for 970 reflections with $I \geq 1.5\sigma(I)$ was 0.060 (R_w = 0.050). The weighing scheme in the last cycle was $w = 2.6[\sigma^2(F) + 0.0003(F)^2]^{-1}$.

Isomer δ. Crystals were obtained from solutions in ethanol. The molecule (M_r 152) crystallizes in space group $P\bar{1}$ with a = 7.811 (5) Å, b = 8.028 (5) Å, c = 6.999 (4) Å, α = 117.5 (1)°, β = 81.2 (1)°, γ = 111.3 (1)°. The cell volume was 326.6 Å^3, Z = 2. Obtainment and treatment of data were similar to those referring to isomer γ. The final R factor for 1157 reflections with $I \geq 1.5\sigma(I)$ was 0.043 (R_w = 0.058).

All calculations for γ and δ isomers were carried out on the IBM 370/158 Computer of the University of Padova.

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Registry No. **1a**, 123-54-6; **1b**, 141-97-9; **1c**, 93-91-4; **1d**, 1118-71-4; **1e**, 120-46-7; **1f**, 108-59-8; **1g**, 367-57-7; **1h**, 22767-90-4; **2a**, 77097-65-5; **2b**, 79593-42-3; **2c**, 79593-43-4; **3a**, 71616-10-9; **3b**, 90281-20-2; **3c**, 90281-23-5; **3d**, 87221-86-1; **3e**, 92220-21-8; **3f**, 90281-22-4; **7a**, 92220-23-0; **7b**, 92220-27-4; **7c**, 92220-26-3; **8a**, 92220-24-1; **9**, 92220-25-2; $C_2H_5O^-$, 16331-64-9; $NC(CH_2)_2CN$, 460-19-5; *N*-ethylaniline, 103-69-5; triethylamine, 121-44-8.

Supplementary Material Available: Analytical data for compounds **3**, **7**, **8**, **9**; tables of X-ray data for compounds including fractional coordinates, thermal parameters, and deviations from the least square plane of the ring (8 pages). Ordering information is given on any current masthead page.

Alkynylaryliodonium Tosylates and Aryl[β-(tosyloxy)vinyl]iodonium Tosylates from Reactions of Terminal Alkynes with [Hydroxy(tosyloxy)iodo]benzene

Louis Rebrovic and Gerald F. Koser*

Department of Chemistry, The University of Akron, Akron, Ohio 44325

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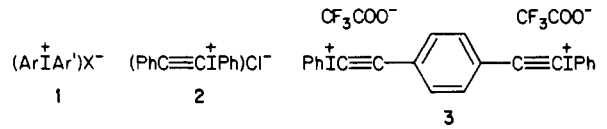
Various terminal alkynes have been found to react with [hydroxy(tosyloxy)iodo]benzene (**4**) in $CHCl_3$ to give either aryl[β-(tosyloxy)vinyl]iodonium tosylates **5** or alkynylaryliodonium tosylates **6** or a mixture of the two. The product composition is subject to steric control. Among the α-branched alkyl groups R in RC≡CH, the isopropyl group seems to define the steric median: those alkynes with R larger than isopropyl (i.e., R = *t*-Bu, *sec*-Bu, cyclohexyl) give only alkynylaryliodonium tosylates while those alkynes with R smaller than isopropyl give only aryl[β-(tosyloxy)vinyl]iodonium tosylates. 3-Methyl-1-butyne and 4-methyl-1-pentyne (β-branching) give a mixture of **5** and **6**. (Trimethylsilyl)acetylene reacts with **4** in a different way; the trimethylsilyl group is cleaved from the alkyne, and phenyl[β-(tosyloxy)vinyl]iodonium tosylate is obtained.

