complexes of the form [propene $PhOHD^+$]. While such a mechanism might conceivably account for additional randomization within the alkyl chain of deuterated *n*-propyl phenyl ethers all by itself, the observed proportions of scrambling are more easily fit by a pathway that includes complexes of the form [iPr^+ phenol] (which scramble all seven of the alkyl hydrogens) as intermediates. Ring hydrogens do not participate in the scrambling. Our analyses of the experiments presented here contrast two mechanisms, both of which invoke gas-phase ion–neutral complexes that resemble ion–molecule pairs formed in S_N1 reactions.[17] One mechanism involves a heterolysis corresponding to reaction 1, whose similarity to the first step of solvolytic elimination has been discussed for more than a decade. The alternative, elimination–readdition, does not bear a formal resemblance to solvolysis; this mechanism is ruled out unless direct elimination is presumed to occur concurrently in competition with ion–neutral complex formation. The quantitative data at hand are well fit as a "gas-phase solvolysis" using a reasonable kinetic scheme with three relative rate constants. While the elimination–readdition mechanism alternative represents a plausible sequence of steps, it can be made to fit the experimental observations only if four relative rate constants are used as parameters (since the ion neutral complexes in eq 4 scramble only six of the alkyl hydrogens). We conclude that parallels between the gas phase and solution are aptly drawn and that studies of cation chemistry in one regime will continue to illumine the other.

Acknowledgment. Support of this work by NSF grant CHE8802086b is gratefully acknowledged.

Supplementary Material Available: Algebraic derivations of steady state expressions for Schemes I–IV and expressions for *m/z* 95:*m/z* 96 predicted for M + 2 ions of d_0 (4 pages). Ordering information is given on any current masthead page.

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Synthesis, Structure, and Some Properties of 2,5,7,10-Tetraiodo-1,6-methano[10]annulene

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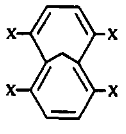
The title compound (**1a**) was prepared by a two-step procedure via an organomercurial. Both **1a** and its 11,11-difluoro analogue are crystalline solids that were fully characterized. The tetraiodide **1a** is unusual in that the aromatic character of the π periphery is almost destroyed as determined from ring current effects on the 11,11 hydrogen atoms. The crystal structure of **1a** is reported and it bears out the "localization" of the π electrons with a concomitant opening of the transannular bridge angle.

Introduction

The subtle interplay between structural parameters and electronic character at the molecular level is a subject of ongoing interest in organic chemistry.[1] The 1,6-methano[10]annulene is a particularly interesting molecule from the point of view of the effect of structural parameters on the electronic delocalization of its 10 π electrons. Sterically demanding substituents in the 2,5,7,10-positions are expected to have a deleterious effect on cyclic delocalization in this bicycle. Trimethylsilyl substituents, indeed, distort the ring sufficiently to cause electronic localization.[2]

In this paper we describe the preparation and full characterization of the previously unknown title compound **1a**, prepared by a route not previously applied to the methano[10]annulenes and described below. The reactivity of the "peri" positions toward coupling reactions was expected to be enhanced in the title compound. By comparison with the parent and tetrakis(trimethylsilyl) derivative, we find that the tetraiodo derivative shows bond localization but the bond alteration is not as severe as in the case of the tetrakis(trimethylsilyl) derivative. This "intermediate localization" is also substantiated in the ^1H NMR chemical shift of the methano bridge.



1a, X = I
1b, X = Br
1c, X = TMS

Experimental Section

General. All reactions were performed under an inert atmosphere. Commercial solvents were dried by using standard conditions, e.g. CaH_2 for acetonitrile, etc. IR spectra were recorded with a Perkin-Elmer Model 1300 spectrophotometer under a nitrogen flow as KBr disks (unless stated otherwise) and are reported in ν (cm^{-1}). NMR spectra were recorded with a General Electric GN500, 500-MHz instrument. Data are reported in δ relative to TMS (internal) in d_8-THF, unless stated otherwise. Melting points were obtained with a Büchi oil bath apparatus and are reported uncorrected.

