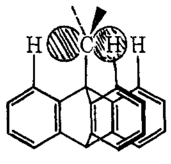

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sibly by capture of **2** prior to rearrangement by chloride ion. In our case, **2** upon formation is surrounded by a potential hydride donor solvent and formation of **1**-H is thereby favored, all the more so because eq 1 should be sizably exothermic.

Finally, the relationship of **1**-Br to **5** is reminiscent of the similar relationship between 1-triptycyl and triphenylmethyl halides[11] and demonstrates once more the dramatic effect of pinning back the aromatic rings in these compounds.

Experimental Section

General.—Microanalyses were done by Micro-Tech Laboratories, Skokie, Ill., and by M-H-W Laboratories, Garden City, Mich. Spectral data were obtained on Varian A-60A (nmr, $CDCl_3$ solutions) and Beckman IR-5A (ir, KBr discs) instruments.

1-Bromomethyltriptycene (1-Br).—Reaction of yellow 9-bromomethylanthracene, mp 145–147° (lit.[12] mp 137.5–142° dec), anthranilic acid, and isoamyl nitrite in dioxane, as described for similar preparations,[13] led to colorless **1**-Br: 39.5% on a 22-mmol scale; mp 217–218.5° from benzene–petroleum ether (bp 30–60°); nmr δ 7.5 (m), 7.0 (m, ArH), 5.37 (s, bridgehead H), 4.85 (s, CH_2Br).

Anal. Calcd for $C_{21}H_{15}Br$: C, 72.63; H, 4.35. Found: 72.61; H, 4.31.

Crude product from this reaction was yellow. Purification was tedious, requiring chromatography on silica gel for final processing. Use of carboxybenzenediazonium chloride[14] as the benzyne precursor in this reaction gave **1**-Br contaminated with **1**-Cl (82.5:17.5), probably *via* some prior conversion of 9-bromomethylanthracene to its 9-chloro analog by chloride ion displacement. Chloride **1**-Cl was apparent from its $-CH_2Cl$ resonance at δ 5.07.[3]

2-Chloro-1,1,1-triphenylethane (5).—The chloride was prepared as reported,[15] mp 99–101° (lit.[14] mp 101.0–101.8°), nmr δ 7.33 (s, ArH), 4.67 (s, CH_2Cl).

Solvolysis Studies.—*m*-Cresol was purified by distillation from zinc dust, bp 50–52° (0.5 mm), homogeneous by glpc. The solvolysis was conducted on *ca.* 0.02 *M* solutions of purified **1**-Br in *m*-cresol sealed in Carius tubes, following closely a reported procedure.[5] A Carius tube furnace equipped with a thermocouple for temperature measurement was used. The reactions were carried to ~80% completion and processed as reported.[5] The liberated bromide was titrated potentiometrically at 25° with standard 80% ethanolic silver nitrate (0.010 *M*), using a Leeds and Northrup Model 7402 pH meter. The kinetic data are given in Table I.

The solvolysis product from **1**-Br was isolated from the titrated samples by removal of silver bromide by filtration and *m*-cresol by codistillation with water followed by chromatography of the residue on a silica gel column. Elution with petroleum ether (bp 30–60°) gave **3,6-dimethylxanthene** (**6**): 16.5% based on **1**-Br; mp 195–200° (lit.[16] 197.5–203.5°); ir, nmr, and uv spectra agreed with those reported;[16] mass spectrum (70 eV) *m/e inter alia*, 210 (P), 209 (P − 1), 195 (P − CH_3). Elution with benzene–petroleum ether gave **1-methyltriptycene** (**1**-H, 31% based on consumed **1**-Br, melting point, mixture melting point with authentic sample, and nmr spectrum agreed with those reported[3]). No other characterizable products were eluted. No homotriptycyl products were detected. A control study of *m*-cresol itself at 370° for 6 hr afforded no **6**. Degassed reaction conditions showed no difference.

