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5-METHYLDIHYDROFLAVASPERONE, A DIHYDRONAPHTHOPYRAN FROM *GUIERA SENGALENSIS*

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Key Word Index—*Guiera senegalensis*; Combretaceae; leaves; naphthopyran; spectroscopy.

Abstract—5-Methyldihydroflavasperone, a novel naphthopyran, was isolated from the chloroform extract of the leaf of *Guiera senegalensis*. The structure was established as 2,3-dihydro-5,8,10-trimethoxy-2-methyl-4*H*-naphtho[1,2-b]pyran-4-one by 1D and 2D NMR spectroscopy (H-H COSY; H-C COSY, DEPT; INADEQUATE) and other spectroscopical techniques. © Elsevier Science Ltd

INTRODUCTION

Guiera senegalensis Lam. (Combretaceae) is commonly used in African traditional medicine to treat dysentery, chest infection and rheumatic pain. Leaves are taken after childbirth to increase lactation. Pharmacological investigations of this taxon showed a depressive action on CNS, as well as anti-inflammatory and antidiarrhoeic activities [1]. The presence of indole alkaloids harmine and tetrahydroharmine has been reported previously [2]. However, our phytochemical study on the leaves of this taxon revealed no trace of these alkaloids. The present paper describes the isolation and structural determination of a new naphthopyran 5-methyldihydroflavaspersone, from the leaves of *G. senegalensis*.

RESULTS AND DISCUSSION

The chloroform extract of the leaves was treated as described in the Experimental to yield 5-methyldihydroflavasperone (1). Elemental analysis gave the formula $C_{17}H_{18}O_5$. The UV spectrum exhibited maxima at 241, 246, 305, 259, 312 and 318 nm. The IR spectrum showed absorption bands due to a conjugated carbonyl (1670 cm^{-1}) and aromatic rings (1620, 1570 and 1390 cm^{-1}). ^1H NMR spectrum showed a doublet at δ 1.60 indicative of a methyl group, a multiplet at δ 2.70 associated with a methylene group and three singlets at δ 3.90. 3.92 and 3.96 (3H each) attributed to three methoxyl groups at C-8, C-10 and C-5, respectively, in addition to a multiplet at δ 4.70 integrated to one proton. The aromatic region exhibited two doublets (δ 6.30 and 6.49) and a singlet at 6.52 (1H each). The doublet (3H) at 1.60 gave a correlation to the multiplet (1H) at δ 4.70 in homonuclear COSY spectrum which also gave a correlation to the overlapping multiplet (2H) at δ 2.70. The presence of a heteroatom (oxygen) accounted for the chemical shift of the C$\underline{H}$ proton at δ 4.70. The methylene protons must be non-equivalent due to restricted rotation, giving a complex splitting pattern at δ 2.70. This partial structure being fused to an aromatic ring would explain this restricted rotation. The two doublets at δ 6.30 and 6.49 correlated with each other ($J = 2.5$ Hz) and the coupling constant indicated a *meta* substitution pattern.

The ^{13}C NMR spectrum confirmed that there are 17 carbon atoms and exhibited a chemical shift of δ 190.6 indicative of a carbonyl carbon which accounts for the last oxygen atom. The DEPT spectrum exhibited one methyl group at δ 20.6, three methoxy groups at δ 55.2, 55.7 and 55.8, one methylene group at 45.3, one methine gorup at δ 74.6 ($CH_3\underline{C}H$) and three aromatic carbons at δ 97.2, 98.4 (ring A) and 98.9 (ring B).

Unambiguous resonance identification was achieved through the application of long-range CH COSY and INADEQUATE spectra as well. Based on the above spectroscopic evidence, the structure of **1** was established as 2,3-dihydro-5,8,10-trimethoxy-2-methyl-4*H*-naphtho [1,2-bl]pyran-4-one.

The parent compound flavasperone was previously isolated from the fungus *Aspergillus niger* [3]. To date, no report is available on the isolation of related naphthopyrans from higher plants except for eleutherinol and dihydroeleutherinol from *Eleutherine bulbosa* and *Cassia torosa* Cav. [4], respectively.

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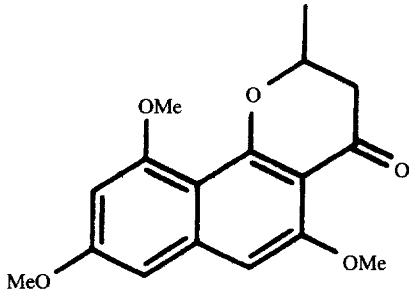

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EXPERIMENTAL

Plant Material. Leaves of *G. senegalensis* were collected in North Kordofan (SUDAN) in 1988. A voucher specimen has been deposited in the Department of Pharmacognosy, Faculty of Pharmacy, University of Khartoum.

Extraction and Isolation. Powdered leaves (300 g) were extracted with petrol (40–60°), $CHCl_3$ and finally MeOH. TLC revealed the presence of a fluorescent compound in the $CHCl_3$ extract, which was concd and subjected to column chromatography over silica gel. Elution with $CHCl_3$ containing 1% MeOH gave **1**, which was further purified by prep. TLC (silica gel; $CHCl_3$–MeOH = 19:1) to give pure **1** (25 mg).

5-*Methyldihydroflavasperone* (**1**). Yellow amorphous compound, UV_{max}^{EtOH} nm: 241 (3.11), 246 (3.10), 252 (3.07), 259 (3.05), 267 (3.05), 305 (3.16), 312 (3.14), 318 (3.15); IR_{max} cm^{-1}: 1670, 1620, 1570, 1390; NMR (20 mg) was dissolved in $CHCl_3$ (1 ml) and 1D and 2D NMR spectra were taken on a JEOL AM-500 instrument. For ^1H NMR and ^{13}C NMR data see Table 1. Elemental analysis (Calcd for C, 67.54; H, 6.00. $C_{17}H_{18}O_5$ Found: C, 66.91; H, 6.06). MS 70 eV *m/z* (rel. int.): 302$[M]^+$ (100), 274 (30), 231 (25), 175 (15), 69 (7), 41 (5).

Table 1. ^1H and ^{13}C NMR spectral data ($CDCl_3$, δ-values) of 5-methyldihydroflavasperone (**1**)

No.	^1H	^{13}C
2	4.70 (*m*)	74.6
2-CH_3	1.60 (*d*)	20.6
3	—	45.3
3-CH_2	2.70 (*m*)	—
4	—	190.6
4a	—	108.1
5	—	157.6
5-OCH_3	3.96 (*s*)	55.7
6	6.52 (*s*)	98.9
6a	—	141.4
7	6.49 (*d*)	98.4
8	—	161.5
8-CH_3	3.90 (*s*)	55.2
9	6.30 (*d*)	97.2
10	—	160.1
10-OCH_3	3.92 (*s*)	55.8
10a	—	107.3
10b	—	164.0

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