Identification of Sex-Pheromone Components of the Podborer, *Matsumuraeses falcana* (WALSHINGHAM) (Lepidoptera: Tortricidae)¹

Sadao WAKAMURA

*Laboratory of Applied Entomology, Shikoku National Agricultural Experiment Station,
Zentsuji, Kagawa 765, Japan*

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Analyses of female secretions of *Matsumuraeses falcana* by means of GC-MS equipped with a glass capillary column showed the presence of ten aliphatic acetates. Dodecyl acetate was the main component in the extracts at amounts of about 13 ng/female. (*E*)-8-Dodecenyl acetate and (*E,E*)-8, 10-dodecadienyl acetate were present to the extent of 27.8% and 23.3% of the main component, respectively. Minor components were identified as (*E*)-10-dodecenyl acetate (2.1%), (*E,Z*)-7, 9-dodecadienyl acetate (6.6%), (*E,E*)-7, 9-dodecadienyl acetate (2.7%), (*E,Z*)-8, 10-dodecadienyl acetate (2.1%), tetradecyl acetate (0.8%), (*E*)-10-tetradecenyl acetate (0.4%) and octadecyl acetate (tr). Bioassay revealed that both (*E*)-8-dodecenyl acetate and (*E,E*)-8, 10-dodecadienyl acetate were essential to show sex-attractive and sex-stimulative activities to the male moths.

INTRODUCTION

In Japan, there are five species of the genus *Matsumuraeses*: *M. phaseoli*, *M. azukivora*, *M. falcana*, *M. ussuriensis*, and *M. aeses*; all except *M. azukivora* injure soybean crops (OKU et al., 1983). Among these species, *M. falcana* predominates and causes serious injury to soybean crops throughout Japan (OKU et al., 1983). This species' similarity to other species makes it difficult to identify without detailed morphological examination, so that it has often been confused, especially with *M. phaseoli* (ISHIKURA et al., 1952; OKU et al., 1983).

This paper describes the sex-pheromone components of *M. falcana* females and their activities. They will be useful in determining the abundance and distribution of this species and for studying the behavior of male moths.

MATERIALS AND METHODS

Insect. Pupae of *M. falcana* were obtained from a successive culture as described by WAKAMURA and KOZAI (1984), which was maintained on a 16-hr light-8-hr dark cycle at a temperature of $23 \pm 1^\circ\text{C}$ throughout all stages. The insects were sexed as pupae and placed in separate containers. After emergence, about 20 moths were kept in a 380-ml

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glass pot with a damp piece of absorbent cotton.

Observation of mating behavior. Each pair of moths was placed in a 330-ml clear plastic pot for mating observation. A dim red light permitted viewing during the dark period. To determine the timing of the start of mating, observations were made at 15-min intervals during the entire dark period, and the number of mating pairs was recorded. The calling behavior of the female was noted under the same conditions; females were regarded as calling when they exposed their ovipositor.

Bioassay. Both sex-attractive and sex-stimulative activities to male moths were examined. The attractive activity was evaluated using field traps. Several water-pan traps (30 × 24 × 15 (h) cm, Takeda Chem. Ind., Ltd.) were set 1 m above the ground at 1-m intervals. A cotton ball wrapped with tetron-gauze and containing test materials was placed in each trap. Then, 100–120 males which were marked with a small spot of an oily dye on their forewings were released. Tests were conducted when the response of males to the pheromone reached the maximum level at night.

Sex-stimulative activity was examined according to the method described by KARLSON and BUTENANDT (1959). For assay, the substance was dissolved in ether; a glass rod was moistened with this solution and put into a 330-ml clear pot in which five 5-day-old males were placed. The number of males showed typical precopulatory behavior, including the mating dance, was recorded. Each concentration was evaluated on about 30 males.

Preparation of extracts. Extracts were prepared from 5-day-old females within a 2-hr period when calling activity reached maximum. About 100 females were dipped into 5 ml of dichloromethane for 5 min. Female bodies were rinsed twice using another 5 ml of solvent and the rinsings were added to the extract. The accumulated extract was stored at -20°C .

