under vacuum) 111–112.5°; ir (CH_2Cl_2) no OH absorption, bands at 1235, 1220, 1154, and 1031 cm^{-1}; nmr ($CHCl_3$) δ 1.0–1.25 (12 H, m), 1.4–2.1 (16 H, m), and 3.15–3.65 (8 H, m).

Anal. Calcd for $C_{17}H_{36}F_3NO_3S$: C, 52.15; H, 9.27; N, 3.58; S, 8.19. Found: C, 52.17; H, 9.23; N, 3.65; S, 8.38.

Method B.—A solution of 10 g (35.5 mmol) of trifluoromethanesulfonic anhydride and 45 ml of methylene dichloride was stirred and cooled in an ice bath. Dry pyridine (2.80 g, 35.5 mmol) was slowly added while the temperature was maintained below 10°. Dry *n*-butyl alcohol (2.63 g, 35.5 mmol) was then slowly added and the mixture was filtered. The filtrate, containing the butyl trifluoromethanesulfonate, was cooled in an ice bath and 6.58 g (35.5 mmol) of tri-*n*-butylamine (purified by distillation from naphthyl isocyanate) was slowly added. The solution was taken down to dryness and the solid was triturated with ether. The ethereal solution was filtered and taken down to dryness, and the product was recrystallized as above to give 6.9 g (50%).

Purification of Solvents.—Methylene dichloride was refluxed for 24 hr over, and distilled from, calcium hydride under argon, and stored over 3A and 5A molecular sieves. The specific resistance was 1.44 × 10^6 ohm cm.

Acetonitrile was refluxed for 2 days over, and then distilled from, calcium hydride (10 g/l.) under argon. This distillate was then twice distilled from P_2O_5 (5 g/l.) under argon and stored over 3A molecular sieves. The specific resistance was 9.9 × 10^5 ohm cm.

Dimethylformamide (spectra grade, Eastman) was stored over 4A molecular sieves. The specific resistance was 5.67 × 10^5 ohm cm.

Tetrahydrofuran was refluxed over and distilled from sodium–potassium alloy under argon.

Solubility Measurements.—Mixtures of the purified solvents and an excess of the salt (pulverized and dried under vacuum at 50°) were heated until most of the salt had dissolved and then held at 28° until equilibrium was reached. Three separate aliquots of the supernatant liquid were then taken down to dryness and dried to constant weight under vacuum.

Specific Resistance Measurements.—Measurements were made using an Industrial Instruments conductivity cell with platinized platinum electrodes. The cell constant (0.098) was determined using 0.100 and 0.010 *M* aqueous solutions of KCl. Solutions were protected from the atmosphere and immersed in an oil bath maintained at 25.0°. The resistances were measured on a Serfass conductivity bridge, Model RCM 15, at a frequency of 1 KHz.

Measurements of Limiting Anodic and Cathodic Potentials.—A mercury drop (25.1 mm^2), hanging from the end of a capillary, was used for the cathodic measurements against mercury, and a platinum disc (7.1 mm^2) was used for the anodic and cathodic measurements against platinum. The reference electrode consisted of a long, thin (0.4 × 15 cm) tube with a cracked tip at the bottom. This tube was filled with the solvent and supporting electrolyte under investigation, and the aqueous reference electrode was isolated *via* a KCl–agar bridge placed in it. The counter electrode was in every case a small platinum sphere. Readings were taken using an M. I. Associates polarographic instrument with a Tetronics oscilloscope for the display. The limiting potential was taken to be that voltage at which dI/dV equalled 5×10^{-6} A V^{-1}.

Registry No.—$Et_4NCF_3SO_3$, 35895-69-3; $(n\text{-}Pr)_4NCF_3SO_3$, 35925-48-5; $(n\text{-}Bu)_4NCF_3SO_3$, 35895-70-6; Et_4NBF_4, 429-06-1.

Synthesis of the Housefly Sex Attractant

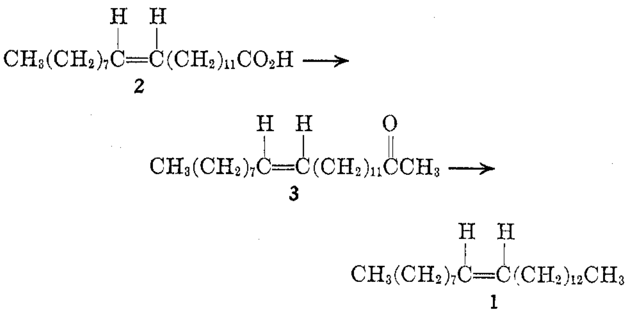
Robert L. Cargill* and M. G. Rosenblum

Department of Chemistry, University of South Carolina, Columbia, South Carolina 29208

Received June 6, 1972

The isolation, identification, and one synthesis of the sex attractant, (*Z*)-9-tricosene (1), of the housefly (*Musca domestica* L.) were recently reported.[1] We described here a very efficient synthesis of 1 from readily available starting materials.