Chloride **5** was solvolyzed analogously. Sealed ampoules containing *ca.* 0.02 *M* solutions of **5** in *m*-cresol with an equimolar amount of redistilled 2,4-lutidine added were held at various temperatures. Processing and chloride determination were as described above. See Table I for further details. From reactions taken to *ca.* 80% completion, the only product isolated (chromatography on silica gel) was **triphenylethylene**, 95% based on consumed **5**, mp and mmp with authentic material 67.5–68.5°, coincidental ir and nmr spectra.

Miscellaneous.—Among the triptycenes prepared in this study were those following. Their syntheses followed standard or cited procedures and their properties are briefly reported here for documentation purposes.

1-Diazoacetyltriptycene was yellow: mp 220–222° dec; 89% from 9-triptoyl chloride and diazomethane in ether; λ 4.8, 6.14 ($COCHN_2$); nmr δ 8.02 (m, 3, peri ArH's), 7.45 (m), 7.08 (m, remaining ArH's), 5.80 (s, $-CHN_2$), 5.42 (s, bridgehead H).

Anal. Calcd for $C_{22}H_{14}ON_2$: C, 81.97; H, 4.37; N, 8.69. Found: C, 81.67; H, 4.40; N, 8.41.

1-Chloroacetyltriptycene was colorless: mp 200–202°; 85% from reaction of the diazo ketone above and hydrogen chloride[17] in tetrahydrofuran at 50°; λ 5.82 (CO); nmr δ 7.75 (m, 3, peri ArH's), 7.50 (m), 7.10 (m, remaining ArH's), 5.40 (s, bridgehead H), 4.80 (s, $-CH_2Cl$).

Anal. Calcd for $C_{22}H_{15}OCl$: C, 79.88; H, 4.57. Found: C, 80.07; H, 4.59.

1-Triptycylacetic acid was colorless: mp 298–300°; 10% from the above diazo ketone upon uv irradiation in 20% aqueous tetrahydrofuran;[18] λ 3.3 (broad) 5.82 (COOH); nmr δ 9.6 (broad s, COOH), 7.33 (m), 7.03 (m, ArH), 5.40 (s, bridgehead H), 4.03 (s, CH_2).

Anal. Calcd for $C_{22}H_{16}O_2$: C, 84.59; H, 5.16. Found: C, 84.23; H, 5.33.

Attempted conversion of the diazo ketone above to this acid using silver benzoate and triethylamine in methanol[19] followed by saponification gave intractable material. Reaction of silver 1-triptycylacetate with bromine in carbon tetrachloride to form **1**-Br seemed partially successful. However, the easier preparation given above made further work on this reaction unnecessary.

Registry No.—**1** (X = Br), 34858-83-8; **5**, 33885-01-7; *m*-cresol, 108-39-4; triphenylethylene, 58-72-0; 1-diazoacetyltriptycene, 34887-50-8; 1-chloroacetyltriptycene, 34858-85-0; 1-triptycylacetic acid, 34858-86-1.

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Intramolecular Addition of 4-Alkynyloxy Radicals

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It was recently reported by Rieke and Cooke, that alkoxy radicals fail to add intramolecularly to alkynes.[1] Analysis of the photolysis products of several 4-alkynyl nitrites has provided us with evidence for the occur-

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rence of this reaction in low yield. Whereas numerous studies have been devoted to free-radical chemistry of alkenes, little seems to be known on the behavior of alkynes,[2a] particularly toward alkoxy radicals.[3] As an example, the interaction of *tert*-butyl hypobromite or hypochlorite with alkynes leads to an explosive homolytic decomposition which is apparently induced by the triple bond. Abstraction of the propargylic hydrogen by alkoxy radicals is the sole reaction generally observed.[4] However some additions to the triple bond occur in the case of conjugated enynes only.[5] Nevertheless, although hydrogen abstraction is impossible, addition products do not form with phenylacetylene.[4a]

