIMENTAL

MS spectra were measured with a Hitachi M-80A and M-003 data processing system. IR spectra were recorded in KBr discs. UV spectra were recorded in MeOH. ¹H and ¹³C FT-NMR spectra were measured in DMSO at 399.8 MHz (¹H) and 100.5 MHz (¹³C) with TMS as internal standard.

3,3'-Di-*O*-methylellagic acid. Synthesized by the method of Jurd [2]. It was recrystallized from dioxan-MeOH (1:1) to give the yellow needles, mp 332–335°; MS: C₁₆H₁₀O₈ (*m/z*: 330.0372). The IR spectrum was the same as that provided by Dr. M. A. M. Nawwar.

4,4'-Di-*O*-methylellagic acid. Synthesized by the method of Jurd [2], but methyl 3-*O*-methylgallate was prepared by the procedure of Scheline [3]. It was recrystallized from dioxane-MeOH (1:1) to give colourless crystals, mp 360–363°; MS: C₁₆H₁₀O₈ (*m/z*: 330.0368).

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whereas the ¹³CFT-NMR spectra (Table 2) are very different from each other. Thus, it is possible to distinguish their closely related isomers by spectral measurements.