Although the diaryliodonium salts **1** have been known for 90 years and a number of them have been prepared, iodonium salts with an alkynyl ligand bound to the iodine atom are rare.[1] The first example of an alkynyliodonium salt was reported by Beringer and Galton in 1965 who prepared phenyl(phenylethynyl)iodonium chloride (**2**) in



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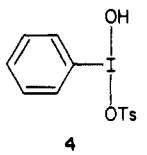

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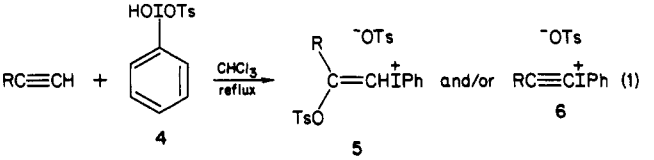
yields of 12–20% by the condensation of lithium phenylacetylide with (dichloroiodo)benzene in ether/hexane at 0–5 °C.[2] The iodonium salt decomposed upon standing for several hours at room temperature into a 1:1 mixture of chlorophenylacetylene and iodobenzene. More recently, the condensation of 1,4-diethynylbenzene with [bis(trifluoroacetoxy)iodo]benzene in dry chloroform to give the alkynyliodonium salt **3** has been reported.[3] In a recent preliminary communication, we described the reactions of several alkenes and several alkynes with [hydroxy(tosyloxy)iodo]benzene (**4**).[4] Particularly relevant is the observation that phenylacetylene and cyclohexylacetylene were converted directly by **4** into the corresponding alkynylphenyliodonium tosylates (60% and 5% yields, respectively).

In this paper, we report the reactions of ten terminal alkynes with [hydroxy(tosyloxy)iodo]benzene and the use of steric bulk in the alkyne to direct a one-step synthesis of alkynylphenyliodonium salts. The treatment of terminal alkynes with **4** in chloroform under reflux affords either phenyl[β-(tosyloxy)vinyl]iodonium tosylates **5** or alkynylphenyliodonium tosylates **6** or a mixture of both (eq 1). For example, 1-pentyne reacted with **4** to give the



4 5 6

vinyliodonium tosylate **5** (R = *n*-Pr, 58% yield), but when 3,3-dimethyl-1-butyne was the reactant, only the alkynyliodonium tosylate **6** (R = *t*-Bu, 74%) was obtained. 3-

(1) See: Koser, G. F. In "The Chemistry of the Functional Groups", Supplement D; Patai, S., Rappoport, Z., Ed. Wiley: Chichester, 1983; Chapter 25 and references cited therein.
(2) Beringer, F. M.; Galton, S. A. *J. Org. Chem.* **1965**, *30*, 1930.
(3) Merkushev, E. B.; Karpitskaya, L. G.; Novosel'tseva, G. I. *Dokl. Akad. Nauk. SSSR* **1979**, *245*, 607.
(4) Koser, G. F.; Rebrovic, L.; Wettach, R. H. *J. Org. Chem.* **1981**, *46*, 4324.

0022-3263/84/1949-4700$01.50/0

Table I. Reactions of Terminal Alkynes with [Hydroxy(tosyloxy)iodo]benzene

RC≡CH				yield,[e] %	
R	mmol[a]	4, mmol	time	5	6
n-Pr	51	20.0[b]	3 h	58[f]	
n-Bu	44	20.0[b]	3 h	52[g]	
n-C_5H_{11}	38	20.0[b]	3 h	26[h]	
i-Pr	29	10.0[c]	1.5 h	11	15
sec-Bu	26	10.0[c]	3.5 h		50.5
i-Bu	52	20.0[b]	3 h	29	33
c-C_6H_{11}	46	21.1[b]	1.5 h		47
t-Bu	73	20.0[b]	5 h		74
Ph	182	30.0[d]	20 min		61

[a] Based on the density and added volume of the alkyne. The volumes, from top to bottom, were 5.0, 5.0, 5.0, 3.0, 3.0, 6.0, 6.0, 9.0, and 20.0 mL. [b] in $CHCl_3$, 50 mL. [c] in $CHCl_3$, 25 mL. [d] in $CHCl_3$, 30 mL. [e] Based on the amount of product obtained prior to purification. [f] High melting isomer, 21%; low melting isomer, 37%. [g] High melting isomer, 27%; low melting isomer, 24%. [h] A second fraction (1.75 g) of solid product of lower melting point was obtained which appears, by ^1H NMR analysis, to be a dihydrate of the isomeric vinyliodonium salt. However, the elemental analysis is not consistent with such a structure (see Experimental Section).

Methyl-1-butyne gave a mixture of **5** (R = *i*-Pr, 11%) and **6** (R = *i*-Pr, 15%).