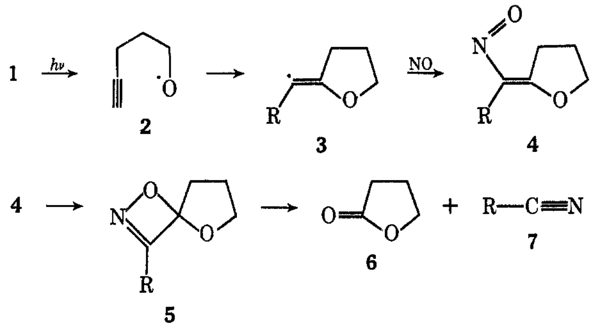
Surprisingly enough, in the case of 4-alkenyloxy radicals the preferred reaction is an intramolecular addition,[6] whereas allylic hydrogen abstraction occurs essentially in intermolecular reactions.[3] On the other hand interesting synthetic reactions could be achieved by intramolecular addition of carbon,[2b,7] and thiyl radicals[9] on alkynes. Accordingly it was of interest to investigate the intramolecular interaction of a nonconjugated triple bond with alkoxy radicals obtained by photolysis of 4-alkynyl nitrites

$$R{-}C{\equiv}C{-}(CH_2)_3{-}O{-}NO$$

1

Results and Discussion

Nitrogen was slowly bubbled through a 0.05–0.1 *M* benzene solution of the nitrites **1**, which was irradiated with a Hanau TQ 81 (70 W) high-pressure lamp equipped with a Pyrex filter. The same compound, identified as γ-butyrolactone by comparison with an authentic sample, was obtained in poor yields (1–6%) from the three nitrites under investigation (R = H, CH_3 or C_6H_5). In order to explain these results we suggest the following scheme.



Irradiation of nitrite **1** yields the alkoxy radical **2** which adds intramolecularly to the triple bond. The vinylic cyclic radical **3** is trapped by nitric oxide to form the nitroso compound **4**. Such nitrosovinylic intermediates have been shown to be unstable by a study of photolytic intramolecular addition of alkyl and chlorine radicals to alkynes in presence of nitric oxide.[10] Indeed, in the present case they fragment into γ-butyrolactone **6** and nitrite **7**. When R = C_6H_5, benzonitrile is detected. This is in agreement with the present mechanism involving an intermediate such as **5**.

In all the cases the other products which are normally expected from the evolution of the alkoxy radical **2**[3] as 4-alkynols (R = H (30–35%), CH_3 (45–50%), and C_6H_5 (50–55%)) were identified besides approximately 25% polymeric material. For R = H 4-pentynal was detected together with four as yet unidentified compounds (less than 10% by vpc). The reaction was very sensitive to experimental conditions. For instance, when the nitrogen flow rate was increased the yield of γ-butyrolactone decreased (when R = H). The use of a Hanau TQ 150 (150 W) lamp lowered the yield of γ-butyrolactone; this could be an explanation for the somewhat different results reported by Rieke and Cooke[1] who used a 450-W lamp. Nitric oxide was bubbled through the solution in order to more efficiently trap **3** and get better yields of **4**; this experiment was unsuccessful since the quantities of γ-butyrolactone formed were not modified. However, this failure might be due to competitive reactions between nitric oxide and the alkyne.[11]

These results strongly support the possibility of intramolecular addition of alkoxy radicals to isolated triple bonds. However, the low yields of cyclic products show that this reaction is more difficult than with a double bond. As a conclusion, we wish to point out that five-membered rings are obtained with substituted alkynes (R = CH_3 or C_6H_5). With monosubstituted alkynes (R = H) no hypothesis on the orientation of the cyclization can be made, as several products need to be identified.

Experimental Section

The ir spectra were measured with a Perkin-Elmer 337 grating ir spectrophotometer. The nmr spectra were obtained on Varian A-60 and HA-100 instruments, chemical shifts were recorded as δ values (parts per million) relative to tetramethylsilane as an internal reference.

4-Pentynol, bp 67° (15 mm) [lit.[12] bp 70–71° (29 mm)], was prepared from tetrahydrofurfuryl chloride according to "Organic Syntheses."[12]

4-Hexynol, bp 78° (13 mm), $n^{18}\mathrm{D}$ (1.4602 [lit.[13] bp 85° (20 mm), $n^{18}\mathrm{D}$ 1.4604)] was prepared according to a procedure described for 4-undecynol[14] from lithium (7 g), excess of propyne, and 3-bromopropanol yielding 11.4 g of 4-hexynol (46%).