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‡ Institut für Organische Chemie der Universität, Greinstrasse 4, D-5000, Köln 41, Federal Republic of Germany.

[1] Inter alia: Isaacs, N. S. *Physical Organic Chemistry*; Wiley: New York, 1987. Lowry, T. H.; Richardson, K. S. *Mechanism and Theory in Organic Chemistry*, 2nd ed.; Harper & Row: New York, 1981.

[2] Neidlein, R.; Wirth, W.; Gieren, A.; Lamm, V.; Hübner, T. *Angew. Chem.* **1985**, *97*, 580.

2,5,7,10-Tetrakis(acetoxymercurio)-1,6-methano[10]-annulene. A solution of 1.704 g (12 mmol) of 2 (kindly supplied by Prof. E. Vogel, Cologne) in 75 mL of absolute methanol was added dropwise to a solution of 15.03 g (48 mmol) of mercuric acetate in 300 mL of absolute methanol. After ca. 30 min, a light yellow precipitate separated, but in a few hours a more voluminous precipitation took place. After the reaction mixture was allowed to stir for 4 days at room temperature, it was passed through a medium frit glass funnel, the filtrate was discarded, and the solid was washed with small amounts of dry methanol. Drying the off-white solid in vacuo afforded 8.6–10 g (60–70%, three different runs). The material was used in the next step without further manipulation. The tetramercurial is insoluble in all common solvents and is slowly attacked by extremely polar solvents such as DMSO. Its structure was proved by conversion to the known tetrabromomethano[10]annulene.

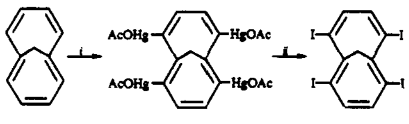
2,5,7,10-Tetrabromo-1,6-methano[10]annulene[3] (1b). The above mercurial (1.18 g, 1 mmol) was suspended in 300 mL of dry acetonitrile and treated dropwise, at ambient temperature and with vigorous stirring, with a solution of 800 mg of bromine in 100 mL of dry acetonitrile. As the reaction proceeds, most of the solid dissolves to produce a dark yellow solution with some suspended solid. After overnight stirring, the mixture was filtered, the filtrate was evaporated, and the residue was chromatographed on neutral alumina (act. III, 30 × 2.5 cm), using hexane as eluent. The yellow fraction afforded 0.1 g (22%) of pure 2,5,7,10-tetrabromo-1,6-methano[10]annulene with spectroscopic properties (NMR, IR, LRMS) identical with an authentic sample.

2,5,7,10-Tetraiodo-1,6-methano[10]annulene (1a) and 2,5,7-Triiodo-1,6-methano[10]annulene. The above mercurial (7.05 g, 6 mmol) was suspended in 1200 mL of dry acetonitrile and treated dropwise, at ambient temperature and with vigorous stirring, with a solution of 5.83 g (36 mmol) of iodine chloride in 60 mL of dry acetonitrile. after overnight stirring, the mixture was filtered, the filtrate was evaporated, and the residue was chromatographed on neutral alumina (act. III, 30 × 3 cm), using hexane as eluent. The yellow fraction afforded 1.03 g (33%) of the triiodide as a yellow, crystalline solid, mp 106–107 °C (EtOH/THF): ^{1}H NMR 0.05 (AB q, 2 H), 6.95 (tr, 1 H), 7.25 (d, 1 H), 7.54 (d, 1 H), 7.59 (d, 1 H), 7.93 (d, 1 H); IR 3000 w, 2920 w, 1890 w, 1620 w, 1545 w, 1455 sh, 1440 s, 1395 m, 1345 m, 1285 w, 1255 w, 1210 w, 1185 s, 1155 s, 1090 w, 1050 w, 995 m, 945 vs, 875 s, 775 s, 745 vs, 700 s, 630 m, 600 w, 470 m. Anal. Calcd for $C_{11}H_7I_3$: C, 25.4; H, 1.3; I, 73.3. Found: C, 25.51; H, 1.35; I, 73.04.