Among the α-branched alkyl groups, the isopropyl group seems to define the steric divide: those alkynes with R larger than isopropyl (i.e., *t*-Bu, *sec*-Bu, cyclohexyl) give **6** while those with R smaller than isopropyl (i.e., *n*-Pr, *n*-Bu, *n*-C_5H_{11}) give **5**. If the Taft E_s constants are employed as a gauge of steric bulk, the isobutyl group "behaves" somewhat anomalously since a mixture of **5** (R = *i*-Bu) and **6** (R = *i*-Bu) was obtained from **4** and 4-methyl-1-pentyne. In at least two cases (R = *n*-Pr, *n*-Bu), vinyliodonium tosylates were formed as a mixture of high melting and low melting isomers which could be separated by selective crystallization. These are presumably geometric (i.e., *E*, *Z*) isomers, but we have been unable to assign a specific geometry to a given isomer by either ^1H NMR or ^{13}C NMR analysis. Only one geometric isomer (i.e., *E* or *Z*) of **5** was apparently obtained from 3-methyl-1-butyne, but the product from 4-methyl-1-pentyne appears to be a mixture of isomers.

Workups were straightforward. For example, the solution that resulted from the reaction of **4** with 3-methyl-1-butyne was dried and concentrated. The residual oil was washed with ether and crystallized from ether:acetone (5:1 v/v) to give **5** (R = *i*-Pr). The mother liquor was diluted with ether and cooled at −20 °C whereupon **6** (R = *i*-Pr) crystallized from solution. The structure of **5** was determined by elemental (C, H, I) and NMR (^1H, ^{13}C) analyses and the structure of **6** by elemental (C, H, I), ^1H NMR, and IR (C≡C) analyses.

Reaction conditions and yields for the reactions of terminal alkynes with **4** are summarized in Table I.

(Trimethylsilyl)acetylene reacted with **4** in a somewhat different way. Phenyliodination did occur, but the reaction proceeded with cleavage of the trimethylsilyl group to give phenyl [(β-tosyloxy)ethenyl]iodonium tosylate in 22% yield (eq 2).

$$Me_3SiC{\equiv}CH + PhI(OH)OTs \xrightarrow[\Delta]{CHCl_3} TsOCH{=}CHI^+Ph + {}^-OTs \quad (2)$$

Experimental Section

I. General Methods. ^1H NMR spectra (60 MHz) were recorded on a Varian Model EM-360 spectrometer. Chemical shifts are expressed relative to internal Me_4Si, and the number of "protons" reported for a given multiplet is based on the combined integrations of all resonances in a spectrum (except for those of minor impurities) divided by the total number of "protons" in the molecule under consideration. ^{13}C NMR spectra were recorded on a Varian Model FT-80A spectrometer. The acronyms ORD and APT refer to "off resonance decoupling" and "attached proton test", and the symbols u and d for APT spectra stand for "up" and "down". Chemical shifts are expressed relative to internal Me_4Si. IR spectra were recorded on a Perkin-Elmer Model 597 spectrophotometer. Elemental compositions were determined by Galbraith Laboratories Inc., Knoxville, TN. Melting points and decomposition points are uncorrected.

II. Reactions of Terminal Alkynes with [Hydroxy(tosyloxy)iodo]benzene. Initial Workup Procedure. The reaction mixtures described in Table I were colored solutions and were generally treated with Na_2SO_4, sometimes after initial washing with H_2O, and concentrated under aspirator vacuum to an oil.

1-Pentyne. The yellow oil was dissolved in boiling acetone (20 mL), and the solution was cooled at −20 °C whereupon 1.30 g (21.2%) of (*E* or *Z*)-phenyl[β-(tosyloxy)-β-*n*-propylvinyl]-iodonium tosylate separated; mp 136–138 °C. Recrystallization of 1.17 g from acetone (20 mL) returned 0.989 g: mp 138.5–140.5 °C; ^1H NMR ($CDCl_3$) δ 0.48–1.04 (m, 3.1 H), 1.04.–1.84 (m, 2.3 H), 2.14–2.74 (m, 7.8 H, singlets at 2.31 and 2.43), 6.74–8.08 (m, 13.8 H); ^{13}C NMR ($CDCl_3$) (ORD multiplicity) δ 12.94 (q), 19.71 (m), 21.18 (m), 21.70 (m), 36.19 (t), (75.59, 77.19, 78.82; $CDCl_3$), 90.96 (d), 114.53 (s), 125.89–134.92 (m), 139.25 (s), 143.05 (s), 146.82 (s), 161.71 (s).