1-Phenyl-1-pentyn-5-ol was prepared according to a procedure described for 5-hexynol[15] using the reaction pathway chloride, iodide, acetate, and alcohol. 5-Chloro-1-phenyl-1-pentyne was prepared in 70% yield (125 g), bp 146° (15 mm), from lithium (8 g), phenylacetylene (112 g), and 3-bromo-1-chloropropane

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(157 g) in 1 l. of liquid ammonia. A mixture of 3-bromo-1-chloropropane (125 g) and sodium iodide (110 g) was refluxed and stirred for 24 hr in 1 l. of dried acetone. The excess of solvent was then distilled and the cooled residue treated with water. The upper layer was washed, dried, and rectified to yield 140 g (74%) of 5-iodo-1-phenyl-1-pentyne, bp 95–100° (0.05 mm). The iodide (43 g) was added while stirring to silver acetate (27 g) in 150 ml of benzene. After refluxing for 6 hr the cooled mixture was filtered and the benzene was evaporated. Distillation gave 25 g (72%) of 5-acetoxy-1-phenyl-1-pentyne, bp 100–105° (0.05 mm). The ester (25 g) was heated at reflux for 2 hr in a solution of potassium hydroxide (12 g) in water (20-ml)–ethanol (50 ml). Ethanol was removed by distillation and the residue was extracted with ether. The ether extract was washed with dilute acid, water, and dried. Distillation gave 11 g (53%), of 1-phenyl-1-pentyn-5-ol: bp 100–104° (0.04 mm), n^{20}_{D} 1.5765 [lit.[16] bp 122° (2 mm), n^{20}_{D} 1.5769]; ir (neat) 3500, 3200, 3080, 3020, 2220, 1600, 1500, 1060, 750, 690 cm^{-1}; nmr (CCl_4) 1.75 (q, J = 6.5 Hz, 2 H) 2.4 (t, J = 6.5 Hz, 2 H), 3.65 (t, J = 6.5 Hz, 2 H), 4.05 (s, 1 H), 7.1 (m, 5 H).

Preparation of 4-Alkynyl Nitrites.—They were prepared, like alkenyl nitrites,[6c] by alkynol esterification with nitrous acid at 0°.[17] Alkynol (0.2 mol) and sodium nitrite (21 g) were dissolved in water (75 ml). Concentrated sulfuric acid (15 g) in water (10 ml) slowly added with vigorous stirring to the solution maintained at 0° with external cooling and swept by a nitrogen stream. The upper layer was dried and 4-alkynyl nitrites $RC{\equiv}C(CH_2)_3$-ONO (**1**) distilled at temperature below 50°. **1a**: (R = H) (70%); bp 35° (25 mm); n^{20}_{D} 1.4168; ir (neat) 3300, 2110, 1640, 1600, 780 cm^{-1}. **1b** (R = CH_3) (66%): bp 42° (15 mm); n^{25}_{D} 1.4309; ir (neat) 2210, 1640, 1600, 700 cm^{-1}. **1c** (R = C_6H_5) (85%, crude because it decomposed by distillation): ir (neat) 2220, 1640, 1600, 790. [All these compounds have characteristic uv absorption spectra of nitrites[18] between 320 and 380 nm (hexane).]

Photolysis of 4-Alkynyl Nitrites.—The nitrite (0.1–0.05 mol) dissolved in 100 ml of benzene was added during 2 hr to 900 ml of benzene irradiated by an inside Hanau TQ 81 lamp provided with a Pyrex filter. A slow stream of nitrogen was maintained before and during the irradiation. The solution was maintained between 10 and 15° by external cooling. The photolysis was followed by uv spectra and carried to 80% completion. Benzene was removed under reduced pressure at temperature below 50°. The residue was distilled and fractions analyzed by vpc (Carbowax 20M). Compounds were isolated by preparative vpc and identified by comparative spectral analysis with authentic samples. Yields were calculated from the weight of nitrite ester used.