Chromatography. Column chromatography on Florisil (100–200 mesh, Floridin Co.) was carried out according to a class-separating method for lipids (CARROLL, 1961). Hexane, and 5, 15, 25, and 50% ether in hexane were used successively as eluting solvent. A column of silicic acid (Wakogel C-200, 100–200 mesh, Wako Pure Chem. Ind., Ltd.) impregnated with AgNO_3 (AgNO_3 : 16.7%) was used for the further separation. The eluting solvents were hexane, 1, 2, 3, 4, 5, 7, and 10% ether in hexane and ether.

For a certain separation a high performance liquid chromatograph (HPLC, Yanaco model L-2000) was used. It was equipped with a column (4 mm i.d., 50 cm long) which was packed with porous polymer beads of polystyrene gel (Yanaco gel 5510) for separating acetates by their carbon-chain lengths. As eluting solvent, 15% tetrahydrofuran (THF) in methanol was flowed at a rate of 0.43 ml/min. Substances were dissolved in ethanol and injected through an injector, Rheodyne model 7125. Acetates eluted were detected by a UV monitor, Yanaco model M-215, at the wavelength of 235 nm.

Gas chromatograph-mass spectrometer-data analysis system (GC-MS-COM). Mass spectra were measured with a mass spectrometer, JEOL model JMS-D300, combined with a gas chromatograph, JEOL model JGC-20KP, and a computer, JEOL JMS-2000 data analysis system. The ionization voltage was 70 eV and temperatures of the ionization chamber and enricher were 175°C and 200°C , respectively. A glass column of 2-mm i.d. and 1-m length was packed with 10% Thermon 1000 on Chromosorb W AW DMCS (100–120 mesh).

A SCOT column of 0.28-mm i.d. and 30-m length coated with Silar-10C was used for the separation of the geometrical isomers. The sample was injected in a splitless mode

at 200°C when the column was maintained at 50°C; then it was heated to 150°C at a rate of 5°C/min. Compounds were detected by mass fragmentographical technique for some characteristic ions. In certain analyses, dodecyl, tetradecyl, and hexadecyl acetates (12, 14, and 16:Ac) were added as references for the calculation of retention values relative to the straight-chain acetate [equivalent chain length (ECL) values].

Diimide reduction. A polyenyl compound was partially hydrogenated according to the method described by YAMAOKA et al. (1976) in order to estimate both positional and geometrical isomerism. To the solution of a polyenyl compound concentrated to about 20 μ l in a 0.5 ml taper-bottomed vial, 50 μ l of hydrazine solution and 50 μ l of hydrogen peroxide solution were added [N_2H_4 : 1.5 ml of hydrazine hydrate in 50 ml of ethanol, H_2O_2 : 0.2 ml of 31% hydrogen peroxide in 50 ml of ethanol]. The mixture was maintained at 50°C to accelerate the hydrogenation and monitored using a GC at about 1-hr intervals. When the amounts of the saturated and each of the mono-unsaturated compounds were almost equal (about 4 hr), the mixture was extracted with 0.4 ml of hexane or cooled to -20°C to stop the hydrogenation. The extract or the cooled reaction mixture was analyzed by GC-MS equipped with a glass capillary column.

Chemicals. Dodecyl, tetradecyl, and hexadecyl acetates were respectively synthesized by acetylation of the corresponding alcohols. (Z)-8-, (Z)-9-, and (Z)-10-dodecenyl acetates (Z8-, Z9-, Z10-12:Ac) and (Z)-10-tetradecenyl acetate (Z10-14:Ac) were provided by Drs. M. HORIIKE and C. HIRANO of Kochi University. (Z)-7-Dodecenyl acetate (Z7-12:Ac) was obtained from the Farchan Chemical Company. (E)-7-, (E)-8-, (E)-9-, and (E)-10-dodecenyl acetates and (E)-10-tetradecenyl acetate were synthesized from the corresponding (Z)-isomers by *cis-trans* isomerization reaction (SGOUTAS and KUMMEROW, 1969). Each of these monoenyl acetates was purified by column chromatography on silicic acid containing 16.7% AgNO_3 . The purified compounds were more than 99.9% pure with respect to positional and geometrical isomers.