Anal. Calcd for $C_{25}H_{27}S_2O_6I$: C, 48.86; H, 4.43; I, 20.65. Found: C, 48.79; H, 4.41; I, 20.60.

The mother liquor from the first acetone crystallization was concentrated to a yellow oil. The oil was dissolved in warm Et_2O (5 mL), and the solution was cooled at −20 °C to give a white precipitate which was washed with Et_2O (10 mL), H_2O (10 mL), and again with Et_2O (10 mL), and dried over P_2O_5 under vacuum: yield, 2.251 g (36.6%); mp 104–114 °C. Recrystallization of 2.03 g from Et_2O/CH_2Cl_2 (25 mL; 20/5) at −20 °C returned 1.73 g of (*Z* or *E*)-phenyl[β-(tosyloxy)-β-*n*-propylvinyl]iodonium tosylate: mp 118–122 °C; ^1H NMR ($CDCl_3$) δ 0.48–1.80 (m, 5.9 H), 2.12–2.72 (m, 7. 6 H, singlets at 2.28 and 2.30), 6.68–8.13 (m, 13.5 H); ^{13}C NMR ($CDCl_3$) (ORD multiplicity) 13.08 (q), 19.17 (m), 21.04 (m), 21.47 (m), 36.26 (t), (75.58, 77.19, 78.82; $CDCl_3$), 92.65 (d), 115.77 (s), 125.26–134.71 (m), 139.24 (s), 142.46 (s), 145.91 (s), 161.70 (s).

Anal. Calcd for $C_{25}H_{27}S_2O_6I$: C, 48.86; H, 4.43; I, 20.65. Found: C, 48.65; H, 4.39; I, 20.53.

1-Hexyne. The yellow oil was dissolved in boiling acetone (20 mL), and the solution was cooled at −20 °C whereupon 1.72 g (27.4%) of (*E* or Z)-phenyl-[β-(tosyloxy)-β-*n*-butylvinyl]iodonium tosylate separated: mp 167–168.5 °C dec; ^1H NMR ($CDCl_3$) δ 0.51–1.58 (closely spaced m's, 7.3 H), 2.18–2.84 (m's, 7.9 H, singlets at 2.28 and 2.38), 6.68–8.01 (m, 13.8 H); ^{13}C NMR ($CDCl_3$) (ORD multiplicity) 13.51 (q), 21.34 (m), 21.83 (m), 28.44 (t), 34.40 (t), 75.59, 77.19, 78.79; $CDCl_3$), 90.86 (d), 114.79 (s), 126.06–135.09 (m), 139.47 (s), 143.00 (s), 147.00 (s), 162.16 (s); ^{13}C NMR APT δ 13.51 (u), 21.34 (u), 21.83 (d), 28.44 (d), 34.40 (d), (75.59, 77.19, 78.79 all d), 90.86 (u), 114.79 (d), 126.06–135.09 (u and d), 139.47 (d), 143.00 (d), 147.00 (d), 162.16 (d).

Anal. Calcd for $C_{26}H_{29}S_2O_6I$: C, 49.68; H, 4.65; I, 20.19. Found: C, 49.75; H, 4.86; I, 20.32.