Elution of the same column with methylene chloride afforded 1.35 g (35%) of the tetraiodide **1a** as a yellow solid, mp 209–211 °C: ^{1}H NMR 0.705 (s, 2 H), 7.343 (s, 4 H); ^{13}C NMR 36.71, 94.36, 122.97, 142.299; IR 3000 vw, 2920 vw, 1600 br w, 1560 vw, 1490 vw, 1445 w, 1350 m, 1200 vw, 1155 s, 955 s, 815 m, 775 s, 720 w. Anal. Calcd for $C_{11}H_6I_3$: C, 20.4; H, .9; I, 78.6. Found: C, 20.52; H, .94; I, 78.38.

2,5,7,10-Tetraiodo-11,11-difluoro-1,6-methano[10]annulene and 2,5,7-Triiodo-11,11-difluoro-1,6-methano[10]annulene. Mercuric oxide (6.49 g, 30 mmol) was heated (reflux temperature) in 10 mL of trifluoroacetic acid until it dissolved. The reaction mixture was then heated to 180 °C to remove all volatiles and produce molten mercury trifluoroacetate. To the molten salt was added 11,11-difluoro-1,6-methano[10]annulene (1.3 g, 7.3 mmol) at 130 °C. A vigorous evolution of trifluoroacetic acid ensued with a concomitant color change of the reaction mixture to orange. After being heated to 180 °C for a few minutes to remove the last traces of acid, the mixture was cooled and evacuated to ensure complete removal of volatile components. The residue was dissolved in 500 mL of dry acetonitrile (orange solution) and treated with a solution of 5.67 g (35 mmol) of iodine chloride in 50 mL of dry acetonitrile. The reaction mixture was stirred overnight at room temperature and the solvent was removed under vacuum to yield an orange solid, which was flash chromatographed on 230–400-mesh silica gel (column 30 × 3 cm) with hexane as eluent. The first fraction, after removal of solvent (500 mg) and recrystallization from hexane (250 mg, 6%), gave fine yellow needles with a decomposition point >180 °C: ^{1}H NMR 6.98–8.40 (m); IR 3040 w, 1640 br w, 1500 m, 1350 m, 1320 sh, 1235 s, 1140 s, 1115 vs, 1030 w, 1000 w, 900 s, 870 w, 840 m, 825 m, 810 m, 770 w, 745 m, 670 w, 610 w; HRMS 554.8648 (M$^+$).

Scheme I[a]



2

1a

[a] i = Hg(OAc)$_2$/MeOH; ii = ICl/CH$_3$CN.

The next fractions from the above flash chromatography were always contaminated with triiodo products. To effect separation, the combined fractions were re-flash-chromatographed three times, followed by fractional crystallization from hexane to afford 70 mg (1.5%) of red-brown crystals, mp 206–208 °C: ^{1}H NMR 7.189 (s); IR 3030 vw, 1630 vw, 1560 w, 1465 w, 1410 w, 1350 w, 1240 vs, 1200 s, 1140 s, 1100 m, 1050 w, 1010 m, 810 m, 800 s, 790 m, 760 m, 660 vw, 615 w, 580 w; UV-vis λ_{max} (THF) 284 nm. Anal. Calcd for $C_{11}H_4F_2I_4$: C, 19.37; H, 0.59; F, 5.58; I, 74.47. Found: C, 19.24; H, 0.56; F, 5.65; I, 74.18.