Photolyses were also run in the cavity of an epr (Varian E_3) apparatus irradiated with an SP 500 Philips lamp. Spectra of nitroxides were observed but these spectra were complex and important modification were observed during and after irradiation, not permitting yet, direct verification of the mechanism proposed as in the photolysis of 4-alkenyl nitrites.[6d]

Photolysis of 4-Hexynyl Nitrite.—As described above 9 g (0.07 mol) of the nitrite was irradiated in 1 l. of benzene for 20 hr. Distillation gave 3.7 g, bp 75–90° (13 mm), and undistillable residue, 1.84 g (21%). Only two compounds could be detected by vpc of the distilled fraction; they were identified after vpc preparative and comparison with authentic samples of 4-hexynol (47%) and γ-butyrolactone (6%).

Photolysis of 5-Phenyl-4-pentynyl Nitrite.—An amount of 9.9 g (0.048 mol) was irradiated in 1 l. of benzene for 60 hr. Distillation gave a first fraction, 0.1 g, bp 36–40° (0.05 mm), a second fraction, 1.9 g, bp 110–115° (0.2 mm), and an undistillable residue, 2 g (20%). Only two compounds were detected in the first fraction, identified as γ-butyrolactone (1%) and benzonitrile (1%). 1-phenyl-1-pentyn-5-ol was the major product of the second fraction (57%).

Photolysis of 4-Pentynyl Nitrite.—As above 6.7 g (0.059 mol) of the nitrite was irradiated for 18 hr. Distillation gave fraction 1, 1.8 g, bp 50–65° (13 mm), fraction 2, 0.7 g, bp 60–65° (0.2 mm), and an undistillable residue, 1.6 g (24%). Fraction 1 was composed of 4-pentynol (32%) and traces of 4-pentynal and unreacted nitrite ester. Fraction 2 was composed of six compounds. Two of them were identified as 4-pentynol (6%) and γ-butyrolactone (2%). The photolysis of 4.13 g (0.047 mol) of 4-pentynyl nitrite in 250 ml of benzene as above but with a plunging lamp, Hanau TQ 150 (150 W), gave still two fractions: fraction 1, 0.7 g, bp 32–50° (0.05 mm), fraction 2, 0.7 g, bp 50–80° (0.05 mm), undistillable residue, 1 g (24%), 4-pentynal (4%), 4-pentynol (16%), and traces of nitrite ester composed the first fraction. Six compounds were present in the second fraction, two of them were identified as 4-pentynol (7%) and γ-butyrolactone (traces).

Photolysis of 7.83 g of nitrite ester in 1 l. of benzene with a TQ 81 lamp as above but with a quicker stream of nitrogen gave 4-pentynal (8%) and 4-pentynol (23%) in fraction 1. In fraction 2, the proportion of two unidentified compounds increased but γ-butyrolactone could only be detected. When the stream of nitrogen was replaced by nitric oxide we observed the formation of γ-butyrolactone (2.2%), 4-pentynal (traces), 4-pentynol (10%), four unidentified compounds, and polymeric material (40%).

Registry No.—**1a** (R = H), 30428-24-1; **1b** (R = CH_3), 34886-47-0; **1c** (R = Ph), 34886-48-1; 5-chloro-1-phenyl-1-pentyne, 24463-87-4; 5-iodo-1-phenyl-1-pentyne, 34886-50-5; 5-acetoxy-1-phenyl-1-pentyne, 29313-49-3; 1-phenyl-1-pentyn-5-ol, 24595-58-2.

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A Terminology for the Chiral Attributes of Steric Elements[1]

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In a recent analysis of stereoisomerism we concluded[2] that the conventional types of stereoisomerism (the center, axis, plane, "conformational helix," and cis–trans isomerism at double bonds)[3,4] could be reduced to two elements, the center and the line of torsion, and that these elements of stereoisomerism may possess or lack one or both of two distinct chiral characteristics. The first of these determines whether the configuration of the element by itself has to be specified with a chiral descriptor and the second whether the element can contribute to the chirality of a compound. Either of these tests may be thought to be suitable for determining the chiral character of the element. We suggested, at least as a temporary expedient, to call an element chiral if it meets both of these tests, as this would preserve existing practices. The problem of selecting the most useful criterion for a chiral element, however, remained unsolved. We now find that the need for making this difficult choice would be avoided

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