Phytochemistry, 1975, Vol. 14, pp. 599–601. Pergamon Press. Printed in England.

THE ESSENTIAL OIL OF *DISTICHLIS SPICATA**

NARESH V. MODY, A. A. DE LA CRUZ† and D. HOWARD MILES‡

Department of Chemistry, Mississippi State University, Mississippi State, MS 39762

and

PAUL A. HEDIN

Boll Weevil Research Laboratory, United States Department of Agriculture, Mississippi State, MS 39762 U.S.A.

(*Received* 12 *May* 1974)

Key Word Index—*Distichilis spicata*: gramineae; essential oils; hydrocarbons; carbonyl compounds, phenols; *p*-tolualdehyde; phenyl acetaldehyde; carvacrol; benzyl alcohol; indole; dichloroethylbenzene.

The Mississippi salt marsh is an irregularly flooded estuary dominated by the needlerush, *Juncus roemerianus* (Juncaceae), the giant cordgrass *Spartina cynosuroides* (Graminae), *Scirpus americanus* (Cyperaceae), and *Distichlis spicata* (Gramineae) [1]. Studies by de la Cruz [2,3] on the ecology of Mississippi salt marshes include those on primary production and decomposition of *Distichlis spicata* and other salt marsh plants and their food value to marsh and estuarine animals. To our knowledge, there is no report of a detailed study of the organic constituents of *D. spicata*. This communication on the essential oil of *D. spicata* is a part [1,4–6] of a continuing chemo-ecological study of the Mississippi salt marsh.

An investigation of the essential oil of the marsh grass, *D. spicata*, by GC–MS resulted in the identification of 37 compounds that comprise 67·6% of the total oil. The structures for 18 aliphatic and aromatic hydrocarbons, five aldehydes, six alcohols, six phenols, indole and dichloroethylbenzene have been determined. *p*-Tolualdehyde, carvacrol, phenyl acetaldehyde and benzyl alcohol were the major components of the oil. Indole was the only heterocyclic compound found. Dichloroethylbenzene was the only unusual compound found in the oil. Halogenation of aromatic nuclei might be expected because of the growth of this plant in salt water and apparently does occur to some extent since tetrachlorobenzene was found in *J. roemerianus* [5]. It is also possible that these hydrocarbons could have arisen from polluted water since it is well known that plants can take up certain compounds and partially metabolize them. Due to the presence of dichloro-ethylbenzene and indole, other halogenated compounds and perhaps alkaloids can be anticipated.

This chemo-ecological study demonstrates that the four major species of marsh plants mentioned above have four constituents in common. These include *m*-xylene, *p*-xylene, benzaldehyde, and 2-furaldehyde. Some 14 compounds are common to at least three of the species. *D. spicata*, *J. roemerianus*, and *S. cynosuroides* contain naphthalene and methylnaphthalene. *n*-Nonane, *n*-decane, 3-ethyl-*o*-xylene, *n*-tridecane, *n*-tetradecane, *n*-pentadecane, *n*-hexadecane, *n*-heptadecane, phenyl acetaldehyde, *p*-tolualdehyde, phenol, and benzyl alcohol are common to *D. spicata*, *S. americanus*, and *S. cynosuroides*. Thus the environmental surroundings may play a role in contributing to the compounds present in plants indigenous to the Mississippi salt marsh.

* Part V in the series "Constituents of Marsh Grass". For Part IV, see ref. 1.

† Department of Zoology, Mississippi State University.

‡ To whom inquiries concerning this publication should be sent.