X-ray Structure. Intensity data were collected on an Enraf-Nonius CAD-4 automatic diffractometer using graphite monochromated Mo Kα radiation. Unit cell constants were determined by least-squares refinement of the setting angles of 25 reflections, each collected at $\pm 2\theta$. A total of 2189 unique reflections were measured up to $2\theta = 60°$ using the $\omega-2\theta$ scan technique. Three reflections measured every 2 h showed no significant intensity variations.

Intensities were corrected for Lorentz and polarization effects and an empirical absorption correction was also applied.

The structure was solved by direct methods (MULTAN 11/82)[4] and Fourier techniques. Full-matrix least-squares refinement of the non-hydrogen atoms with anisotropic thermal factors in the final stages were carried out. Hydrogen atoms located from the difference Fourier map were included and refined with the arbitrary thermal factor $B = 4 Å^2$.

Crystal data for $C_{11}H_6I_4$: $M_r = 645.786$; orthorhombic, space group $Pna2_1$ (No. 33), $a = 20.697$ (4), $b = 14.704$ (3), and $c = 4.413$ (1) Å, $V = 1343.00$ Å^3, $Z = 4$, $D_{calc} = 3.194$ g cm^{-3}; μ(Mo Kα) = 91.547 cm^{-1}. Empirical absorption correction: minimum and maximum transmission 58.97 and 99.76%; respective minimum and maximum correction factors: 0.768 and 0.999. Of 2189 unique reflections, 1833 with $I > 2\sigma(I)$ were taken as observed. The refinement of 160 variables led to a final value of $R = 0.029$ and $R_w = 0.035$ (eq 1). Scattering factors are taken from Cromer and Waber.[5] All the calculations were carried out using the Structure Determination Package SDP.[6]

$$R \equiv \Sigma||F_o| - |F_c||/\Sigma|F_o|$$

$$R_w \equiv [\Sigma\omega(|F_o| - |F_c|)^2/\Sigma\omega|F_o|^2]^{1/2} \qquad (1)$$

$$w = 1/\sigma_F^2$$

Results and Discussion

Synthesis. Although 2,5,7,10-tetrabromo-1,6-methano[10]annulene (**1b**) was prepared before by the use of *N*-bromosuccinimide,[10] a similar reaction for aromatic iodination is unknown. Standard aromatic iodination procedures[7] produced mixtures of polyiodinated 1,6-methano[10]annulene, which were difficult to separate. Attempts to exchange the four bromine substituents of **1b**

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[3] Bornatsch, W.; Vogel, E. *Angew. Chem.* **1975**, *87*, 412; *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 420.

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[5] Cromer, D. T.; Waber, J. T. *International Tables for X-ray Crystallography*; Kynoch: Birmingham, 1974; Vol. IV, Table 2.2B.

[6] Frenz, B. A. and Associates, Inc. College Station, TX 77840, and Enraf-Nonius, Delft, Holland, 1983.

[7] See, for example: Allen, C. F. H.; Cressman, H. W. J.; Johnson, H. B. *Organic Synthesis*; Wiley: New York, 1955; Collect. Vol. 3, p 796.

