be explained as products of fatty acid, carbohydrate, and amino acid biodegradation. Mass spectral evidence was also obtained for the presence of a substituted indole (M^+ 175): on the basis of fragments at *m/e* 103, 132, and 160 and published spectra for other indoles [5], a tentative assignment of 3-ethyl-5-methoxyindole is suggested.

In earlier work on the components in the cotton bud essential oil that contributed to attraction, a mixture of β-bisabolol, β-carophyllene oxide, β-caryophyllene, α-pinene, and limonene effectively attracted boll weevils in laboratory bioassays [6]. Later, several mixtures of commercially available terpenoids proved to be nearly as attractive as extracts of cotton buds [7]. In view of the diversity of the compounds found in the air space volatiles, it was concluded that no individual chemical is likely to be attractive in itself.

EXPERIMENTAL

Air sampling procedures. Air was drawn by a vacuum pump through a 30-ml fritted glass filtering funnel that contained a 5·0 × 2·5 cm column of 60/80 mesh Chromosorb 102, a styrene–divinylbenzene polymer. The vacuum pump was placed outside the greenhouse cubicle to minimize contamination of the sample. Volatiles were desorbed by Soxhlet extraction for 24 hr with pentane; then solvent was reduced to a minimum for GLC and GLC–MS analysis. The pentane was doubly distilled before use; even so, analysis of solvent residue demonstrated the presence of traces of aromatic hydrocarbons, phthalates, phenols, and cresols. The Chromosorb 102 was also Soxhlet extracted with several changes of pentane for several days. After solvent extraction, the polymer beads were transferred to a gas chromatographic column (*ca* 15 mm diam), attached only at the inlet fitting in a GLC oven and purged at 180–200° overnight in a slow N_2 flow. The beads must be activated by heating each time before use.

Quantitative aspects of collection. In a typical air sampling period of 12 hr, *ca* 6000 l. of air were drawn through the polymer. GLC analysis of the pentane extract was performed on a 76 m × 0·8 mm capillary column coated with OV-17. The temperature was raised from 120 to 180° at 1°/m. The flow rate was 5 ml/min N_2. The total yield of volatiles was estimated by comparing the peak areas with those of an internal, standard hydrocarbon mixture. In this analysis, the 73 maxima observed had a total mass of 8·9 μg (1·5 ng of volatiles per liter of sampled air).

Analytical GLC–MS. Before analysis, combined concentrates from collections made over 1–2 weeks were fractionated on a Si gel-G TLC plate that was irrigated with 5% Et_2O in pentane. 4 fractions were obtained that consisted chiefly of the hydrocarbons and the oxygen-containing compounds. GLC–MS was obtained by introduction of the four TLC fractions from the OV-17 column into a Hewlett-Packard 5930 quadrupole mass spectrometer. Spectra were obtained at 70 eV. The gas chromatographic profile obtained with an FID was used to estimate the relative concentrations of the oil components. Material balance observations were made by peak triangulation and normalization to 100%. Peak identity was confirmed by comparison with standards where possible.

Mention of a proprietary product does not necessarily imply endorsement of the product by the U.S. Department of Agriculture.

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LIGNANS IN THE SEEDS OF *PIPER LONGUM**

C. P. DUTTA and NOTON BANERJEE
Department of Chemistry, University of Kalyani, Kalyani, Nadia, West Bengal, India
and
D. N. ROY
Faculty of Forestry, University of Toronto, Toronto, Canada

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Key Word Index—*Piper longum*; Piperaceae; seeds; sylvatin; lignan; (+)-Diaeudesmin and sesamin.

As a part of our studies on the genus *Piper*, we have now examined the seeds of *Piper longum* L. Earlier investigations [1–3] on the whole plant have led to the isolation of several alkaloids, isobutylamides and the lignan, sesamin.

The light petroleum ether (bp 40–60°) extract

of the seeds of the same plant upon careful chromatographic separation over Si gel furnished three crystalline compounds, sylvatin, sesamin and diaeudesmin. *Sylvatin*, $C_{24}H_{33}NO_3$ (M^+ 383), mp 114–115° $[\alpha]_D \pm 0$ ($CHCl_3$)[4] exhibited IR and NMR spectra suggestive of its identity which was confirmed by direct comparison (mmp, Co-TLC and mixed IR) with an authentic sample. *Sesamin*, $C_{20}H_{18}O_6$ (M^+ 354), mp 122–124°, $[\alpha]_D$ +78·4 ($CHCl_3$) was identical with an authentic sample (mmp, Co-TLC, mixed IR). *Diaeudesmin*, $C_{22}H_{26}O_6$ (M^+ 386), mp 153°, $[\alpha]_D$ +325° ($CHCl_3$); λ_{max}^{EtOH} 230 and 280 nm (log ϵ, 4·49, 3·54) exhibited characteristic IR bands at 1600, 1588, 1512, 1235, 1140 and 1085 cm^{-1} for aromatic and cyclic diethers, and the 100 MHz. NMR data [($CDCl_3$): δ 3·04–3·24 m (2H, C-1 and C-5 bridgehead protons), δ 4·90, *d*, *J* 5 (2H, C-2 and C-6 methine protons), δ 3·42–3·60 m, and δ 3·66, 3·78, *dd*, J 3 (4H, C-4 and C-8 methylene protons), δ 3·87 and 3·90, *s*, (3H each, two methoxyls), δ 6·90–6·98, *d*, *J* 8 (3H, aromatic protons)] showed it to be symmetrical. A two protons doublet around δ 4·90 together with the appearance of the methylene proton signals below δ 4·00 clearly revealed the presence of two diaxial aryl groups in the compound [5].

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ACYLATED BETACYANINS OF *PORTULACA OLERACEA*

FILIPPO IMPERATO

Istituto di Chimica Organica dell'Università di Catania, Catania, Italia

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Key Word Index—*Portulaca oleracea*; Portulacaceae; betacyanins; ferulic acid derivatives; betanidin and isobetanidin 5-cellobiosides.

Two red–violet pigments (Oleracin I and II) have been found in *Portulaca oleracea* L. [1]. In the present study, these pigments have been reinvestigated in more detail and found to be acylated betacyanins [2–7]. Alkaline hydrolysis of the total betacyanin fraction gave ferulic acid and two new pigments which were proved to be 5-*O*-β-cellobiosides of betanidin and isobetanidin.

The total betacyanin fraction was isolated by chromatography on Dowex 50 W-X2 resin. Column chromatography on polyamide yielded two red–violet bands (Oleracin I and II) which