The mother liquor was concentrated to yellow oil. The oil was dissolved in warm Et_2O (10 mL), and the solution was allowed to stand for 12 h at room temperature whereupon the crystallization of 1.53 g (24.3%) of (*Z* or *E*)-phenyl[β-(tosyloxy)-β-*n*-butylvinyl]iodonium tosylate occurred: mp 140–143 °C; ^1H NMR ($CDCl_3$) δ 0.47–1.50 (closely spaced m's, 7.5 H), 2.13–2.67 (closely spaced m's, 7.9 H, singlets at 2.27 and 2.32), 6.73–8.07, 13.6 H); ^{13}C NMR ($CDCl_3$) (ORD multiplicity) δ 13.66 (q), 21.28 (m), 21.71 (m), 22.04 (m), 28.00 (t), 34.65 (t), (75.60 , 77.19, 78.80; $CDCl_3$), 92.56 (d), 115.98 (s), 126.04–136.08 (m), 139.58 (s), 142.46 (s), 146.09 (s), 162.24 (s); ^{13}C NMR APT δ 13.66 (u) 21.28 (u), 22.04 (d), 28.00 (d), 34.65 (d), (75.60, 77.19, 78.80 all d), 92.56 (u), 115.98 (d), 126.04–136.08 (u and d), 139.58 (d), 142.46 (d), 146.09 (d), 162.24 (d).

Anal. Calcd for $C_{26}H_{29}S_2O_6I$: C, 49.68; H, 4.65; I, 20.19. Found: C, 49.51; H, 4.76; I, 20.30.

1-Heptyne. The oil was dissolved in boiling acetone (20 mL)

and slowly cooled to −20 °C whereupon 1.66 g (25.8%) of (*E* or *Z*)-phenyl[β-(tosyloxy)-β-*n*-pentylvinyl]iodonium tosylate separated: mp 152–153 °C; ^{1}H NMR ($CDCl_3$) δ 0.51–1.54 (closely spaced m's, 9.0 H), 2.19–2.71 (closely spaced m's, 7.8 H, singlets at 2.31 and 2.42), 6.71–8.04 (m, 14.2 H).

Anal. Calcd for $C_{27}H_{31}S_2O_6I$: C, 50.47; H, 4.86; I, 19.75. Found: C, 50.46; H, 4.88; I, 19.94.

The mother liquor was concentrated to a yellow oil. The oil was then taken up in warm Et_2O (13 mL), and the solution was slowly cooled to −20 °C whereupon 1.75 g (25.8%) of a solid product separated: ^{1}H NMR ($CDCl_3$, relative areas based on formulation of product as phenyl[β-(tosyloxy)-β-*n*-pentylvinyl]-iodonium tosylate·$2H_2O$) δ 0.55–1.35 (m, 8.5 H), 1.43 (s, 5.0 H, some overlap with preceeding multiplet), 2.05–2.78 (m, 7.9 H, prominent singlets at δ 2.31 and 2.35), 6.85–8.05 (m, 13.7 H).

Anal. Found: C, 51.54; H, 5.57; I, 21.10. Calcd for dihydrate: C, 47.70; H, 5.21; I, 18.70.

3-Methyl-1-butyne. The oil was washed with Et_2O (2 × 10 mL) and crystallized from Et_2O/acetone (30 mL; 5/1) to give 0.351 g (11.4%) of phenyl[β-(tosyloxy)-β-isopropylvinyl]iodonium tosylate; mp 168–172 °C. Recrystallization of 0.288 g from acetone/CH_2Cl_2 (10 mL; 9/1) returned 0.257 g: mp 175.5–177 °C; ^{1}H NMR ($CDCl_3$) δ 0.73, (d, 5.6 H), 2.30 and 2.35 (singlets, 5.7 H), 3.17 (m, 1.4 H), 6.85–8.15 (m, 14.3 H); ^{13}C NMR ($CDCl_3$) (ORD multiplicity) δ 19.05 (m), 21.38 (m), 21.81 (m), 34.70 (d), (75.60, 77.19, 78.79; $CDCl_3$), 88.18 (d), 115.96 (s), 126.19–134.81 (m), 139.64 (s), 142.65 (s), 146.23 (s) 165.46 (s); IR (CH_2Cl_2) *no* C≡C peak.

Anal. Calcd for $C_{25}H_{27}IS_2O_6$: C, 48.86; H, 4.43; I, 20.65. Found: C, 48.94; H, 4.45; I, 20.51.

The mother liquor from the first crystallization was diluted with Et_2O (ca. 30 mL) and cooled at −20 °C, resulting in the separation of 0.650 g (14.7%) of phenyl (β-isopropylethynyl)-iodonium tosylate; mp 109–112 °C dec. Recrystallization returned 0.529 g: mp 115–118 °C dec; ^{1}H NMR ($CDCl_3$) δ 1.13 (d, $J \simeq$ 7 Hz, 5.7 H), 2.33 and 2.78 (s and m, 4.1 H), 6.90–8.07 (m, 9.2 H); IR (CH_2Cl_2) 2180 cm^{-1} (C≡C).