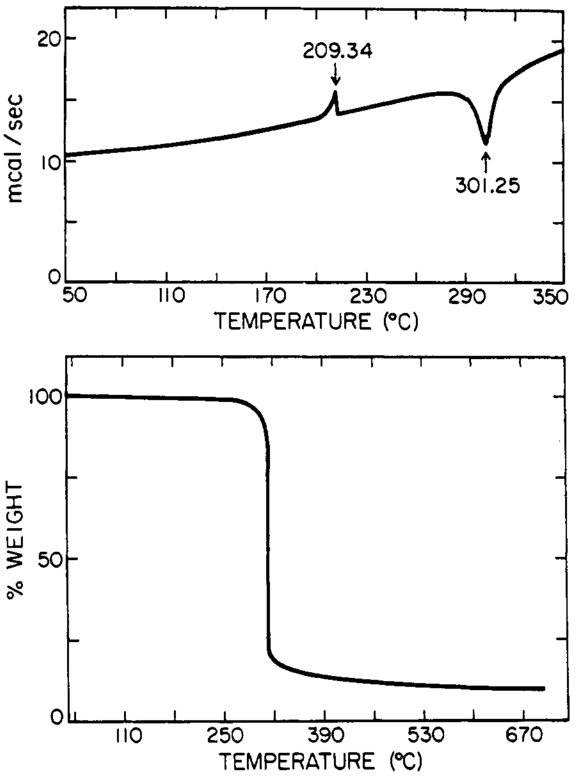


Figure 1. (a) Differential scanning calorimetry of a 5.7-mg sample of **1a**; scan rate, 10 deg/min. (b) Thermal gravimetric analysis of a 15.7982-mg sample of **1a**; scan rate, 20 deg/min. The change in mass from 274 to 318 °C is 78.97%.

for iodine atoms using boron triiodide also failed. The preparation of the desired iodide was eventually accomplished by the two electrophilic substitution steps outlined in Scheme I.

The first step proceeds in 60–70%. Attempted mercuration of the hydrocarbon with mercuric chloride in methanol produced only partially mercurated product as well as mercury metal even after 4 days of reflux. On the other hand, the mercuration of the hydrocarbon with mercuric acetate in methanol is also slow (four days at ambient temperature) but useful to achieve the reported yields, without mercury metal contamination. The mercurial begins to precipitate out of the reaction mixture within 30 min and is an off-white, completely insoluble material in all common organic solvents. Attempts to effect the mercury displacement with iodine alone under various conditions failed, but electrophilic substitution of the tetramercurial with bromine produced **1b** in 22% yield. This approach, with the minor variation of substituting mercury trifluoroacetate in Scheme I, is also applicable to the iodination of 11,11-difluoro-1,6-methano[10]annulene, producing the desired tetraiodide in the paltry yield of 1.5%. No attempts were made to maximize yields, particularly in the latter case. The main side products in the iodination of the difluoro derivative were the 2,5,7-triiodo derivative (6%) and a pentaiodo derivative (~1%).

Properties. The tetramercurial derived from compound **2** is thermally labile; decomposition in a capillary tube is already noticeable between 220 and 230 °C by the appearance of a small amount of a yellow distillate concomitant with brown discoloration of the solid. The yellow distillate was 1,6-methano[10]annulene (**2**) as shown by TLC. The source of the hydrogen atoms in the decomposition product is unknown.

In Figure 1 we show the differential scanning calorimetry/thermal gravimetric analysis (DSC/TGA) of **1a**. The endothermic transition at 209.34 °C corresponds to the melting point, also observed in bulk (209–211 °C, capillary) and the exothermic transition centered at 301.25 °C coincides with a catastrophic loss of mass observed in the TGA. There is a 79–86% mass loss; the theoretical value for the loss of four iodine atoms is 78.64%. The difference between the calculated and observed values is probably due to the above-mentioned loss of a small amount of **2**.

