

Subscripts

M	methane
D	diphenylmethane
MN	1-methylnaphthalene
T	tetralin

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NEW COMPOUNDS

Syntheses of Trans Alken-1-ols as Candidates for Insect Sex Attractants

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The syntheses of 21 acetylenic alcohols of the general formula $\text{CH}_3(\text{CH}_2)_m\text{C}\equiv\text{C}(\text{CH}_2)_n\text{OH}$, where $m = 0-6$ and $n = 3-6, 8-11$, were accomplished by both solution and solid-phase methods. Conversion of the acetylenic alcohols to the geometrically pure trans alken-1-ols was done. The IR and NMR spectra and some physical properties of the alkynols and alkenols are given.

Studies directed toward the determination of the insect sex attractants of a host of forest and orchard pests (1) have led to the synthesis (2-4) of a large number of candidate pheromones suitable for field testing and laboratory assays. Most of the alkynols and trans alkenols described herein were previously unknown or incompletely characterized.

Alkynols 4-11 were prepared by our solid-phase method (5-8) (method I) and alkynols 14-26 by previously reported solution methods (9) (method II). In method I, polymer-bound diol monomesylates (1, $n = 4, 6, 8, 9$) [P-Tr - represents a 2% divinylbenzene-styrene copolymer containing trityl groups] react with 1-lithioalkynes (2, $m = 0-4, 6$) [or with lithioacetylide and alkyl halide treatment in a two-step reaction (7)] to give the polymer-bound alkynes (3, $m = 0-4, 6; n = 4, 6, 8, 9$). Cleavage of 3 with acid yielded alkynols 4-11, respectively (Scheme I). In method II, tetrahydropyranyl (THP) ethers of ω -bromo- or ω -chloroalkanols (12, $X = \text{Br}, \text{Cl}; n = 3-6, 10, 11$)

react with 1-lithio-1-alkynes (2, $m = 1-5$) to give the tetrahydropyranyl ethers of the alkynols (13, $m = 1-5; n = 3-6, 10, 11$) which on acid hydrolysis yield alkynols 14-26. The alkynols 4-11 and 14-26 were converted to their tetrahydropyranyl ethers where necessary, reduced with sodium in liquid ammonia and THF (9, 10), and treated with acid to give exclusively the trans alkenols (27-47) (Scheme I). The geometrical purities of 27-47 were confirmed by vapor-phase chromatography (VPC) and high-pressure liquid chromatography (LC). Trans alkenols 28, 37, 39, and 41 were converted to their acetates 28a, 37a, 39a, and 41a, respectively. Previous syntheses of sex attractants on solid phases yielded millimole quantities of attractants. We describe here the scaled up synthesis on solid phases of gram quantities of sex attractants in a modified procedure (method I). The physical and spectral properties of 4-11, 14-26, 27-47, 28a, 37a, 39a, and 41a are recorded in Tables I-V, respectively.

Experimental Section

A Bausch and Lomb Abbé 3L refractometer was used to record the refractive indices. Infrared spectra were recorded on a Unicam SP1000 IR spectrophotometer as neat films between NaCl disks unless otherwise specified. Nuclear magnetic resonance spectra were recorded on a Varian EM360 spectrometer using deuteriochloroform as solvent and tetramethylsilane as internal standard.

