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Research Article

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Secondary metabolite from *Gladiolus segetum*

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ABSTRACT

Chemical investigation of the aerial part of Gladiolus segetum has yielded a secondary metabolite (2, 5, 6-trihydroxy-2, 4-dimethyl-6-metoxy-1-benzofuran-3-one,). The metabolite is reported for the first time and the chemical structure is established by a variety of one and two-dimensional NMR experiments.

Keywords: *Gladiolus segetum*, Iridaceae, NMR, Secondary metabolite, Structure.

INTRODUCTION

Gladiolus segetum (Iridaceae) [1, 2] is used in Algeria as a traditional medicine. However, it is a toxic plant lethal to livestock. . We aimed to elucidate the structure of the toxic compounds responsible for the lethal effect of *Gladiolus segetum*. Previous investigations of *Gladiolus segetum* have led to the isolation of saponins [3,4] and anthraquinones [5,6]. In earlier works [7], we reported the isolation and characterization of two novel anthraquinones.

EXPERIMENTAL SECTION

General Experimental Procedures

Melting points were determined on an electrothermal apparatus. The IR and UV spectra were recorded on a Perkin-Elmer 281 spectrophotometer and a Shimadzu UV-3101 PC spectrophotometer, respectively. Nuclear magnetic resonance (NMR) data were recorded on a Bruker DMX spectrometer. Chemical shifts δ are reported in ppm.MS spectra (HR-ESI-MS) were carried out on a Q-TOF 98 micro instrument (Bruker).

Plant Material

The plant material was collected in the region of Batna (Algeria) and was identified by Dr. Bachir Oudjehih, Institut of Veterinary and Agronomic sciences, Department of Agronomic science, Batna University, where a voucher specimen (183DAUB2004) is deposited.

Extraction and Isolation of Constituents

400g of dry matter (aerial part) of *Gladiolus segetum* was subjected to subsequent extraction by *n*-hexane (3 L), $CHCl_3$ (3 L), EtOAc (3 L). The EtOAc fraction (50 g) subjected to silica gel column chromatography. Elution was started with hexane and continued with gradients of EtOAc, and MeOH in order of increasing polarity yielding (6) fractions. Fractions were controlled via TLC (silica gel 60 F254, 0.25mm) techniques using $CHCl_3$–MeOH (9:1) as the eluent and visualized by spraying with 20% H_2SO_4 in ethanol and similar fractions were combined. Fractions II, was subjected to silica gel column chromatography eluting with ($CHCl_3$: MeOH gradient elution of increasing polarity) to yield the compound (8 mg).

Structure and Identification

The Compound was obtained as Yellow color needles: Mp 160–162°C, UV (MeOH): λ_{max} = 230, 275, 340, 420 nm; IR (KBr): *v*= 3.337, 3.200, 3.000, 1.720, 1.665, 1.630, 1.594, 1.420, 1.380, 1.275, 1.214, 1.180, 1.088, 997 cm^{-1}. HR ESIMS at m/z 263.3501 $[M + Na]^+$ (corresponding to $C_{11}H_{12}O_6$). For ^1H NMR ($CDCl_3$, 300 MHz), ^{13}C NMR ($CDCl_3$, 75 MHz) and 2D NMR, see Table 1

RESULTS AND DISCUSSION

The Compound was obtained as Yellow color needles, Mp 160–162°C and having the molecular formula of $C_{11}H_{12}O_6$ as determined by HR ESIMS and elemental analysis. The IR absorptions of the compound at 3,337 and 1,665 cm^{-1}showed the presence of hydroxyl and carbonyl group. In the ^1H NMR spectrums, there was a phenolic hydroxyl signal at δ 9.22. The ^{13}C NMR spectrum disclosed the presence of a ketone carbonyl group at δ 201.4 and signals for all 11 carbons; those included two methyl groups δ 22.4 and 17.4 a methoxy group δ 61.2, six aromatic carbons at δ 120, 116, 162.5, 132.3, 150 and 165.4, and a quaternary carbon at δ 105.1. The data suggested that the compound had a benzene ring system, a ketone [8], and a hemiacetal functionality δ 105.1. [9, 10, 11]

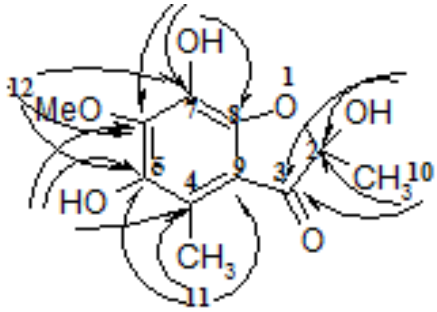

Figure 1: structure of the isolated compound and the key HMBC (H → C) correlations

The HMBC data established the structure of compound 1 (Figure 1), especially the multiple correlations from the OH-7 δ 9.22 to C-6, C-7 and C-8, and from the hydroxy δ 6.50 to C-2, C-3 and C-10 which assigned the positions of the hydroxyl groups. Signals attributable to a methoxy and methyl groups were observed at, 3.94 (C6), *δ* 1.53 (C2) and 1.60 (C4). The signal at *δ* 1.53 was assigned to hydrogen of the methyl group on C-2. This assignment was further confirmed from the HMBC correlations between the hydrogen signal of the methyl group on C-2 and C-3(*δ* 201.1) and C-2 (*δ* 105.2). HMBC correlations between signals at *δ* 3.94 and C-6, C-7 and C-5were used to place the methoxy on C-6. The signal at *δ* 1.60 was assigned to hydrogen of the methyl group on C-4. This assignment was further confirmed from the HMBC correlations between the hydrogen signal of the methyl group on C-4 and C-9(*δ* 120.0) and C-5 (*δ* 160.0). Based on these results, the novel compound was established to be 2, 5, 6-trihydroxy-2, 4-dimethyl-6-metoxy-1-benzofuran-3-one.

Table 1: NMR spectral data of the compound in $CDCl_3$

position	Compound		
	^{13}C-NMR	^1H-NMR	HMBC
1			
2	105.1		H-10, OH-2
3	201.4		H-10, OH-2
4	132.3		H-11, OH-5
5	165.4		H-11, OH-5, H-12
6	162.5.		H-12, OH-5, OH-7
7	150.0		H-12, OH-7
8	120.0		OH-7
9	116.0		H-11
10	22.4	1.53 (s)	OH-2
11	17.4	1.60(s)	
12	61.2	3.94 (s)	
2-OH		6.50 (s)	
7-OH		9.22 (s)	

CONCLUSION

In this study, the fractionation of the EtOAc extract of the aerial parts of *Gladiolus segetum* led to the isolation of a new compound: 2, 5, 6-trihydroxy-2, 4-dimethyl-6-metoxy-1-benzofuran-3-one.

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