The geometrical isomers of 7,9- and 8, 10-dodecadienyl acetates, originally supplied by Dr. H. ARN, were provided to the author by Dr. Y. TAMAKI and Mr. H. SUGIE of the National Institute of Agro-Environmental Sciences. They were also more than 99.9% pure, except E8Z10-12:Ac which contained 2.5% of E8E10-12:Ac, and Z8E10-12:Ac which contained an almost equal amount of E8E10-12:Ac and a small amount of E8Z10-12:Ac.

All the solvents were distilled immediately before use.

RESULTS

Mating and calling behavior

Mating usually started 8 hr after the beginning of the dark period (Fig. 1); the mean duration was 34 min ($N=81$). Calling of females was maximally observed 8.5 to 10 hr after the beginning of the dark period (Fig. 1). The rate of calling reached the maximum level 2 days after insect emergence.

Identification of the pheromone candidates

The accumulated extracts of 1,000 females were fractionated by column chromatography on 5 g of Florisil. The attractive component was eluted with 5% ether in hexane.

From a GC-MS analysis using 100 FE [FE: female equivalent] of the active fraction,

Fig. 1. Timing of mating and calling behavior of *M. falcana*. A: number of pairs whose mating was first recorded from observations every 15 min. (3 to 5-day-old, $N=81$). B: percentage of females calling, calculated from observations every 15 min. (3 to 5-day-old, $N=90$).

Fig. 2. Reconstructed ion chromatogram (RIC) of the 5%-ether-in-hexane fraction from Florisil column chromatography. GC: 10% Thermon 1000, 1 m $\times$ 2 mm ID, 50°C to 200°C at 5°C/min, MS: EI 70 eV, chamber at 175°C, Enricher: 200°C.

5 acetates were found (Fig. 2, peaks A to E). These were designated as dodecyl acetate (peak A), dodecenyl acetates (peaks B and C) and dodecadienyl acetates (peaks D and E). This fraction was further fractionated by column chromatography on 5.5 g of $\text{AgNO}_3\text{-SiO}_2$. The acetates were separated into the following groups:

1. *Saturated acetates*

Component A and two other minor components, F and H, were eluted with 2% ether in hexane. These were identified as 12:Ac, 14:Ac and 18:Ac, respectively, from their mass spectra and retention values.

2. *Mono-unsaturated acetates*

Components B and C and a minor component G were eluted with 4% ether in hexane.

As for the positional isomers of dodecenyl acetate, HORIIKE and HIRANO (1982) stated that they were easily distinguishable by their fragment-ion patterns. Thus, components B and C were designated as 8- and 10-dodecenyl acetate, respectively. Their calculated ECL values (B: 12.36, C: 12.61) corresponded to those of E8-12:Ac (12.39) and E10-12:Ac (12.61), respectively. Therefore, components B and C were respectively identified as E8-12:Ac and E10-12:Ac.

The isolated amount of component G was less than 50 ng which was too small for ozonolysis, so that another 5,000 FE of extract was used for identification. A fraction of 4% ether in hexane from the $\text{AgNO}_3\text{-SiO}_2$ column was further purified using HPLC equipped with a column packed with porous polymer beads of polystyrene gel. About 200 ng of component G was obtained as more than 99% pure material. Ozonolysis of about 100 ng yielded 10-acetoxydecanal. Mass spectra of component G was identical to that of authentic E10-14:Ac; the calculated ECL value (14.32) was similar to that for E10-14:Ac (14.33). Thus component G was identified as E10-14:Ac.

3. *Di-unsaturated acetates*

The dodecadienyl acetates (components E and D) were respectively eluted with 7% and 10% ether in hexane, but they were accompanied by their geometrical isomers, components E' and D', to the extent of ca. 10% and 20%, respectively.

Double bond isomerism of dienyl acetate was revealed by the following procedure: Isolated material (ca. 1 μg) was partially hydrogenated by treatment with diimide to yield a mixture of saturated, mono-unsaturated, and non-reacted dienyl compounds. The mixture was then analyzed by GC-MS to identify the saturated and mono-unsaturated compounds. As no isomerization reaction occurred during the diimide reduction (PRIVETT and NICKELL, 1966), these results permitted reconstruction of the original structure.