Anal. Calcd for $C_{18}H_{19}ISO_3$: C, 48.88; H, 4.33; I, 28.69. Found: C, 48.79; H, 4.40; I, 28.36.

3-Methyl-1-pentyne. Trituration of the yellow oil with Et_2O (ca. 20 mL) gave 2.303 g (50.5%) of phenyl (β-*sec*-butylethynyl)iodonium tosylate; mp 101.5–110 °C. Recrystallization of 1.056 g from Et_2O/CH_2Cl_2 (33 mL; 10/1) at −20 °C returned 0.982 g: mp 114–116 °C dec; ^{1}H NMR ($CDCl_3$) δ 0.40–1.80 (m's, 7.8 H), 2.10–2.80 (m with s at 2.27, 3.9 H), 6.73–8.10 (m, 9.3 H); IR (CH_2Cl_2) 2180 cm^{-1} (C≡C).

Anal. Calcd for $C_{19}H_{21}SO_3I$: C, 50.01; H, 4.64; I, 27.81. Found: C, 50.00; H, 5.01; I, 27.67.

4-Methyl-1-pentyne. Crystallization of the yellow oil from Et_2O/acetone (26 mL; 18/8) at 0 °C yielded 1.80 g (28.6%) of phenyl[β-isobutyl-β-(tosyloxy)vinyl]iodonium tosylate; mp 144–147 °C. Recrystallization of 1.668 g from acetone (30 mL) at 0 °C returned 1.33 g: mp 148–152 °C; ^{1}H NMR ($CDCl_3$) δ 0.71 (d, 5.9 H), 1.67 (br m, 1.4 H), 2.30, 2.34, 2.40 (overlapping s, s and d, 7.5 H), 6.73–8.20 (m, 14.2 H); ^{13}C NMR APT ($CDCl_3$) [both *E* and *Z* isomers are present] δ 21.26 (u), 21.77 (possibly two peaks superimposed) (u), 26.33 (u), 43.08 and 43.27 (d) (75.59, 77.19, 78.79; $CDCl_3$), 92.01 and 93.20 (u), 114.97 and 116.04 (d), 126.02–135.11 (u except for δ 132.12 and δ 131.89 d), 139.41 (d), 142.83 (d), 146.10 and 146.92 (d), 160.93 and 161.44 (d).

Anal. Calcd for $C_{26}H_{29}S_2O_6I$: C, 49.68; H, 4.65; I, 20.19. Found: C, 49.77; H, 4.65; I, 20.39.

The Et_2O/acetone mother liquor was diluted with Et_2O (ca. 30 mL) and cooled at 0 °C for 3 h to give 2.03 g of phenyl(β-isobutylethynyl)iodonium tosylate; mp 96–98.5 °C dec. When cooled at −20 °C, the filtrate yielded 1.016 g more of the alkynyliodonium tosylate: mp 99–101 °C dec; total yield, 3.05 g (33.4%); ^{1}H NMR ($CDCl_3$) δ 0.88 (d, $J \sim$ 7 Hz, 5.6 H), 1.40–2.03 (m, 1.5 H), 2.17–2.57 (d centered on s at 2.31, 4.7 H), 6.78–8.27 (m, 9.2 H); IR (CH_2Cl_2) 2180 cm^{-1} (C≡C).

Anal. Calcd for $C_{19}H_{21}SO_3I$: C, 50.01; H, 4.64; I, 27.81. Found: C, 49.78; H, 4.63; I, 26.05.

Cyclohexylacetylene. Crystallization of the yellow oil from Et_2O/pentane (30 mL; 25/5) at −20 °C gave 4.80 g (47.2%) of phenyl(β-cyclohexylethynyl)iodonium tosylate: mp 127–128 °C; ^{1}H NMR ($CDCl_3$) δ 0.80–1.93 (br envelope, 10.1), 2.23 and 2.55 (s and featureless m, 4.0 H), 6.77–8.10 (m, 8.9 H); IR (CH_2Cl_2) 2160 cm^{-1} (C≡C).