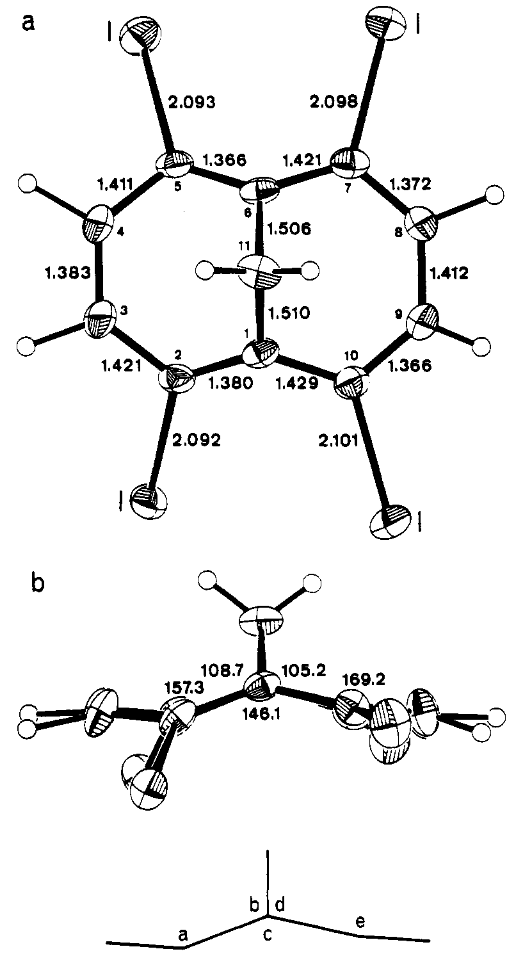
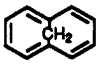
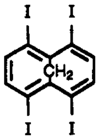
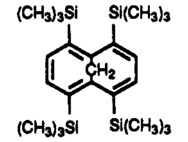


Figure 2. Molecular structure of **1a**, showing 40% probability thermal ellipsoids for the non-hydrogen atoms: (a) top view, bond distances in Å, esd range 0.007–0.011 Å (cf. Table I); (b) side view, dihedral angles in deg.

Structure. The solution high-field ^{1}H NMR spectrum revealed that compound **1a** is considerably distorted in comparison to the parent bicycle as judged from the position of the methano bridge hydrogens (+0.705 ppm, d_8-THF vs −0.51 ppm for the parent) but not as distorted as 2,5,7,10-tetrakis(trimethylsilyl)-1,6-methano[10]annulene (**1c**) (+1.53 ppm, acetone-d_6).[2]

This was borne out by an X-ray structure determination. In Figure 2 we show top and side views of the molecular structure of **1a** and in Table I are collected bond distances and angles for compounds **1a**, **1c**, and **2** with an emphasis on the C_1–C_6 distances (transannular distance) and the C_1–C_{11}–C_6 angles for compounds **1a**, **1c**, and **2**. As can be seen from the table, the effect of iodine substitution is to increase bond alternation in **1a** relative to **2** but the bond alternation is not as dramatic as in **1c**. The net result from this comparison appears to be that the halogens open up (flatten) the structure of the bicycle as inferred from the following: (1) the transannular distance and angle are the largest for **1a** among the three and (2) the dihedral angle "c" (cf. drawing for Table I) is the largest for **1a** (146.1°), compared to 139° for **2** and 139.7° for **1c**.

Table I. Structural Parameters

	compound		
	 **2**	 **1a**	 **1c**
	Bond Distances (Å)		
C_1–C_2	1.403 (3)	1.380 (10)	
C_2–C_3	1.379 (4)	1.421 (10)	
C_3–C_4	1.417 (4)	1.383 (10)	1.35
C_4–C_5	1.378 (3)	1.411 (9)	1.45
C_5–C_6	1.402 (3)	1.366 (10)	1.36
C_6–C_7	1.410 (3)	1.421 (10)	1.47
C_7–C_8	1.376 (4)	1.372 (10)	1.35
C_8–C_9	1.419 (4)	1.412 (10)	1.47
C_9–C_{10}	1.373 (3)	1.366 (10)	
C_{10}–C_1	1.405 (3)	1.429 (10)	
C_1–C_{11}	1.487 (3)	1.510 (11)	
C_{11}–C_6	1.484 (3)	1.506 (11)	1.50
C_1–C_6	2.235	2.357	2.298
	Bond Angles (deg)		
C_1–C_2–C_3	122.8	122.1 (6)	119
C_2–C_3–C_4	127.3	127.1 (6)	132
C_3–C_4–C_5	126.8	128.5 (7)	
C_4–C_5–C_6	122.7	121.8 (7)	
C_5–C_6–C_7	126.2	133.0 (7)	
C_6–C_7–C_8	122.6	121.5 (6)	
C_7–C_8–C_9	127.6	130.3 (7)	
C_8–C_9–C_{10}	127.1	130.9 (7)	126
C_9–C_{10}–C_1	123.1	121.4 (7)	118
C_{10}–C_1–C_2	126.8	132.9 (7)	128
C_1–C_{11}–C_6	97.6	102.8	100

