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ISOLATION AND CHARACTERIZATION OF AN ALIPHATIC SATURATED ESTER FROM *Ipomoea carnea* LEAVES

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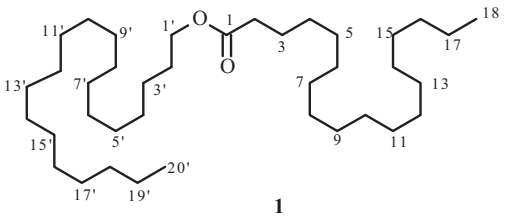
India has a rich traditional background in herbal medicines. Detailed accounts of remedies are given in texts like the Ayurveda and Rigveda. In the "Atharva Veda," one finds more varied use of drugs. Today there is a "Return to Nature" call in both advanced countries as well as countries like India. Plants still constitute one of the major raw materials for drugs for treating various ailments of humans, although there has been significant development in the field of synthetic drug chemistry and antibiotics [1]. Plants produce several straight-chain, branched-chain, aliphatic, and aromatic esters as secondary metabolites. *Ipomoea belongs* to the family Convolvulaceae and is represented by approximately 18 species in India [2]. One of the species is *Ipomoea carnea.* It is commonly called Besharam and Mahananda in Marathi. It is native to South America. *I. carnea*, locally known as Awier, occurs in tropical areas of the world such as North America, South America, Africa, Asia, and Australia [3]. It is available in ample amounts in all states of India due to its adaptation to Indian climatic conditions [4]. *I. carnea* is an exotic weed in Chattisghara, India, and only a few decades back it was introduced as Green Manure Crop [5]. It is a potential source of alternate feedback of biogas production and pulp for the paper industry [6–9]. There have been reports on the synergistic effect of insecticides with plant extracts of *I. carnea* against the malarial vector *Anopheles stephensi* [10]. *Metschnikowia drosophilae*, a new yeast species, was isolated from insects associated with flowers of *I. carnea* [11]. The presence of hexadecyl, octadecyl, and eicosyl esters of *p*-coumaric acid in the vine and root latex of sweetpotato [*Ipomoea batatas* (L.) Lam.] has been reported [12]. Compound **1** was isolated from an ethyl acetate extract prepared by a Soxhlet extractor using air shade-dried and pulverized leaves. Broad fractionation is performed using a column chromatographic technique. Compound **1** was isolated from the *n*-hexane–ethyl acetate (9.5:0.5) fraction, which was purified by repeated crystallization using a mixed solvent system. The structure was determined by spectral methods. Analysis reveals that compound **1** is octadecanoic acid eicosyl ester (eicosyl stearate). This compound was isolated for the first time from this plant source.

Compound **1**, a white amorphous solid, is purified by repeated crystallization using mixed solvents. It exhibits a sharp melting point at 132°C. The mass spectrum shows a molecular ion peak $[M + 1]^+$ at *m/z* 565 amu and a base peak at *m/z* 267 amu, in agreement with the molecular formula $C_{38}H_{76}O_2$.

IR spectrum (nujol, ν, cm^{-1}): 1747 (ester carbonyl), 1140, 849 (C-O stretching). PMR spectrum (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 0.87 (6H, t, J = 6.8, CH_3-18, 20′), 1.25 (62H, m, CH_2-4 to CH_2-17 and CH_2-3′ to CH_2-19′), 1.60 (4H, m, CH_2-3, 2′), 2.28 (2H, t, J = 7.6, CH_2-2), 4.05 (2H, t, J = 6.8, CH_2-1′). ^{13}C NMR spectrum (100 MHz, $CDCl_3$, δ, ppm, J/Hz): 168 (s , C-1), 64.43 (t, C-1′), 34.44 (t, C-2), 26.71 to 30.19 (C-3 to C-15 and C-2′ to C-17′), and 14.20 (C-18 and C-20′).



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The COSY spectrum indicates a sharp triplet at δ 4.0 (2H, t, J = 6.8 Hz, CH_2-1′), which shows connectivity to δ 1.60 (2H, m, CH_2-2′). Another sharp triplet at δ 2.28 (2H, t, J = 7.6 Hz, CH_2-2) indicates connectivity to δ 1.60 (2H, m, CH_2-3). The intense crowded cross peaks at δ 1.25 (methylene envelop) indicates connectivity to protons of other methylene protons. A visibly strong triplet at δ 0.87 (6H, t, J = 6.8 Hz, CH_2-18, and CH_2-20′) reveals connectivity to CH_2-17 and CH_2-19′ at δ 1.30, respectively, which merge in the methylene envelop. On the basis of spectral analysis and COSY experiments, compound **1** was established as a saturated straight-chain aliphatic ester, octadecanoic acid eicosyl ester.

The plant *Ipomoea carnea* was collected from the river side of Pune, Maharashtra, India. It was authenticated by comparison with the herbarium voucher specimen deposited at Botanical Survey of India, Pune, India. Its authentication number is E LICAI BSI/WC/Tech/2009/96.

Air shade dried, powdered leaves (200 g) were extracted using a Soxhlet extractor with solvent *n*-hexane followed by ethyl acetate, acetone, and ethanol. Entomological activity study was performed for all the crude extracts against two mosquito species *Aedes aegypti,* which is responsible for Dengue fever, and *Culex quinquefasciatus,* which is a vector of filariasis. Results of the experiment show that the ethyl acetate, acetone, and ethanol extracts were active against both mosquito species. Thus, bioassay-guided fractionation was initiated with the ethyl acetate extract using column chromatography.

Column Chromatography of Ethyl Acetate Extract. The crude ethyl extract (2.5 g) was broad fractionated using silica gel CC (60–120, 100 g). It was fractionated using polarity gradient solvents from *n*-hexane (100%), *n*-hexane–ethyl acetate (9.5:0.5), *n*-hexane–ethyl acetate (9:1), *n*-hexane–ethyl acetate (8:2), chloroform (100%), ethyl acetate (100%), acetone (100%), ethanol (100%), and methanol (100%). Fractions of 200 mL volume were collected. The progress of the column chromatographic separation was monitored by thin layer chromatography of the fractions. Fractions showing similar composition were combined to obtain nine fractions.

Purification and Characterization of Compound 1. In the *n*-hexane–ethyl acetate (9.5:0.5) fraction, a mixture of unidentified compounds along with compound **1** was detected. Efforts were made towards the separation and purification of the said fraction. Repeated crystallizations using mixed solvent systems were performed to yield compound **1** in pure form. The purified compound **1** was a white amorphous solid, a saturated long-chain ester. The structure was characterized by LC-MS, IR, ^{1}H NMR, ^{13}C NMR, DEPT, and COSY spectra. Compound **1** has been corroborated as a saturated straight-chain aliphatic ester, octadecanoic acid eicosyl ester. The compound is isolated for the first time from this plant source.

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