and locating the sapogenins with 50% H_2SO_4.[7] The keto-sapogenins were isolated by PLC on air dried silica gel G layers, 500 μm, with double development in cyclohexane–EtOAc–H_2O (600:400:1). A sample of the keto-sapogenin mixture was reduced by the Huang-Minlon modification of the Wolff-Kishner method.[13] Acetates of the compounds formed were prepared by refluxing the sapogenins with Ac_2O for 30 min and sapogenin trifluoroacetates were prepared by the method of Bennett and Heftmann.[7] The sapogenin acetates and trifluoroacetates were examined on activated silica gel G layers, 250 μm, using $CHCl_3$–toluene (9:1). The three keto-sapogenins were separated from each other by PLC on silica gel G layers, 500 μm, developing 4× in *n*-hexane–EtOAc (6:1).

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THE ESSENTIAL OIL IN *SCIRPUS AMERICANUS**

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Key Word Index—*Scirpus americanus*; Cyperaceae; essential oil; phenol; 2 phenyl ethanol; p-tolualdehyde; cuminyl alcohol.

The Mississippi salt marsh is an irregularly flooded estuary dominated by the needle-rush, *Juncus roemerianus* (Juncaceae), the giant cordgrass *Spartina cynosuroides* (Graminae), and *Scirpus americanus* (Cyperaceae). Preliminary studies by Odum[2] include those on primary production and decomposition of *Scirpus americanus*, and the food value of this species to marsh and estuarine organisms. To our knowledge, there is no report of a detailed study on the organic constituents of *S. americanus*. This communication on the essential oil of *S. americanus* is a part[3,4] of a continuing chemoecological study of the Mississippi salt marsh.

An investigation of the essential oil of the marsh grass, *S. americanus*, by combined GC–MS resulted in the identification of 40 compounds that comprise 78·8% of the total oil.

* Part IV in the series "Constituents of Marsh Grass". For Part III, see Ref. 1.
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The structures of 15 aliphatic and aromatic hydrocarbons, 9 aldehydes, 2 ketones, 12 alcohols, phenols and ethers, and benzothiazole are proposed. Phenol, 2-phenyl ethanol, *p*-tolualdehyde, cuminyl alcohol, (4-methyl-pent-3-enyl) cyclohexene-1-carboxaldehyde, 2,4-ditert-butylphenol and *n*-heptadecane are the major components of the oil. Benzothiazole was the only heterocyclic compound found.

EXPERIMENTAL

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

Column chromatography. The oil was chromatographed on a 2 × 25 cm jacketed Florisil column cooled with ice water to prevent column cracking. The column was eluted with 200 ml portions of pentane; 10% 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 5700 Å gas chromatograph from a 74 m × 0·75 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. The final temperature was maintained for 20 min. MS were obtained at 70 ev. Fragment

TABLE 1. ANALYSIS OF THE ESSENTIAL OIL IN *Scripus americanus*

Compound	I_KOV-17*	Fragmentation†	(%)‡
Hydrocarbons			
Toluene	978	R	Trace
m-Xylene	1040	R	0·4
p-Xylene	1055	R	0·2
n-Decane	1010	R	0·2
n-Propylbenzene	1090	R	3·2
Isopropylbenzene	1120	R	2·5
Trimethylbenzene	1130	R	3·2
3-Ethyl-*o*-xylene	1210	R	0·6
Indane	1230	R	0·1
Tetramethylbenzene	1240	R	Trace
n-Tridecane	1300	R	1·0
n-Tetradecane	1400	R	0·8
n-Pentadecane	1505	R	3·0
n-Hexadecane	1600	R	0·6
n-Heptadecane	1700	R	4·2
Carbonyl compounds			
C_6H_8O aldehyde	1030	43, 44, 41, 57, 55, 96	Trace
Benzaldehyde	1160	R	0·2
$C_7H_{14}O$ aldehyde	—	43, 41, 57, 55, 70, 114	1·0
2-Furaldehyde	1062	R	0·2
4-Methylcyclohexanone	1190	R	0·2
$C_8H_{12}O$ ketone	—	68, 41, 39, 43, 55, 124	Trace
Phenyl acetaldehyde	1250	R	1·8
p-Tolualdehyde	1515	R	16·1
Geranial	—	R	0·5
(4-Methyl-pent-3-enyl) cyclohexadiene-1-carboxaldehyde[3,4]	1540	R	2·5
(4-Methyl-pent-3-enyl) cyclohexene-1-carboxaldehyde[3]	1570	R	7·9
Alcohols, phenols and miscellaneous			
3-Hexene-1-ol	—	R	0·1
Phenol	1230	R	6·2

TABLE 1. *contd.*

Compound	I_KOV-17*	Fragmentation†	(%)‡
Benzyl alcohol	1262	R	0·2
o-Methoxyphenol	1312	R	2·7
2-Phenylethanol	1450	R	4·0
Cuminyl alcohol	1560	R	8·0
Butyl phenol	—	119, 150, 135, 91, 107, 150	0·1
$C_{10}H_{16}O$ phenol	—	43, 41, 69, 117, 107, 152	1·3
p-Tert-butylphenetole	1640	R	1·0
2,4-Ditert-butylphenol	1705	R	3·1
$C_{10}H_{14}O$	—	43, 41, 53, 81, 69, 150	Trace
$C_{10}H_{16}O$	—	41, 43, 67, 55, 81, 152	0·6
Benzothiazole	1475	R	1·1
$C_{13}H_{20}O$	—	55, 57, 163, 177, 79, 192	Trace

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† The five most intense fragment ion values (*m/e*) arranged in order of decreasing relative abundance with the proposed parent ion presented sixth: R means that the spectrum corresponded with literature values.[3–6]

‡ Percentage of total oil.

ion values were compared with literature values.[3–6] GLC retention times are presented as Kovats indices[7] (I_k). 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.

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CORN BUD ESSENTIAL OIL

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As a part of current studies of the essential oils of plants of commercial and economic importance,[1,2] the steam distillate of the whorl (bud) of corn, *Zea mays* L., in the mid-whorl stage was subjected to GC–MS analysis.

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