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

* Kovates, E. (1961) *Anal. Chem.* **181**, 351.

† 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.

[5] Stenhagen, E., Abrahamsson and McLafferty, F. W. eds. (1969) *Atlas of Mass Spectral Data.* Interscience, New York.

[6] Cornu, A. and Massot, R. (1966) *Compilation of Mass Spectral Data*, Heyden, London.

[7] Kovats, E. (1961) *Anal. Chem.* **181**, 351.

Phytochemistry, 1974, Vol. 13, pp. 2029 to 2032. Pergamon Press. Printed in England.

CORN BUD ESSENTIAL OIL

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(*Received* 2 *January* 1974)

Key Word Index—*Zea mays* L.; gramineae; corn; bud essential oil.

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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[1] Minyard, J. P., Tumlinson, J. H., Thompson, A. C. and Hedin, P. A. (1966) *J. Agr. Food Chem.* **14,** 332.

[2] Hanny, B. W., Thompson, A. C., Gueldner, R. C. and Hedin, P. A. (1973) *J. Agr. Food Chem.* **21,** 1004.

TABLE 1. PERCENTAGES OF CHEMICAL CLASSES FROM ESSENTIAL OIL OF CORN BUD

Class	Percentage 1971	Percentage 1972	Class	Percentage 1971	Percentage 1972
Hydrocarbons	4·6	4·8	Ketones	25·9	15·4
Alcohols	38·9	38·5	Esters	2·5	2·4
Aldehydes	17·3	11·2	Ethers	1·2	3·2

An investigation of the essential oil of the corn bud resulted in the identification of 59 components that comprise 92·1% of the oil. The structures of 13 aliphatic and aromatic hydrocarbons, 27 aldehydes and ketones (including isomers), 11 alcohols and ethers and four formate esters were proposed. The essential oil components were analyzed during each of two growing seasons (Table 1). Benzaldehyde (6·3%), 2-ethyl-l-cyclohexene-l-yl methyl ketone (8·3%), *beta*-ionone (10·7%), nerolidol (14·9%), and *beta*-bisabolol (9·0%) account for nearly 50% of the total essential oil (Table 2). These components were isolated, and further structural proof was obtained by IR and PMR analysis.[3]

EXPERIMENTAL

Isolation of corn bud oil. Twenty-five kg of corn whorls (var. "Dixie 55", a double cross of MP 305 × T101 and MP 311 × MP 307) was ground and immediately steam distilled in an all glass system. The essential oil was extracted from the distillate with dichloromethane and dried over anhyd. Na_2SO_4, and the solvent was removed under vacuum to give 3·6 g oil. Yield 0·014% (calc. on wet wt basis of corn whorl).

Column chromatography. The oil (0·98 g) was placed on a 2 × 25 cm calcium silicate (Florisil) (4) column in pentane. The hydrocarbons were eluted with pentane; the carbonyls, esters, and ethers with 2% ether in pentane;

TABLE 2. ANALYSIS OF THE CORN WHORL ESSENTIAL OIL

Compound*	I_k C20M*†	Fragmentation‡	Total (%)
n-Tridecane	1300		0·2
2,5-Dimethyldecane	1320		0·1
3-Methyltetradecane	1540	57, 43, 41, 55, 69; 212	0·5
7-Pentadecene	1655	57, 43, 71, 41, 55; 212	0·5
6-Pentadecene	1718	57, 43, 41, 55, 85; 212	0·1
Nonanal	1745		1·2
Hexadecadiene	1815	57, 43, 41, 55, 69; 222	0·2
Decanal	1827		1·6
Heptadecadiene	1910	57, 43, 41, 55, 69; 236	0·5
Undecanal	1975		0·4
1,2-Dimethylnaphthalene	1900		0·4
1,3-Dimethylnaphthalene	1940		0·3
2-Ethylnaphthalene	2006		0·1
1-Acetonaphthone	2058	170, 155, 161, 69, 115; 170	1·3
2-Acetonaphthone	2085	170, 155, 169, 105, 153; 170	2·7
1-Methyl-2-acetonaphthone	2148	155, 170, 169, 153, 152; 184	0·1
2,3-Dimethoxybenzaldehyde	2240		0·7
1,1-Diphenylethane	2365		0·4
Tetradecenal	2410	44, 195, 165, 43, 210; 210	0·8
Tetradecanal	2440	44, 195, 43, 165, 57; 210	0·4
Decenyl formate	2510	184, 138, 152, 92, 79; 184	0·9
Phenanthrene	2560	5, 7	1·1
Undecyl formate	2510	198, 44, 199, 43, 165; 198	0·8
Undecyl formate	2620	198, 97, 165, 43, 199; 198	0·5
2,2,6-Trimethylcyclohexan-1-one	1290	6	0·7

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[3] MINYARD, J. P., THOMPSON, A. C. and HEDIN, P. A. (1968) *J. Org. Chem.* **33**, 909.