The diimide reduction of component E and a minor component E' yielded 12:Ac, E8-12:Ac, E10-12:Ac and Z10-12:Ac [minor, ECL: 12.95 (authentic: 12.96)], which indicated that component E was E8E10-12:Ac and that one of the double bonds of component E' was Z10. The calculated ECL value for component E was 14.42 which was similar to that of E8E10-12:Ac (14.43), and mass spectra were also identical to those of authentic 8, 10-12:Ac isomers. Thus component E was identified as E8E10-12:Ac.

The mass spectra and calculated ECL value (14.64) of component E' were identical to those of E8Z10-12:Ac (ECL:12.64); therefore, component E' was E8Z10-12:Ac.

For the identification of component D and D' another 5,000FE were needed because the previous amount was insufficient for diimide reduction. The partial hydrogenation

yielded 12:Ac, E7-12:Ac [ECL: 12.35 (authentic: 12.36)], Z9-12:Ac [12.67 (12.65)], and E9-12:Ac [minor, 12.47 (12.50)], which indicated that component D was E7Z9-12:Ac and that one of the double bonds of component D' was E9. The calculated ECL value for component D (14.49) was the same as that of E7Z9-12:Ac (ECL: 14.48) and mass spectra were also identical to those of 7, 9-12:Ac isomers. Thus component D was identified as E7Z9-12:Ac.

The mass spectra of component D' were identical to those of 7,9-12:Ac isomers, and the ECL value (14.64) was similar to that of E7E9-isomer (14.63). Therefore, component D' was identified as E7E9-12:Ac.

The components identified and their amounts found in female extracts are shown in Table 1. No other compound with a chemical structure similar to moth pheromones,

Table 1. Components identified in secretions of female *Matsumuraeses falcana*

	Component	ng/female	%
A	Dodecyl acetate	13.3	100
B	(<i>E</i>)-8-Dodecenyl acetate	3.7	27.8
C	(<i>E</i>)-10-Dodecenyl acetate	0.29	2.1
D	(<i>E,Z</i>)-7, 9-Dodecadienyl acetate	0.88	6.6
D'	(<i>E,E</i>)-7, 9-Dodecadienyl acetate	0.36	2.7
E	(<i>E,E</i>)-8, 10-Dodecadienyl acetate	3.1	23.3
E'	(<i>E,Z</i>)-8, 10-Dodecadienyl acetate	0.28	2.1
F	Tetradecyl acetate	0.1	0.8
G	(<i>E</i>)-10-Tetradecenyl acetate	0.05	0.4
H	Octadecyl acetate	tr	—
	Total	22.1

Fig. 3. Response of males exposed to extracts of virgin females and mixtures of synthetic components. VF: extracts of virgin females, MX: mixture of all C₁₂-components at a natural ratio, BE: binary mixture of E8-12:Ac and E8E10-12:Ac at a natural ratio.

such as hydrocarbon, aldehyde, ketone, or alcohol with a straight carbon-chain, was detected throughout the analyses, including the fractions without activity.

Sex-stimulative activity

Since the total amount of the components without C₁₂-acetate elements was less than 1%, the activity of the mixture of C₁₂-acetates at the natural ratio (Table 1) was evaluated as a sex-stimulant. This showed potent stimulative activity with the threshold from 10⁻³ to 10⁻⁴ FE/ml, which was almost the same level as that of the virgin-female extracts (Fig. 3, MX, VF). Therefore, the lack of C₁₄- or C₁₈- acetate components should not affect the activity.

To determine which component(s) was essential for strong activity, mixtures which lacked one of the components were examined. Mixture activity was apparently reduced only when E8-12:Ac or E8E10-12:Ac was lacking (Fig. 4). The binary mixture of

Fig. 4. Response of males exposed to synthetic C₁₂-components. MX: mixture of all C₁₂-components at a natural ratio. -A to -E': mixtures lacking the component indicated by the letter.