Table I. Physical Properties of Alkyn-1-ols^a

compd	name of compd	method of synthesis	yield, ^b %	bp, °C (mmHg)	n_D (°C)
4	5-octyn-1-ol	I	35		1.4590 (23.5)
5	10-dedecyn-1-ol	I	47	84-85 (0.02)	1.4592 (23)
6	7-tridecyn-1-ol	I	72	112 (0.08)	1.4592 (23) ^c
7	9-tridecyn-1-ol	I	31	110 (0.03)	1.4589 (24)
8	10-tridecyn-1-ol	I	29	114 (0.02)	1.4594 (24)
9	7-pentadecyn-1-ol	I	63	110-112 (0.03)	1.4589 (24)
10	9-pentadecyn-1-ol	I	38	118-119 (0.02)	1.4606 (25)
11	10-pentadecyn-1-ol	I	32	120-121 (0.02)	1.4606 (25)
14	5-nonyl-1-ol	II	72	76-78 (0.05)	1.4578 (25)
15	6-nonyl-1-ol	II	75	76-78 (0.05)	1.4587 (25)
16	4-decyn-1-ol	II	80	85-86 (0.05)	1.4589 (25)
17	5-decyn-1-ol	II	70	84-86 (0.05)	1.4585 (25)
18	6-decyn-1-ol	II	88	84-85 (0.05)	1.4585 (25)
19	7-decyn-1-ol	II	74	88 (0.03)	1.4608 (22)
20	4-undecyn-1-ol	II	80	80 (0.03)	1.4592 (24) ^d
21	5-undecyn-1-ol	II	70	83-84 (0.01)	1.4586 (25)
22	6-undecyn-1-ol	II	76	90-92 (0.03)	1.4582 (24)
23	7-undecyn-1-ol	II	70	84-86 (0.04)	1.4600 (24)
24	6-tridecyn-1-ol	II	78	110-112 (0.03)	1.4592 (24) ^e
25	11-pentadecyn-1-ol	II	74	126-128 (0.03)	1.4610 (24) ^f
26	12-pentadecyn-1-ol	II	94	125-126 (0.02)	mp 35-36 °C

^a Elemental analyses (C, H) were in agreement with theoretical values and submitted for review. ^b Based on chloro (bromo) alcohols for method II but on symmetrical diol for method I. ^c Lit. (11) bp 110-115 °C (0.4 mmHg), n_D^{25} 1.4699. ^d Lit. (12) bp 70-84 °C (0.01 mmHg). Lit. (11) bp 133-140 °C (20 mmHg), n_D^{25} 1.4520. ^e Lit. (13) bp 107-109 °C (0.1 mmHg), n_D^{25} 1.4608. ^f Crystallized in the refrigerator.

Table II. IR^a and NMR^b Data for the Alkyn-1-ols

compd	IR, cm ⁻¹		NMR, δ (J value in Hz)				
	OH str	C-O str	HOCH ₂ (CH ₂) _{n-2} CH ₂ C≡CCH ₂ (CH ₂) _{m-1} CH ₃				
			a	b	c	d	e
4	3375	1060	3.50 t (8.0)	3.30 s	2.10 m	1.82-1.34 m	1.10 t (8.0)
5	3410	1040	3.65 t (6.0)	c	2.10 m	1.70-1.08 m	
6	3360	1060	3.64 t (7.0)	2.48 s	2.18 m	1.88-1.11 m	0.90 t (4.0)
7	3355	1060	3.62 t (6.2)	1.86 s	2.2 m	1.76-1.20 m	1.00 t (7.5)
8	3375	1060	3.68 t (7.0)	1.79 s	2.14 m	1.60-1.20 m	1.05 t (3.0)
9	3350	1055	3.66 t (8.0)	1.80 s	2.12 m	1.95-1.10 m	0.90 t (7.5)
10	3350	1060	3.64 t (7.0)	1.82 s	2.18 m	1.84-1.08 m	0.87 t (5.0)
11	3375	1060	3.64 t (7.0)	c	2.14 m	1.90-1.11 m	0.90 t (6.3)
14	3355	1060	3.68 t (7.0)	c	2.16 m	1.90-1.12 m	1.10 t (7.0)
15	3355	1060	3.62 t (7.0)	1.80 s	2.26	1.72-1.30 m	1.00 t (8.0)
16	3355	1060	3.73 t (6.5)	2.34 s	2.18 m	1.90-1.10 m	0.89 t (6.0)
17	3375	1060	3.73 t (6.0)	2.31	2.17 m	1.92-1.20 m	0.9 t (4.0)
18	3360	1055	3.68 t (6.0)	1.78 s	2.12 m	1.97-1.26 m	0.97 t (7.0)
19	3355	1060	3.62 t (7.0)	1.75 s	2.13 m	1.69-1.10 m	1.10 t (9.0)
20	3350	1055	3.78 t (6.0)	2.16 s	2.16 m	1.90-1.10 m	0.88 t (4.0)
21	3350	1055	3.68 t (8.0)	c	2.22 m	1.95-1.08 m	0.88 t (7.0)
22	3350	1055	3.78 t (6.0)	2.16 s	2.16 m	1.90-1.10 m	0.88 t (4.0)
23	3380	1060	3.62 t (6.0)	1.70 s	2.12 m	1.63-1.15 m	0.95 t (8.0)
24	3360	1055	3.65 t (7.0)	c	2.18 m	1.96-1.10 m	0.90 t (5.0)
25	3350	1060	3.64 t (8.0)	c	2.14 m	1.82-1.12 m	0.92 t (9.0)
26	3350 ^d	1070 ^d	3.69 t (5.5)	2.02 s	2.20 m	1.95-1.20 m	1.05 t (4.0)

