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THE *N*-2-PHENYLETHYLCINNAMAMIDE FROM *SPILANTHES OCYMIFOLIA**

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Key Word Index—*Spilanthes ocymifolia*, Compositae, Heliantheae, *N*-2-phenylethylcinnamamide, stigmasterol, taraxasteryl and lupeyl acetates

Abstract—A simple amide, *N*-2-phenylethylcinnamamide and three known compounds, stigmasterol and taraxasteryl and lupeyl acetates, were isolated from the leaves of *Spilanthes ocymifolia* The structure of the cinnamamide derivative was established by spectroscopic means and confirmed by synthesis

INTRODUCTION

Spilanthes ocymifolia is a Salvadorian species which is a popular folk remedy for toothache and against the rabies disease, and it is known as 'hierba de la rabia' ('rabies grass') [1] In previous work on this genus have been reported the presence of spilanthol in *S oleracea* [2], α- and β-amyrin esters and sitosterol-*O*-β-D-glucoside in *S acmella* [3] and ten acetylenic amides in *S alba* [4] The isolation and characterization of a new amide, *N*-2-phenylethylcinnamamide, from *S ocymifolia* is described in the present communication It is interesting that this simple compound has not previously been found so far in nature though it has been described as a synthetic product [5]

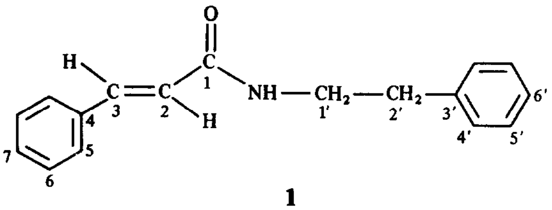
RESULTS AND DISCUSSION

The amide compound, $C_{17}H_{17}NO$, was obtained from the petrol and toluene extracts of the leaves of *S ocymifolia* as a crystalline product, mp 125–126° (cyclohexane), $[M]^+$ at m/z 251 Its IR spectrum had characteristic absorptions at ν_{max}^{KBr} cm^{-1} 3300 (–N$\underline{H}$–) and 1670 (–$\underline{CO}$–NH–) The ^{1}H NMR spectrum showed two doublets of one proton each at δ7 62 and 6 3 (J = 9 Hz) for olefinic protons and a multiplet centred around 7 53–7 0 for ten aromatic protons A triplet at 2 88 (J = 8 Hz) and a double triplet at 3 65 (J = 7 and J' = 8 Hz) were assigned to the ethylene protons In addition there was a signal at 5 66 that disappeared with D_2O which corresponds to the amide proton This was coupled with methylene protons at 3 65 When –NH– was irradiated the double triplet at 3 65 appeared as a triplet (J = 8 Hz) and by irradiation at 2 88 the double triplet became a doublet (J = 7 Hz) If the irradiated signal was that at 3 65 the triplet at 2 88 was converted into a singlet

The amide compound was therefore, formulated as *N*-2-phenylethylcinnamamide and its structure was confirmed by synthesis by heating cinnamic acid with phenylethylamine (yield 25%) A comparison of the ^{13}C NMR chemical shifts of this amide with the calculated values and with those of the amine and acid, respectively (Table 1) permitted assignment of structure **1**

Table 1 ^{13}C NMR spectral data for compound **1** (CDCl$_3$, TMS as internal standard)

Carbon	1	Calc	Acid	Amine
1	165 6	168 3	172 7	
2	120 5	117 5	117 3	
3	140 8	144 7	147 0	
4	134 6	136 1	133 9	
5	128 6	126 7	128 9	
6	128 6	126 7	128 3	
7	129 4	125 0	130 7	
1′	35 6	35 3		39 7
2′	40 7	40 2		43 2
3′	138 6	144 2		139 5
4′	127 6	127 9		128 0
5′	128 5	128 4		128 4
6′	126 3	125 7		125 7



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*Part 7 in the series "Salvadorian Compositae" For Part 6 see *Phytochemistry* **23**, 859 (1984)

In addition, the petrol extract afforded stigmasterol and taraxasteryl and lupeyl acetates The physical constants and spectroscopic data of these compounds were consistent with those reported in the literature [6–8]

EXPERIMENTAL

Mps was measured with a Buchi apparatus and are uncorr The IR spectrum was recorded in KBr

Extraction and isolation *Spilanthes ocymifolia* was collected in 1979 in El Salvador Leaves (3 3 kg) were extracted with EtOH The material obtained after removal of the EtOH was diluted with H_2O, and extracted × 3 with petrol and then × 3 with toluene, for 30 hr The petrol extract (10 g) was chromatographed on a column of silica gel eluted with increasing proportions of toluene followed by EtOAc, yielding the following compounds in order of elution taraxasteryl acetate, lupeyl acetate, stigmasterol and *N*-2-phenylethylcinnamamide (**1**)

The toluene extract (7 5 g) was chromatographed on a silica gel column with $CHCl_3$–Me_2CO mixtures as eluents, yielding stigmasterol and *N*-2-phenylethylcinnamamide (**1**) in order of elution

The previously known products were identified by their mp and spectroscopic (IR, ^1H NMR, ^{13}C NMR and MS) data

N-2-*phenylethylcinnamamide* White needles, mp 125–126° (cyclohexane) IR ν_{max}^{KBr} cm^{-1} 3300, 3100, 3060, 2960, 2960, 1670, 1620, 1570, 1460, 1350, 1230, 1000, 870, 770, 750, 730, 700, etc ^1H NMR see Results and Discussion ^{13}C NMR see Results and Discussion MS (direct inlet) *m/z* (rel int) 252 $[M+1]^+$ (18 8), 251 $[M]^+$ (42 1), 160 $[M-C_7H_7]^+$ (21 1), 146 $[M-C_7H_7-CH_2]^+$ (12 0), 131 $[M-C_8H_{10}N]^+$ (100), 103 $[M-C_9H_{10}NO]^+$ (23 3)

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LAUREQUINONE, A CYCLOLAURANE SESQUITERPENE FROM THE RED ALGA *LAURENCIA NIDIFICA*

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Key Word Index—*Laurencia nidifica*, Rhodomelaceae, red alga, cyclolaurane-type sesquiterpene, laurequinone

Abstract—From the red alga *Laurencia nidifica* a new sesquiterpene of cyclolaurane-type was isolated, and the structure elucidated by spectral analyses and chemical means

INTRODUCTION

Previous investigations of the red alga *Laurencia nidifica* (Rhodomelaceae, Rhodophyta) have revealed that this alga is a rich source of halogenated and nonhalogenated sesquiterpenes, and halogenated C_{15} nonterpenoid compounds [1–3] The present paper describes the isolation of a new cyclolaurane sesquiterpene, laurequinone (**1**) together with aplysin [4–6], debromoaplysin [4–6], laurinterol [7–10] and debromolaurinterol [7, 8]

RESULTS AND DISCUSSION

The fresh alga was extracted with acetone and the resulting extract was further extracted with ethyl acetate The oily extract was separated by column chromatography, TLC and HPLC to afford a new compound, laurequinone (**1**)

Laurequinone (**1**), $C_{15}H_{18}O_2$, pale yellow oil, $[\alpha]_D^{23}$ $-53\ 5°$ (c 0 91, $CHCl_3$) The presence of either a 2-methyl-5-alkyl-1,4-benzoquinone group or a 2-methyl-6-alkyl-