Anal. Calcd for $C_{21}H_{23}ISO_3$: C, 52.29; H, 4.81; I, 26.31. Found: C, 52.52; H, 4.93; I, 26.26.

3,3-Dimethyl-1-butyne. Treatment of the yellow oil with warm Et_2O (40 mL) gave 6.76 g (74.1%) of phenyl(β-*tert*-butylethynyl)iodonium tosylate: mp 137.5–139 °C; ^{1}H NMR ($CDCl_3$) δ 1.16 (s, 8.7 H), 2.27 (s, 3.0 H), 6.83–8.07 (m, 9.3 H); IR (CH_2Cl_2) 2160 and 2195 cm^{-1} (C≡C).

Anal. Calcd for $C_{19}H_{21}SO_3I$: C, 50.01; H, 4.64; I, 27.81. Found: C, 50.04; H, 4.68; I, 27.75.

Phenylacetylene. The reddish brown oil was dissolved in warm Et_2O (20 mL), and the solution was kept at room temperature whereupon 8.72 g (61.0%) of phenyl(β-phenylethynyl)iodonium tosylate separated within 2 h: mp 119–122 °C dec; ^{1}H NMR ($CDCl_3$) δ 2.25 (s, 2.9 H), 6.79–8.19 (m, 14.1 H); IR (film) 2155 cm^{-1} (C≡C).

Anal. Calcd for $C_{21}H_{17}SO_3I$: C, 52.95; H, 3.60; I, 26.64. Found: C, 52.82; H, 3.63; I, 26.41.

(Trimethylsilyl)acetylene. A mixture of 0.72 g (7.3 mmol) of (trimethylsilyl)acetylene, 1.96 g (5.00 mmol) of [hydroxy(tosyloxy)iodo]benzene, and 15 mL of $CHCl_3$ was stirred and heated under reflux for 2 days.

The resulting slightly yellow solution was washed with H_2O (2 × 10 mL), dried over Na_2SO_4, and concentrated under aspirator vacuum to an oil. Crystallization of the oil from Et_2O/CH_2Cl_2 (20 mL; 15/5) gave 0.320 g (22.4%) of phenyl[β-(toxyloxy)-vinyl]iodonium tosylate: mp 146–148 °C; ^{1}H NMR ($CDCl_3$) δ 2.31 and 2.37 (two singlets, 5.8 H), 6.50–8.07 (m, 15.2 H).

Anal. Calcd for $C_{22}H_{21}IS_2O_6$: C, 46.16; H, 3.70; I, 22.17. Found: C, 46.31; H, 3.81; I, 21.90.

Registry No. **4**, 27126-76-7; (*E*)-**5** (R = Pr), 92473-25-1; (*Z*)-**5** (R = Pr), 92473-27-3; (*E*)-**5** (R = Bu), 92473-29-5; (*Z*)-**5** (R = Bu), 92473-31-9; **5** (R = $CH_3(CH_2)_4$), 79069-26-4; **5** (R = *i*-Pr), 92473-33-1; (*E*)-**5** (R = *i*-Bu), 92473-35-3; (*Z*)-**5** (R = *i*-Bu), 92473-37-5; **5** (R = H), 92473-39-7; **6** (R = *i*-Pr), 92473-41-1; **6** (R = *sec*-Bu), 92473-43-3; **6** (R = *i*-Bu), 92473-45-5; **6** (R = c-C_6H_{11}), 79069-34-4; **6** (R = *t*-Bu), 92473-47-7; **6** (R = Ph), 79069-32-2; $CH_3(CH_2)_2C{\equiv}CH$, 627-19-0; $CH_3(CH_2)_3C{\equiv}CH$, 693-02-7; $CH_3(CH_2)_4C{\equiv}CH$, 628-71-7; *i*-PrC≡CH, 598-23-2; *sec*-BuC≡CH, 922-59-8; *i*-BuC≡CH, 7154-75-8; c-$C_6H_{11}C{\equiv}CH$, 931-48-6; *t*-BuC≡CH, 917-92-0; PhC≡CH, 536-74-3; $Me_3SiC{\equiv}CH$, 1066-54-2.