

Spectroscopic characterisation of a rare monoterpene from *Blumea mollis*

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Beginning with earlier studies¹ on the essential oil separated from different species of *Blumea*, this material has been the subject of many chemical²⁻⁵ and biological⁶ investigations. Chemical investigations of the oils of different *Blumea* species have afforded different terpenoids. Thus the oil of *Blumea eriantha* has a major constituent, d-carvo-tanacetone;² oil of *Blumea lacera* contains the cineol, d-fenchone and citral;³ oil of *Blumea malcolmii* yielded d-carvotanacetone and 1-tetrahydrocarvone;⁴ and the oil of *Blumea membranacea* contains 1,4-(8)-p-methadiene; δ -cadinene; 2,3-dimethoxy-p-cymene; 1,2-dimethyl-3-isopropenyl cyclopent-1-en-4-one; 5-hydroxy-p-cymene-2-oxybornylene; 2-methyl-5-isopropyl cyclopent-1-one carboxylic acid methyl ester; and citronellyl acetate.⁵ But this monoterpene has not been reported yet. Among the different species studied, the essential oil of *Blumea mollis*,⁷ DC represents a topic of interest due to the presence of chrysanthemone, a chief constituent for perfume material.

The present work was carried out, therefore, with the aim of separation of compound I in pure form (to prevent photo-oxidation) and its identification by physical and spectral data. Both TLC and GLC techniques were employed for the quantitative determination of percentage.

Experimental

A dark, brownish essential oil (0.034%) was obtained by steam distillation of fresh flowering plants of *Blumea mollis* DC (compositae),

widely distributed in the Malwa region near Ujjain in India.

Neutral oil was obtained after treatment of the oil with sodium hydroxide (5%) solution. The pet. ether (40-60°C) fraction of neutral oil yielded a TLC pure compound I by repeated column chromatography over alumina and silica gel. The relative percentage of compound I was calculated by TLC and quantitative GLC which shows 40% of the total oil. The remaining 60% constituents of this oil contain long chain aliphatic hydrocarbons: 2,3-dimethoxy-p-cymene 2,4,5-trimethoxy allylbenzene; caryophyllene-oxide; and 2-methyl-5-isopropyl cyclopentene carboxylic methyl ester, along with some unidentified residue.

Results and discussion

The physical constants of compound I are: n_D^{25} 1.4729, b.p. 70-75° C/0.6 m.m. (α_D^{26} - 87°C, molecular weight (M^+ , m/e 150) and elemental analysis yielded the molecular formula to be $C_{10}H_{14}O$.

