Pergamon

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AN AMIDE FROM FRUITS OF *PIPER NIGRUM*

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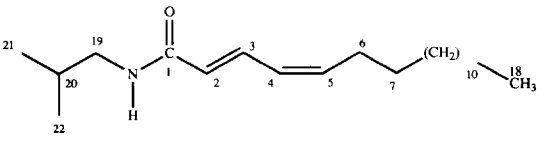
Key Word Index—*Piper nigrum*; Piperaceae; *N*-isobutylamide of octadeca-*trans*-2-*cis*-4-dienoic acid; piperine; 2D NMR.

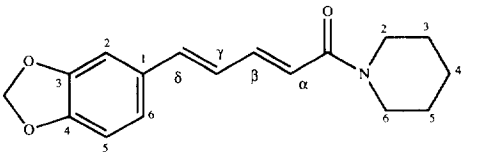
Abstract—A new amide, *N*-isobutyl amide of octadeca-*trans*-2-*cis*-4-dienoic acid has been isolated from the dried and crushed fruits of *Piper nigrum*, along with the known alkaloid, piperine. Complete assignments of the protons and carbons of the new amide and piperine have been made based on 2D NMR studies. © 1997 Elsevier Science Ltd. All rights reserved

INTRODUCTION

Piper nigrum, commonly known as black pepper, is a climbing perennial shrub. The fruit is dark green at first passing through orange-yellow to dull red when ripe. Black pepper is known to be acrid, pungent and hot. Internally, it is used as a stimulant and carminative, and induces secretion of bile. Externally, it is a rubefacient and stimulant to the skin. It is also prescribed for cholera, dyspepsia, flatulence, diarrhoea, various gastric ailments and for paralytic and arthritic disorders [1–4]. Several alkaloidal and non-alkaloidal constituents have been reported from time to time [5]. This report describes the isolation and structure elucidation of a new amide, pipericine (**1**), along with piperine (**2**). Structural determination is based on spectral methods. The ^{13}C NMR shifts of piperine agree well with those reported earlier [6].



Pipericine (1)



Piperine (2)

RESULTS AND DISCUSSION

The molecular formula of **1**, $C_{22}H_{41}NO$, was obtained through a combined application of EI mass spectrometry ($[M]^+$, *m/z* 335) and HREI-mass spectrometry (*m/z* 335.3317). The IR spectrum showed characteristic bands for -NH (3300, 3100 cm^{-1}), C = O (1654 cm^{-1}), C = C (1625 cm^{-1}) and CH = CH (998 and 965 cm^{-1}). The UV spectrum had absorption at 256.8 nm. These data indicated the presence of a *trans*-2-*cis*-4-dienamide system [7]. The ^1H NMR spectrum showed three methyls, one as a triplet at δ 0.85 (J = 7.20 Hz, H-18) and two as doublets at δ 0.93 (6H, J = 6.20 Hz, H-21, H-22), a broad singlet at δ 1.25 (16H) due to methylene protons, a one proton nine-line pattern at δ 1.79 for CH (H-20) of an isopropyl terminal, two one proton multiplets at δ 2.13 and 2.32 attributable to the allylic protons (H-6a and H-6b) and a triplet of two protons centred at δ 3.15 (H-19) accountable for -NH-CH_2-CH typical of isobutylamides [8], which changed to a doublet on shaking with D_2O. The ^1H NMR further displayed a one-proton doublet at δ 5.70 (J = 15.6 Hz, H-2), two one-proton double doublets at δ 7.10 (J = 15.6, 9.0 Hz, H-3) and 6.08 (J = 12.1, 9.0 Hz, H-4), and a one-proton multiplet at δ 5.95 (H-5). These assignments are based on the chemical shifts, coupling constants, proton-proton decoupling experiments and interactions observed in the COSY 45 plot, and agree well with the reported values of *trans*-2-*cis*-4-dienone systems [9]. In the HMQC plot, H-2, H-3, H-4 and H-5 showed their connectivities with C-2 (δ 120.0), C-3 (δ 139.0), C-4 (δ 124.0) and C-5 (δ 114.5), respectively.

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