As a consequence of the above, C_1–C_{11} and C_6–C_{11} bond lengths are lengthened from 1.487 and 1.484 for **2** to 1.510 and 1.506, respectively. Surprisingly, as seen from the side view, the torsional distortion of the bicycle is minimal (all the four iodine atoms are on one side of the C=C double bond plane) compared to the effect of "*peri*" trimethylsilyl substitution[2] or the effect of *peri* bromine substitution in 1,4,5,8-tetrabromonaphthalene.[8]

Summary

The title compound could not be prepared by traditional aromatic iodination procedures. A new approach employing an intermediate mercuration step was successful, provided mercuric acetate or trifluoroacetate are employed. The organomercurial was not characterized due to intractability but subsequent conversion to the known 2,5,7,10-tetrabromo-1,6-methano[10]annulene constitutes a proof of structure.

The tetraiodo bicycle **1a** is surprisingly only slightly distorted in an unusual fashion; the transannular distance is enlarged as are the bridge bonds and the angle subtended by the bridge carbon atoms. The π-delocalization is also somewhat decreased relative to the parent bicycle.

As expected, the carbon–iodine bonds are thermally labile; heating the solid produces a small amount of unsubstituted methano[10]annulene and a black solid.[9]

Acknowledgment. F.W. and K.D.S. thank the Office of Naval Research for support and the donors of the Petroleum Research Fund, administered by the American Chemical Society. We also thank Hugh Webb for high resolution mass spectra and Ata Shirazi for high resolution NMR spectra.

Supplementary Material Available: Tables of final atomic coordinates and anisotropic temperature factors for **1a** (5 pages); observed and calculated structure factors for **1a** (9 pages). Ordering information is given on any current masthead page.

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(9) Sturm, K. D. Unpublished Results. Results of characterization of this solid will be published elsewhere.

(10) Vogel, E. In *Aromaticity*; Ollis, W. D., Eds.; The Chemical Society Special Publications: London, 1967; No. 21, p 113.

Synthesis of Furans by Ag(I)-Promoted Cyclization of Allenyl Ketones and Aldehydes

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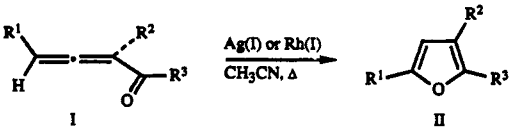
A series of conjugated allenones (**11b**, **12b**, **13c**; **18a**, **18b**; **19b**, **20b**, AB and **27d**) were prepared by [2,3] Wittig rearrangement of (propargyloxy)acetic acids or [(propargyloxy)methyl]stannanes followed by oxidation of the resultant allenylcarbinols. These allenones were readily cyclized to 2,3,5-trisubstituted furans upon treatment with $AgNO_3$–$CaCO_3$ in aqueous acetone. Under these conditions 2-(hydroxymethyl)-3,5-dialkylfurans self-condensed to give 2,2′-difurylmethanes

We recently described a new route to furans involving Ag(I)- or Rh(I)-catalyzed cyclization of allenyl ketones and aldehydes (eq 1).[1,2] The present study was undertaken to examine the applicability of that approach to furans with functionalized C2 substituents. Such systems are of interest as prototypes of intermediates for the synthesis of various natural products.[2-4]

 (1)

R^1 = H, CH_3; R^2 = H, n-C_7H_{15}; R^3 = H, CH_3, $Me_2C{=}CHCH_2CH_2C(Me){=}CH$

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