Table 2. Sex attractive activity of C₁₂-acetate mixtures lacking one component

Component lacking	No. of males caught (mean $\pm$ S.E.)
A 12:Ac	9.3 $\pm$ 2.2
B E8-12:Ac	0.3 $\pm$ 0.3
C E10-12:Ac	11.3 $\pm$ 2.9
D E7Z9-12:Ac	19.0 $\pm$ 7.6
D' E7E9-12:Ac	10.3 $\pm$ 4.8
E E8E10-12:Ac	0
E' E8Z10-12:Ac	13.7 $\pm$ 4.9
Mixture	13.6 $\pm$ 7.2
Solvent	0

Tests were repeated 3 times during the period from May 14 to June 6, 1984.

Table 3. Sex attractive activity of E8-12:Ac and E8E10-12:Ac

Lure	No. of males caught (mean $\pm$ S.E.)
E8-12:Ac 34 ng	0
E8E10-12:Ac 30 ng	0
E8-12:Ac 34 ng + E8E10-12:Ac 30 ng	19.7 $\pm$ 0.9
Mixture of C ₁₂ -acetates	27.0 $\pm$ 13.1
Solvent	0

Tests were repeated 3 times from June 10 to 14, 1984.

E8-12:Ac and E8E10-12:Ac showed almost the same activity as the C₁₂-acetate mixture (Fig. 3, BE, MX).

Attractive activity

The C₁₂-acetate mixture showed strong attractive activity, but this disappeared when the mixture lacked E8-12:Ac or E8E10-12:Ac (Table 2). The binary mixture of E8-12:Ac and E8E10-12:Ac was as attractive as the C₁₂-acetate mixture (Table 3).

DISCUSSION

Ten components were identified as candidates of the sex-pheromone system in *M. falcana* and at least two of them, E8-12:Ac and E8E10-12:Ac, were essential for males to release precopulatory behavior including flight orientation and the mating dance.

These compounds have been identified in several lepidopterous insects. E8-12:Ac was found in female secretions of *Cryptophlebia leucotreta* as an attractive main component (PERSOONS et al., 1976) and *Grapholitha molesta* as a secondary component (CARDÉ et al., 1979), and E8E10-12:Ac was in *Hedya nubiferana* (FREROT et al., 1979), *Rhyacionia rigidana* (HILL et al., 1976), *Cydia nigricana* (GREENWAY and WALL, 1980), and *Melissopus latiferreanus* (DAVIS et al., 1984), as a main component of attraction. E7Z9-12:Ac has been known as a sex-attractive pheromone in *Lobesia botrana* (ROELOFS et al., 1973). All of these species including *M. falcana* are taxonomically classified in the subfamily Olethreutinae, so that their components should be characteristic of this subfamily.

12:Ac has been found in several tortricids such as *Adoxophyes* sp. (TAMAKI et al., 1979), *Archipus argyrospilus* (ROELOFS et al., 1974), *Argyrotaenia velutinana* (ROELOFS et al., 1975), *Cnephasia pumicana* (BIWER et al., 1977), *Homona coffearia* (KOCHANSKY et al., 1978), *Hedya nubiferana* (FREROT et al., 1979), and *Rhyacionia buoliana* (SMITH et al., 1974). 12:Ac is an attractive component in *H. coffearia*, a synergist in *A. velutinana*, *C. pumicana* and *H. nubiferana* and an inhibitor in *R. buoliana*.

E8Z10-12:Ac has been identified as a main component from *M. latiffereanus* (DAVIS et al., 1984). Both E8Z10-12:Ac and E7E9-12:Ac were found in the extract of female secretions of *M. falcana*, but these two components might be produced on the female body or on scales from the corresponding geometrical isomers as a result of a possible isomerization reaction as reported by IDESES et al. (1982) and DAVIS et al. (1984).

In Tortricidae, 14:Ac has been found in the secretions of *Adoxophyes* sp. as a minor component of which the function was not known (TAMAKI et al., 1979), and in *Archippus breviplicanus* as a weak synergist for male attraction (SUGIE et al., 1977).

The functions of the sex-pheromone components should be evaluated in order to

develop an effective synthetic sex pheromone blend for monitoring field populations and/or for use as a tool in the integrated control of soybean pests.

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