^a Only major IR bands are reported. ^b Key: s = singlet, t = triplet, m = multiplet. ^c If OH is absent, it is buried in d, i.e., between δ 1.96 and 1.08. ^d Nujol.

Vapor-phase chromatograms were run with a Perkin-Elmer 990 instrument using a 15 ft \times 1/8 in. column of 10% Silar C on Gaschrom Q (60/80) at a temperature of 170 °C and a nitrogen flow rate of 15 mL/min. High-pressure liquid chromatograms were run with a Waters Associates Model 440 instrument, with an R-400 refractive index detector and a 30 \times 0.4 cm reverse phase μ Bondapak C-18 column using water-acetonitrile mixtures (1:1) for C-10 acetates, 40% water in acetonitrile for C-11 to C-13 acetates, and 25% water in acetonitrile for C-15 acetates at a flow rate of 1.0 mL/min.

Silica gel was used for all thin and preparative layer chromatography. Fractions were extracted with ether in a Soxhlet extractor. Filtration was done under vacuum through sintered glass Büchner funnels.

The solvents benzene, pyridine, dioxane, tetrahydrofuran (THF), hexamethylphosphoric triamide (HMPT), and others were dried and distilled. *Caution: HMPT is a suspected carcinogen!*

The polymers after being washed and air-dried were transferred to a thimble in a Soxhlet extractor in which molecular sieves (3A) had been placed in a second thimble and the polymer was extracted with benzene for 3-5 h under reflux conditions. The polymer was washed with dry ether and dried in vacuo at 0.2-0.5 mmHg for 0.3-3 h.

Microanalyses were performed by G. Gygli of Toronto or Dr. C. Daesslé of Montreal.

Synthesis of Polymer-Bound Diol Monomesylates 1. To a three-necked flask fitted with a mechanical stirrer, a dropping funnel, and a CaCl₂ drying tube and containing 85 g of dry

Table III. Physical Properties of Trans Alken-1-ols^a

compd	name of compd	yield, ^b		bp, °C (mmHg)	n _D (°C)
		%			
27	trans-5-octen-1-ol	85			1.4471 (21) ^{c,d}
28	trans-10-dodecen-1-ol	95		91-92 (0.02)	1.4519 (23) ^e
29	trans-7-tridecen-1-ol	79		95-96 (0.02)	1.4502 (23)
30	trans-9-tridecen-1-ol	72		90-92 (0.03)	1.4504 (23)
31	trans-10-tridecen-1-ol	78		94-95 (0.02)	1.4510 (23)
32	trans-7-pentadecen-1-ol	90		113-114 (0.02)	1.4518 (25) ^{f,g}
33	trans-9-pentadecen-1-ol	80		108-110 (0.02)	1.4521 (25) ^g
34	trans-10-pentadecen-1-ol	90		110-111 (0.02)	1.4521 (25) ^g
35	trans-5-nonen-1-ol	94		60 (0.10)	1.4485 (22) ^{h,i}
36	trans-6-nonen-1-ol	90		70-71 (0.05)	1.4493 (23) ^j
37	trans-4-decen-1-ol	93		63-64 (0.02)	1.4458 (26) ^k
38	trans-5-decen-1-ol	92		57-58 (0.02)	1.4496 (24) ^l
39	trans-6-decen-1-ol	91		72 (0.03)	1.4480 (22) ^l
40	trans-7-decen-1-ol	85		75 (0.02)	1.4496 (24) ^m
41	trans-4-undecen-1-ol	90		73-74 (0.02)	1.4498 (24) ⁿ
42	trans-5-undecen-1-ol	85		85 (0.02)	1.4482 (24)
43	trans-6-undecen-1-ol	90		83-84 (0.03)	1.4485 (24) ^o
44	trans-7-undecen-1-ol	75		75 (0.02)	1.4502 (26)
45	trans-6-tridecen-1-ol	84		93-94 (0.02)	1.4510 (23) ^p
46	trans-11-pentadecen-1-ol	79		112-113 (0.02)	1.4542 (26) ^g
47	trans-12-pentadecen-1-ol	85		108-110 (0.02)	1.4545 (26) ^g