TABLE 2. *cont.*

Compound*	I_k C20M*†	Fragmentation‡	Total (%)
Propoxyanisole	1555	41, 107, 81, 151, 95; 166	1·2
1-(3-Methyl-2-furyl)propane or Ethyl 3-methyl-2-furyl ketone	1450	6	2·8
Benzaldehyde	1538	5, 7	6·3
2-Ethyl-1-cyclohexen-1-yl methyl ketone	1618	137, 152, 109, 123, 67; 152	8·3
p-Tolylacetaldehyde	1670	134, 91, 105, 119, 133; 134	0·9
3-Phenylaldehyde	1677	7	1·4
$C_{12}H_{18}O$ Ketone	1788	28, 43, 41, 69, 53; 178	4·2
Ethylisopropyl-cyclohexadienyl ketone	1900	43, 41, 79, 91, 107; 178	0·4
Vinylethylphenethyl ketone	1925	41, 147, 91, 43, 119; 174	0·6
Phenylacetaldehyde	1645	6	0·7
trans iso-1-Menth-5-en-2-ol	1827	43, 121, 93, 41, 91; 154	0·4
Cubenenol	2080	161, 41, 105, 119, 93; 222	0·5
β-Ionone	1908		10·7
Cyclohexaneethanol	1470		10·7
2-Ethylcyclohexanol	1525		1·9
Citronellol	1630		1·9
Acyclic C_{10} alcohol	1670	81, 41, 91, 79, 55; 154	0·9
Acyclic C_{10} alcohol	1775	41, 43, 55, 69, 93; 154	0·4
Menthol	1555		2·8
Benzyl formate	1705		0·3
Sabinol	1750		0·6
C_{15} Alcohol	1863	123, 43, 41, 55, 69; 226	2·5
2-Thiopheneacetaldehyde	1926	43, 55, 41, 84, 39; 126	0·4
Methyl 2-thienyl ketone	2020	83, 111, 43, 55, 41; 126	1·0
5,6-Dihydro-2-pyran-2-carboxaldehyde	1705	83, 55, 57, 84, 112; 112	0·4
Methyl-2-pyran-2-carboxaldehyde	1788	69, 41, 86, 71, 39; 128	0·4
2,3-Dihydro-2-pyran-2-carboxaldehyde	2050	28, 83, 55, 44, 111; 112	0·3
2,3-Dihydro-2-pyran-2-carboxaldehyde	2150	43, 111, 109, 41, 137; 180	1·7
Nerolidol	2055		14·5
	2155		9·0
	1110		0·2
Indole	1250		0·4
3-Methylindole	1510		0·7
Benzothiazole	1340		2·6
Coumarin	1590		0·2

* Compounds preceded by parenthesis: insufficient data for assignment of isomer.

† KOVATS, E. SZ. (1961) *Analyt. Chem.* **181,** 351.

‡ The five most intense fragment ion values (*m/e*) arranged in order of decreasing relative abundance with the proposed parent ion presented 6th: Fragmentation data for other components is given in Refs. 3, 5–7.

and the alcohols with 10% ether in pentane. 200 ml of each solvent mixture was used. Elution progress was monitored by silica gel TLC and the components were located by heating the developed plate after spraying with 3% vanillin in 0·5% concn H_5SO_4 in MeOH.

Analytical GC–MS. Method 1. Each fraction from the calcium silicate column was introduced via a Watson–Biemann separator from a 15·24 × 0·25 m Scot silanized column coated with 20% carbowax 20 M. Carrier gas flow was 1·0 ml/min. N_2, column temp. manually programed from 130 to 150°. The mass spectrometer was a double focusing Perkin–Elmer 270. Method 2. Components were introduced into a Hewlett–Packard 5930[4] quadrapole mass spectrometer from a 76·2 m × 0·076 cm capillary column coated with OV-17. The GLC unit was temperature programed from 120 to 160° at 1°/min; the final temp. was maintained for 60 min. Mas spectra were obtained at 70 eV. Material balance measurements were obtained by peak triangulation and normalization to 100%.

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[4] Mention of a proprietary product in this paper does not constitute an endorsement of this product by the U. S. Department of Agriculture.

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