Table 1. Analysis of the essential oil in *Distichlis spicata*

Compound	I_K OV-17*	MS fragmentation†	%‡
Hydrocarbons			
n-Heptadecane	1700		1·2
n-Octadecane	1800		1·0
2-Ethyltoluene	1140		0·8
p-Xylene	1055		0·6
n-Hexadecane	1600		0·3
n-Decane	1010		0·3
3-Ethyl-*o*-xylene	1210		0·3
m-Xylene	1040		0·3
n-Pentadecane	1505		0·2
Methylnaphthalene	1475		0·2
n-Tetradecane	1400		0·2
Naphthalene	1390		0·2
o-Methylstyrene	1190		0·2
Isopropylbenzene	1120		0·2
n-Tridecane	1300		0·1
n-Undecane	1100		T§
Toluene	978		T§
n-Nonane	900		T§
Carbonyl compounds			
p-Tolualdehyde	1515		25·7
Phenylacetaldehyde	1250		4·3
(4-Methyl-pent-3-enyl)-cyclohexene-1-carboxaldehyde	1570		1·8
Benzaldehyde	1160		0·6
2-Furaldehyde	1062		T§
Alcohols, phenols and miscellaneous			
Carvacrol	1555		13·4
$C_{13}H_{18}O$		69,83,97,133,148,190	9·3
Benzyl alcohol	1260		2·8
Indole	1590		1·0
$C_7H_{12}O$		41,56,55,43,42,112	0·9
2-Phenylethanol	1290		0·7
$C_{10}H_{14}O$	—	119,135,150,107,91,150	0·4
Methoxyphenol	1310		0·3
$C_{12}H_{18}O$	—	119,178,91,107,41,178	0·2
Dichloroethylbenzene	—		0·1
Furfuryl Alcohol	—		T§
3-Hexene-1-ol	—		T§
$C_6H_{10}O$ 2° Alcohol	—	43,41,55,57,45,98	T§
Phenol	1230		T§

* Kovats [9].

† Mass fragmentation patterns of the majority of the compounds corresponded with those published [7,8]. The fragment ions for (4-methyl-pent-3-enyl) cyclohexane-1-carboxyaldehyde have been determined previously [1,5,6]. For the other compounds the five most intense fragment ion values (*m/e*) are given, arranged in order of decreasing relative abundance with the proposed parent ion presented sixth.

‡ Per cent total oil.

§ Trace.

EXPERIMENTAL

Isolation of the essential oil. Fresh marsh grass (1 kg) was harvested from St. Louis Bay Estuary, Mississippi, and stored at below 0° until it was chopped and steam-distilled in an all-glass system for *ca.* 3 h. The distillate was extracted with redistilled anhydrous Et_2O. The Et_2O was dried (Na_2SO_4) and the solvent removed under vacuum at 35° to give 0·2 g of oil. Yield = 0·02% [calc. on the basis of fresh plant].

Column chromatography. The essential oil was chromatographed on a 2 × 25 cm jacketed Florisil column which was cooled to 0° to prevent cracking. The column was eluted successively with 200 ml portions of pentane, 20% Et_2O in pentane, and 100% Et_2O. The separations were monitored by TLC, and the components were located by heating the developed plate after spraying with 3% vanillin in 0·5% conc H_2SO_4 in MeOH.

Analytical GC MS. The three fractions were introduced separately into a Hewlett Packard 5930 quadrupole mass spectrometer interfaced with a Hewlett Packard 5700A gas chromatograph from a 76 m × 0·76 mm capillary gas chromatographic column coated with OV-17. Carrier gas flow was 8·0 ml/min N_2. The GLC unit was programmed from 120 to 160° at 1°/min rate. The final temperature was maintained for 20 min. MS were obtained at 70 eV. Fragment ion values were compared with those Stenhagen *et al* [7], Cornu and Massot [8], and Miles *et al.* [1,5–6]. GLC retention times are presented as Kovats indices (I_K) [9]. Estimates of the per cent content of each component in the oil were made by peak triangulation of the maxima of the GLC profile trace.

REFERENCES

1. Mody, N. V., Parish, E. J., Bhattacharyya J., Miles, D. H. and Hedin, P. A., (1974) *Phytochemistry* In press.
2. de la Cruz, A. A., Gulf *Research Reports* In press.
3. Odum, E. P. and de la Cruz, A. A. (1963) *AIBS Bull.* 13, 39.
4. Bhattacharyya, J., Mody, N. V., Hedin, P. A. and Miles, D. H. (1974). *Tetrahedron Letters* submitted.
5. Miles, D. H., Mody, N. V., Minyard, J. P. and Hedin, P. A. (1973) *Phytochemistry* 12, 1399.
6. Mody, N. V., Bhattacharyya, J., Miles, D. H. and Hedin, P. A. (1974) *Phytochemistry* **13,** 1175.
7. Stenhagen, E., Abrahamsson, S. and McLafferty, F. W. eds. (1969). *Atlas of Mass Spectral Data.* Interscience Publisher, New York.
8. Cornu, A. and Massot, R. (1966). *Compilation of Mass Spectral Data*, Heyden, London.
9. Kovats, E. (1961). *Analyt. Chem.* 181, 351.