^a Elemental analyses (C, H) were in agreement with theoretical values and are submitted for review. ^b From alkyne-1-ols. ^c Lit. (14) bp 79 °C (8 mmHg), n_D²⁵ 1.4451. ^d Lit. (15) bp 91.5 °C (14 mmHg), n_D²¹ 1.4476. ^e Lit. (16) bp 143-144 °C (13 mmHg). ^f Lit. (17) bp 198-204 °C (5 mmHg). ^g All C₁₅ trans alcohols crystallized in the refrigerator. ^h Lit. (18) bp 107 (18 mmHg), n_D²⁵ 1.4455. ⁱ Lit. (19) bp 107 °C (17.5 mmHg), n_D²⁰ 1.4475. ^j Lit. (18, 20) bp 75-84 °C (0.2 mmHg), n_D²⁵ 1.4471. ^k Lit. (21) bp 228-228.5 °C, n_D²⁰ 1.4471. ^l Lit. (21) bp 228-228.5 °C, n_D²⁰ 1.4499. ^m No chemical or physical data described (22, 23). ⁿ Lit. (14) bp 92 °C (7 mmHg), n_D²⁵ 1.4518. ^o Lit. (24) bp 136 °C (20 mmHg), n_D^{22.5} 1.4511. ^p Lit. (25) bp 89-92 °C (0.44 mmHg), n_D²⁵ 1.4493. ^q Lit. (13) bp 117-118 °C (0.4 mmHg), n_D²⁵ 1.4532.

Table IV. IR^a and NMR^b Data for the Trans Alken-1-ols

	IR, cm ⁻¹			NMR, δ (J value in Hz)					
	OH str	C-O str	trans C-H	HOCH ₂ (CH ₂) _{n-2} CH ₂ CH=CHCH ₂ (CH ₂) _{m-1} CH ₃					
				a	b	c	d	e	f
27	3370	1060	965	3.64 t (6.5)	3.21 s	2.20-1.78 m	1.78-1.10 m	0.90 t (7.0)	5.38 m
28	3350	1060	965	3.60 t (6.0)	c	2.20-1.80 m	1.80-1.03 m	0.87 t (2.0)	5.41 m
29	3355	1060	965	3.65 t (7.0)	c	2.20-1.78 m	1.78-1.10 m	0.89 t (6.0)	5.41 m
30	3365	1060	965	3.66 t (8.0)	c	2.20-1.80 m	1.80-1.10 m	0.90 t (9.0)	5.45 m
31	3340	1070	965	3.62 t (6.0)	c	2.20-1.78 m	1.78-1.08 m	0.90 t (8.0)	5.40 m
32	3350	1060	965	3.68 t (7.0)	c	2.20-1.78 m	1.78-1.08 m	0.90 t (5.0)	5.42 m
33	3350	1060	965	3.66 t (7.0)	c	2.14-1.70 m	1.70-1.02 m	0.86 t (3.0)	5.41 m
34	3370	1060	965	3.68 t (8.0)	c	2.20-1.80 m	1.80-1.10 m	0.90 t (8.0)	5.41 m
35	3350	1060	965	3.64 t (6.0)	c	2.24-1.78 m	1.78-1.02 m	0.90 t (6.5)	5.40 m
36 ^d	3350	1060	965	3.64 t (8.0)	2.0 s	2.15-1.78 m	1.78-1.15 m	0.95 t (8.0)	5.48 m
37	3350	1060	960	3.66 t (8.0)	1.98 s	2.34-1.52 m	1.52-1.04 m	0.98 t (6.0)	5.46 m
38	3375	1060	965	3.68 t (6.0)	1.82 s	2.20-1.80 m	1.80-1.10 m	0.90 t (7.5)	5.42 m
39	3360	1060	965	3.65 t (8.0)	1.68 s	2.30-1.75 m	1.75-1.16 m	0.85 t (8.0)	5.40 m
40	3360	1055	965	3.65 t (7.0)	2.2 s	2.32-1.88 m	1.88-1.12 m	0.95 t (9.0)	5.40 m
41	3350	1060	975	3.66 t (6.0)	c	2.20-1.80 m	1.80-1.10 m	0.88 t (6.0)	5.41 m
42	3380	1070	975	3.60 t (7.0)	c	2.20-1.78 m	1.78-1.10 m	0.88 t (5.0)	5.42 m
43	3360	1060	975	3.60 t (6.0)	c	2.20-1.80 m	1.80-1.10 m	0.88 t (7.0)	5.41 m
44	3350	1060	970	3.68 t (6.0)	c	2.20-1.80 m	1.80-1.15 m	0.88 t (9.0)	5.42 m
45	3350	1060	965	3.65 t (8.0)	c	2.20-1.80 m	1.80-1.10 m	0.89 t (6.0)	5.41 m
46	3350	1060	965	3.65 t (6.0)	c	2.20-1.80 m	1.80-1.10 m	0.90 t (6.0)	5.41 m
47	3350	1060	965	3.68 t (7.0)	1.56 s	2.14-1.62 m	1.50-1.10 m	0.94 t (8.0)	5.48 m