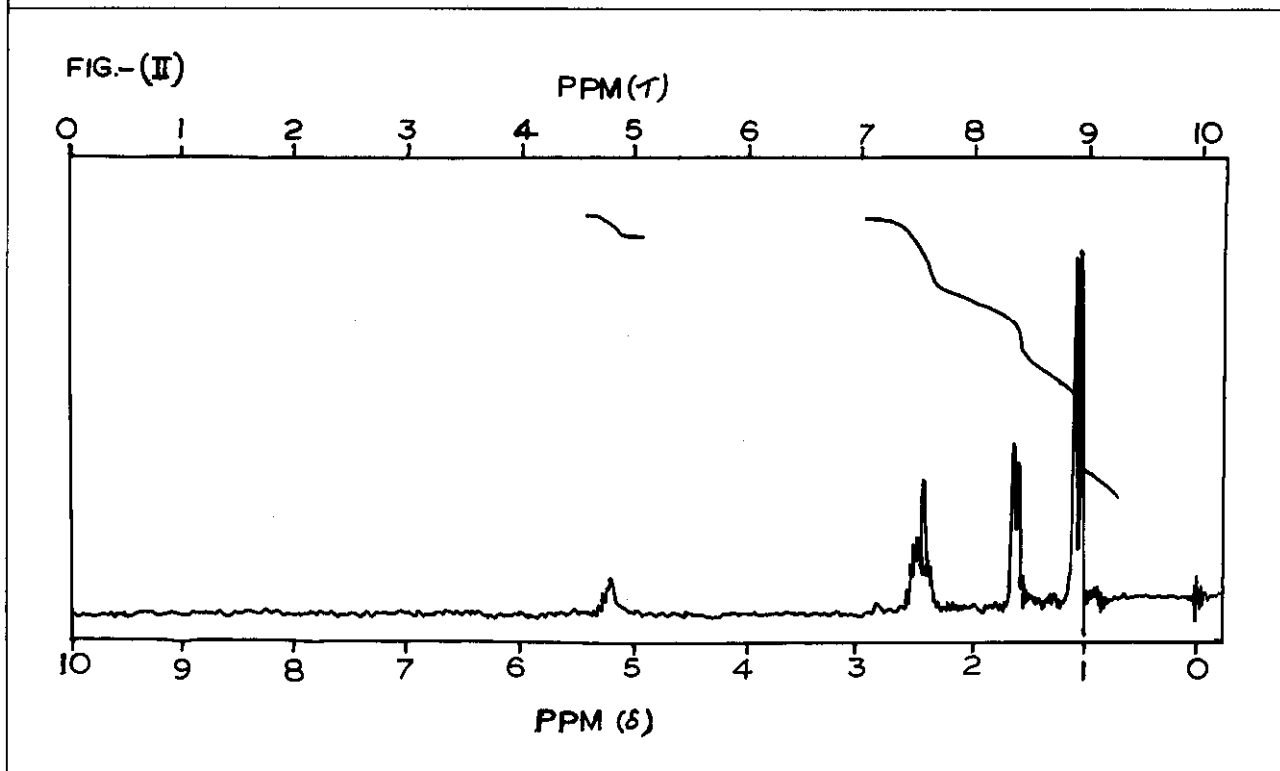
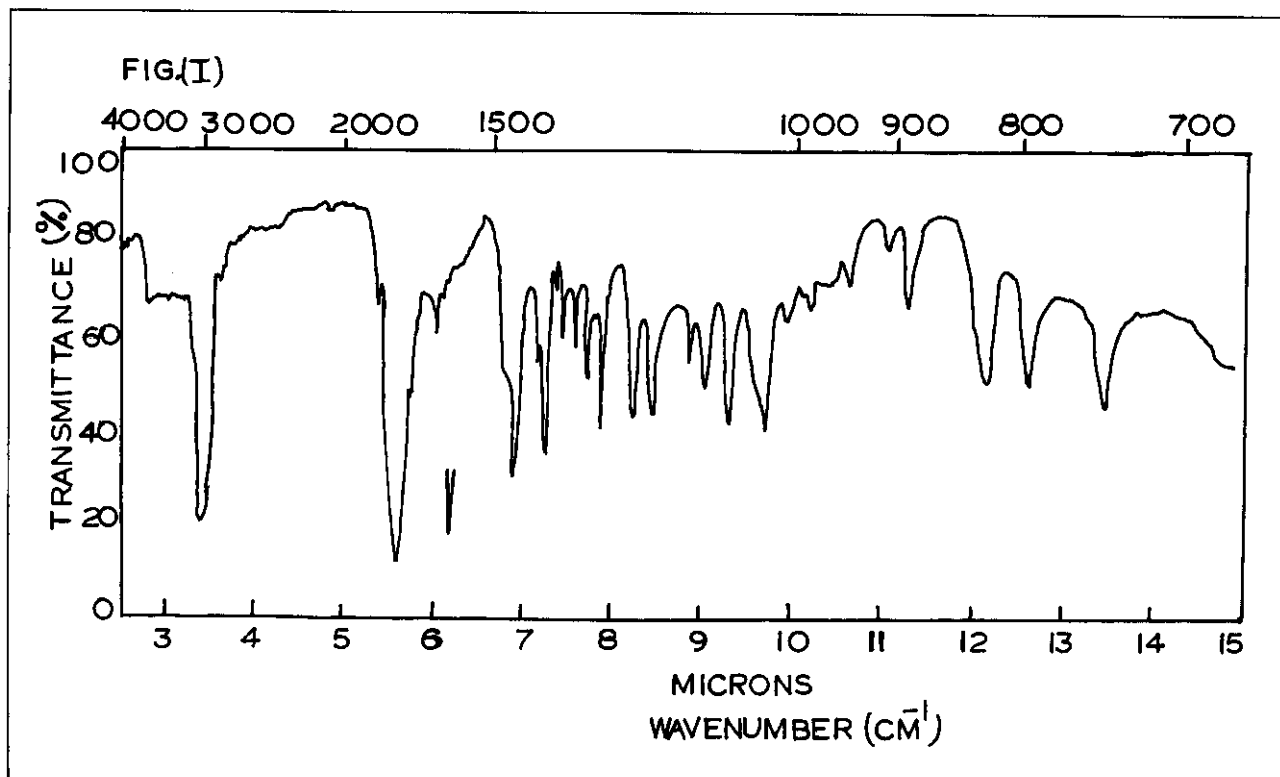
Its IR (fig. 1) absorption band at 2900 cm^{-1} (C-H stretching); 1780 cm^{-1}

(>C=O group, it may be present in cyclobutane ring);

1670, 821 cm^{-1}

(-C=CH-, linkage);

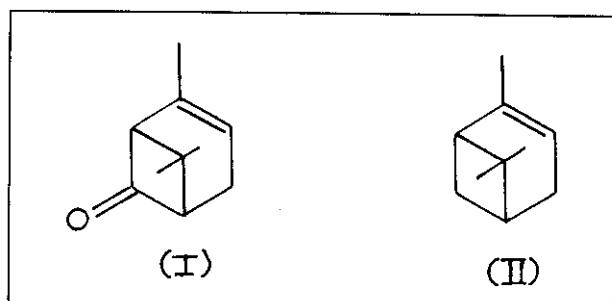
1450 cm^{-1} (—OCH₃ banding); 1380, 1340 cm^{-1} (a doublet, of isopropyl group), NMR spectrum (fig. 2) (CCl_4 , δ) is integration for fourteen pro-



integration for fourteen protons which indicates the bicyclic nature of the molecule with at least one double bond and a keto group in a four membered ring (by IR band at 1780 cm^{-1}). A

doublet ($J=1.8\text{ Hz}$) centered at $1.07\text{ }\delta$, of six protons due to gemdimethyl attached to the cyclobutane moiety. The chemical shift of the methyl group may be explained due to the pre-

sence of a double bond in the cyclohexane ring as in the α -pinene structure (II). The downfield shift of the other methyl groups can be explained due to the keto function in cyclobutane moiety. The methyl groups are deshielded due to the keto group. In structure II, the gem-dimethyl group appears quite separated at 0.84 δ and 1.27 δ . The downfield shift occurs in structure I. It further confirms that one methyl group is in the vicinity of the keto group. A quartet of



three protons at 1.65 δ is due to the allylic methyl group. This position and shape of signals is nearly the same as that of structure II. The one olefinic proton appears as a broad singlet at 5.20 δ , which is also the same as that in structure II, confirming that there is no keto group in a six-membered ring. The remaining four protons show a multiplet centered at 2.5 δ . It may be attributed to one allylic methylene group and also two methine protons at the bridge head. On the basis of the data cited, structure I of this compound is named chrysanthenone.^{8,9}

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