

Communications to the Editor

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ENTADAMIDE A, A NEW SULFUR-CONTAINING AMIDE FROM *ENTADA PHASEOLOIDES* SEEDS¹⁾

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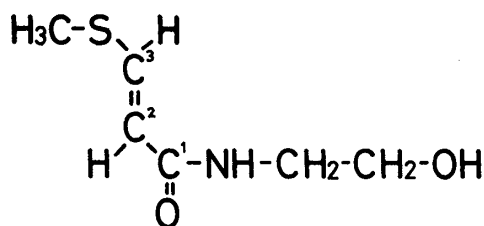
A new sulfur-containing compound (1), named Entadamide A, was isolated from the dry seeds of *Entada phaseoloides* Merr.. The structure of 1 was characterized by spectroscopic methods as *trans-N*-(2-hydroxyethyl)-3-methylthiopropenamide.

KEYWORDS — *Entada phaseoloides*; Leguminosae; seed; isolation; *trans-N*-(2-hydroxyethyl)-3-methylthiopropenamide; Entadamide A

Entada phaseoloides Merr. (Japanese name: Modama, Leguminosae) is a woody climber growing in the tropics. The seeds of this plants are utilizing as a folk medicine to treat skin diseases and as a soap plant in Thailand and other tropics. Barua *et al.* have shown that the seeds contain oleanolic acid and entagenic acid.²⁾

Our current interest in the chemical constituents in legumes³⁾ and in medicinal plants in Thailand led to the isolation of a new sulfur-containing compound (1) from the dry seed kernels of *E. phaseoloides* Merr.. We wish to report the isolation and the structural elucidation of this new natural product in this communication.

The seeds of *E. phaseoloides* Merr. were collected in June in the suburbs of Bangkok. A 75% aqueous ethanol extract of the air-dried powdered seed kernels (750 g) was concentrated *in vacuo*, saturated with anhydrous K₂CO₃ and then extracted with CH₂Cl₂ as described previously.⁴⁾ The basic fraction (806 mg, 0.12% of dry wt) obtained was subjected to Si-gel CC (Merck, type 60, 70-230 mesh, 3 x 20 cm) using Et₂O — MeOH — 28% NH₄OH (70 : 10 : 1, v/v) and CH₂Cl₂ — MeOH (8 : 1, v/v) as eluting solvents and 5 ml fractions were collected monitoring by UV-detection at 254 nm. The 1-rich fraction was further subjected to preparative TLC on Si-gel 60GF₂₅₄ (0.5 mm thick, Merck) and developed with the same solvents to yield a colorless sirup of 1 (186 mg, 0.025% of dry wt). 1 did not react with Dragendorff's reagent, but formed a violet-grey color with Iodoplatinate reagent.

Entadamide A (1)Table I. ^{13}C -NMR Spectral Data for 1

Carbon	Chemical shift (δ)
C-1	165.9 (s)
C-2	115.7 (d)
C-3	143.4 (d)
NH-CH ₂	42.6 (t)
CH ₂ -OH	62.2 (t)
S-CH ₃	14.7 (q)

The molecular formula of 1 was determined to be $\text{C}_6\text{H}_{11}\text{NO}_2\text{S}$ (M^+ , m/z 161.0554, calcd 161.0511) by high resolution MS(EI) measurement. The IR spectrum of 1 in CHCl_3 revealed bands at $3200\text{--}3500\text{ cm}^{-1}$ (br, NH and OH), 1640 cm^{-1} (C=O) and 1580 cm^{-1} (C=C). The ^{13}C -NMR spectrum in CDCl_3 indicated that the molecule was made up by one α,β -disubstituted olefine conjugated with a carbonyl group, one methyl, two methylene and one amide function, as shown in Table I. Considering the ^{13}C -NMR spectral data, all signals of the ^1H -NMR spectrum of 1 in CDCl_3 were assigned as follows: δ 7.64 ppm (1H, d, $J=14.5$ Hz, *trans*-CH=CHCO-), δ 5.68 ppm (1H, d, $J=14.5$ Hz, *trans*-CH=CHCO-), δ 6.20 ppm (1H, br, CONH, disappears on addition of D_2O), δ 3.69 ppm (2H, t, $J=5$ Hz, $-\text{OCH}_2\text{CH}_2\text{N}$), δ 3.20-3.55 ppm (3H, m, OH + $-\text{OCH}_2\text{CH}_2\text{N}$, becomes a 2H triplet ($J=5$ Hz) centered at δ 3.44 ppm on addition of D_2O), δ 2.32 ppm (3H, s, S-CH₃).

From the above results, the structure of Entadamide A (1) was elucidated to be *trans*-*N*-(2-hydroxyethyl)-3-methylthiopropenamide.

Chemical synthesis and screening tests of the biological activity of 1 are in progress.

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REFERENCES AND NOTES

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