^a Only major IR bands are reported. ^b Key: s = singlet, t = triplet, m = multiplet. ^c OH is buried in d between δ 1.80 and 1.02. ^d The NMR spectrum for this compound is in agreement with ref 20.

polymer-bound 1,8-octanediol (**1**, OMs = OH, *n* = 8) (77 mmol/diol) in 1 L of benzene-pyridine (3:2) was added dropwise 85 mL of methansulfonyl chloride (fresh distilled) at -10 to +10 °C. The stirring was continued for 48 h at room temperature. The polymer was filtered, washed with pyridine, ethanol, water, ethanol, and ether (**6**), and, after air-drying, further dried in a Soxhlet extractor. Acid cleavage (**6**) of 1.0 g of **1** (*n* = 8) gave 45 mg (0.27 mmol/g) of 8-chloro-1-octanol, 61 mg (0.27 mmol/g) of 1,8-octanediol monomesylate, and 47 mg (0.32 mmol/g) of 1,8-octanediol. These compounds were identified by comparing their constants with those given in the literature (**6**).

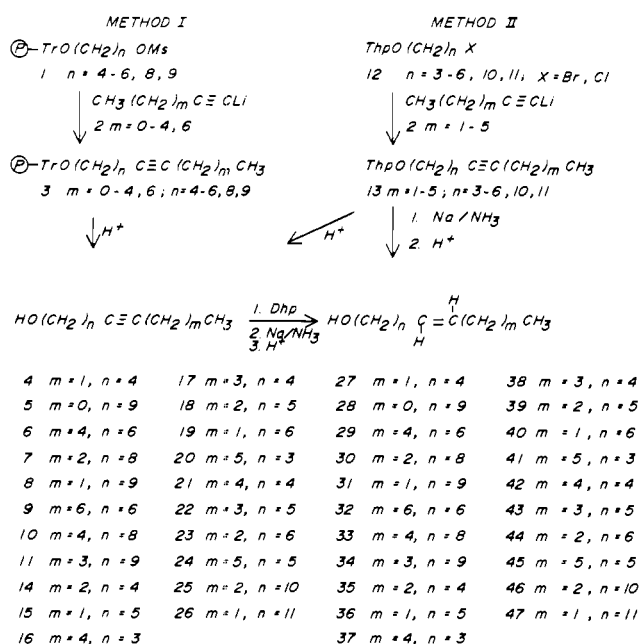
Synthesis of Polymer-Bound Alkynes 3. To a stirred solution of 350 mL of THF under argon was added 40 mL (96 mmol) of *n*-butyllithium (in hexane) and 6.8 g (100 mmol) of 1-pentyne in 50 mL of THF, keeping the temperature at -10 to 0 °C. After 30 min of stirring, 50 g of **1** (*n* = 8) containing 27 mmol of 1,8-octanediol monomesylate and 400 mL of HMPT was added. The stirring was continued overnight at room temperature. The reaction mixture was cooled to -10 °C and the excess lithiopentyne was quenched with THF-water (1:1). The polymer-bound alkyne (**3**, *m* = 2, *n* = 8) was washed with ethanol, water, ethanol, dioxane, and ether and air-dried (**6**).