Erucic acid (2)[2] was converted into ketone 3 by the action of 2 equiv of methyllithium.[3] Huang-Minlon reduction[4] of 3 gave 1 in an overall yield of 85%. This



$$\underset{\mathbf{2}}{CH_3(CH_2)_7\overset{H}{C}{=}\overset{H}{C}(CH_2)_{11}CO_2H} \longrightarrow$$

$$\underset{\mathbf{3}}{CH_3(CH_2)_7\overset{H}{C}{=}\overset{H}{C}(CH_2)_{11}\overset{O}{\overset{\|}{C}}CH_3} \longrightarrow$$

$$\underset{\mathbf{1}}{CH_3(CH_2)_7\overset{H}{C}{=}\overset{H}{C}(CH_2)_{12}CH_3}$$

process is easily adaptable to large-scale preparations and requires no expensive reagents.

Experimental Section[5]

(*Z*)-Tricos-14-en-2-one (3).—To a 10.7-g (31.3 mmol) quantity of erucic acid (2) (Columbia Organic Chemicals) in dry ether (200 ml) containing *o*-phenanthroline (5 mg) was added 31.5 ml (63.0 mmol) of 2.00 *M* methyllithium in hexane (Lithcoa), at a rate such that gas evolution was moderate. The reddish-brown reaction mixture was stirred for 30 min and quenched by cautiously adding 10% NaOH (100 ml), saturated $NaHCO_3$ (10 ml), and saturated $(NH_4)_2SO_4$ (10 ml). The phases were separated and the aqueous phase was extracted with ether (3 × 100 ml). The combined organic phases were dried ($MgSO_4$), concentrated, and distilled to give 9.77 g (93.3%) of ketone 3: bp 140° (0.10 mm); $n^{23}D$ 1.4572; ir (CCl_4) 3015 (olefinic CH), 1725 cm^{-1} (C=O); nmr (CCl_4) δ 0.88–2.50 (m, 42 H, all protons except olefinic; CH_3 s at δ 2.00), 5.27 [t, *J* (apparent) ≅ 4.5 Hz, 2 H, olefinic]; mass spectrum (70 eV) showed $M\cdot^+$ at *m/e* 336; vpc (3% SE-30, 8 ft × 0.125 in., 250°, 50 ml/min) showed one peak.

Anal. Calcd for $C_{23}H_{44}O$: C, 82.07; H, 13.18. Found: C, 82.04; H, 13.30.

(*Z*)-9-Tricosene (1).—To a solution of 3.1 g (47 mmol) of 85% KOH in diethylene glycol (30 ml) was added 4.77 g (14.1 mmol) of ketone 3 and 2.0 g (40 mmol) of 85% hydrazine hydrate. The reaction mixture was heated at 140° until the water had been removed and then at 193° for 4 hr. The cooled reaction mixture was poured into ice–water (150 ml), neutralized with 6 *N* HCl, and extracted with pentane (5 × 100 ml). The combined extracts were dried ($MgSO_4$), concentrated, and distilled to give 4.55 g (88.8%) of olefin 1: bp 170–172° (0.5 mm); $n^{23}D$ 1.4532 [lit.[1] bp 157–158° (0.1 mm); $n^{26}D$ 1.4517]; ir (CCl_4) 3015 cm^{-1} (olefinic CH); nmr (CCl_4) δ 0.67–2.25 (m, 44 H, all protons except olefinic; $CH_2CH{=}CHCH_2$ m, at δ 1.98), 5.25 [t, *J* (apparent) ≅ 4.5 Hz, 2 H, vinyl]; mass spectrum (70 eV) showed $M\cdot^+$ at *m/e* 322; vpc (3% SE-30, 8 ft × 0.125 in., 250°, 50 ml/min) showed one peak.

Anal. Calcd for $C_{23}H_{46}$: C, 85.63; H, 14.37. Found: C, 85.75; H, 14.30.

Registry No.—1, 27519-02-4; 3, 36706-99-7.

---

[1] D. A. Carlson, M. S. Mayer, D. L. Silhacek, J. D. James, M. Beroza, and B. A. Bierl, *Science*, **174**, 76 (1971).

[2] Eurcic acid is available commercially, or it can be isolated from rapeseed oil: C. R. Noller and R. H. Talbot, "Organic Syntheses," Collect. Vol. II, Wiley, New York, N. Y., 1943, p 258.

[3] M. J. Jorgenson, *Org. React.*, **18**, 1 (1970).

[4] Huang-Minlon, *J. Amer. Chem. Soc.*, **68**, 2487 (1946).

[5] Microanalyses were performed by Alfred Bernhardt Microanalytisches Laboritorium, Elbach über Engleskirchen, Mülheim (Ruhr), West Germany.