Table V. Physical Properties and IR^a and NMR^b Data for Trans Alken-1-ol Acetates

		$\text{CH}_3\text{CO}_2\text{CH}_2(\text{CH}_2)_n\text{CH}_2\text{CH}=\text{CHCH}_2(\text{CH}_2)_{n-1}\text{CH}_3$										
		IR, cm ⁻¹					NMR, δ (J value in Hz)					
compd	name of compd	yield, ^c %	$n^{24}\text{D}$	CH ₃ C=O str	C-O str	trans OH	a	b	c	d	e	f
28a	<i>trans</i> -10-dodecen-1-yl acetate ^{d,e}	85	1.4388	1750	1245	965	4.08 t (7.0)	2.045	2.15- 1.58 m	1.58- 1.08 m		5.45 m
37a	<i>trans</i> -4-decen-1-yl acetate ^f	90	1.4365	1740	1240	960	4.10 t (7.0)	2.085	2.30- 1.90 m	1.58- 1.03 m	0.90 t (6.0)	5.42 m
39a	<i>trans</i> -6-decen-1-yl acetate ^{d,g}	89	1.4370	1750	1250	960	4.10 t (7.0)	2.045	2.20- 1.68 m	1.68- 1.03 m	0.89 t (6.0)	5.42 m
41a	<i>trans</i> -4-undecyn-1-yl acetate ^h	90	1.4370	1750	1250	960	4.15 t (8.0)	2.065	2.18- 1.58 m	1.58- 1.08 m	0.89 t (5.0)	5.42 m

^a Only major IR bands are reported. ^b Key: s = singlet, t = triplet, m = multiplet. ^c From *trans* alken-1-ols. ^d These acetates are mentioned in a biological test without physical or spectroscopic data. ^e Reference 26. ^f bp 54–55 °C (0.02 mmHg). ^g References 22 and 23. ^h Lit. (24) bp 145 °C (20 mmHg), n^{22}_D 1.4397.

Scheme I



Synthesis of a Representative Alkyn-1-ol (7) by Acid Cleavage of 3 ($m = 2, n = 8$). After a suspension of 50 g of **3** ($m = 2, n = 8$) in 700 mL of 0.35 M HCl-dioxane solution was stirred at room temperature for 48 h, the polymer was filtered and washed with three 50-mL portions of ethanol, ten 50-mL portions of water, six 50-mL portions of ethanol, and six 100-mL portions of ether. The organic layer was separated and the aqueous layer was further extracted with three 200–300-mL portions of ether. The ether extracts were combined and washed with cool saturated sodium chloride and finally with 10% Na₂CO₃. The solvent was removed by evaporation and the resulting residue (5 g of brown oil) was chromatographed on a column packed with silica gel (70 × 2.5 cm). Elution with benzene, benzene-ether, and ether-THF (after evaporation of solvent) gave, according to TLC, the corresponding fractions: first, 0.1 g (eluant benzene and benzene:ether (95:5)); second, 0.5 g (benzene:ether (90:10)); third, 1.65 g, contained chromatographically pure alkyn-1-ol (**7**) (benzene:ether (85:15));

fourth, 0.2 g + 0.3 g (benzene:ether (80:20) (50:50)); fifth, 1.5 g contained pure diol (ether-THF). The second and fourth fractions were rechromatographed to give an additional 0.2 g of pure alkyn-1-ol (**7**). The combined pure fraction was distilled in vacuo. Analysis by high-pressure LC showed 98–100% purity (Table I).

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Supplementary Material Available: Elemental analyses (2 pages). Ordering information is given on any current masthead page.