oxygenated substrate **1j** (entry 7) led to diquinane **3j** in good yield despite the hydrogen at position 5.

Substitution at position 3 was also significant. Those cases in which $R^3 \neq H$ (entries 1 and 7) showed complete diastereoselectivity for α-disposition of the side chain, corresponding to a compact transition state with the olefin tucked underneath the zwitterion to avoid unfavorable steric interactions with R^3. Those cases with $R^3 = H$ (entries 3, 5, and 6) gave roughly equal amounts of products derived from compact and extended transition states. Cyclization products with β-disposed tertiary carbocations are geometrically precluded from mixed ketal formation and suffer either elimination or solvent capture.[11]

In summary, we have found that photochemically generated oxyallyl zwitterions bearing pendant olefinic groups undergo efficient cyclization to yield fused bicyclic products. Complete selectivity for either 5-exo closure to give diquinanes or 6-endo closure to give hydrindans is possible by varying alkene substitution, and high diastereoselectivity is also possible. The substantial complexity of the products relative to the 4-pyrone precursors renders this a powerful method for the rapid construction of functionalized carbocyclic skeletons. Further applications of this new class of reactions will be reported in due course.

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Supplementary Material Available: Representative experimental procedures for the preparation and photolysis of **1a–d**, and an ORTEP drawing and tables of positional parameters, bond angles, and bond distances for **3a** (11 pages). Ordering information is given on any current masthead page.

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(11) In several cases, we have observed slow consumption of **6** and **7** due to secondary photochemistry. The somewhat higher overall yields seen in entries 1 and 7 may be attributed to their clean formation of mixed ketal **2**, which is not photolabile.

Synthesis of Mixed Copper–Indium Chalcogenolates. Single-Source Precursors for the Photovoltaic Materials $CuInQ_2$ (Q = S, Se)

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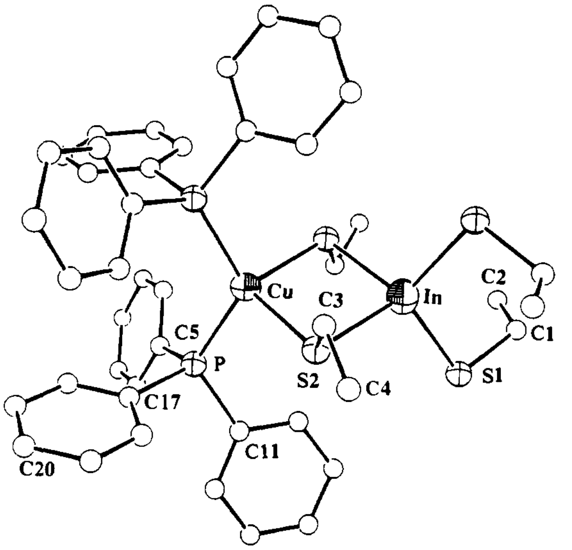

Figure 1. ORTEP drawing of $(Ph_3P)_2CuIn(SEt)_4$. The molecule is sitting on a 2-fold crystallographic axis. Selected bond distances (Å) and angles (deg): Cu–P1 2.278(2), Cu–μ–S2 2.418(2), S2–C3 1.816(7), In–S1 2.419(3); P–Cu–P 120.7(1), S2–Cu–S2 96.3(1), Cu–S2–In 85.58(8), S1–In–S1 112.7(1). The structure of $(Ph_3P)_2CuIn(SeEt)_4$ is essentially the same, the difference being in the conformation of the ethyl groups and the lack of a 2-fold rotation axis. Selected average bond distances (Å) and angles (deg): Cu–P 2.275(3), Cu–^{br}Se 2.526(2), In–^{br}Se 2.611(2); In–^{t}Se 2.557(2); P1–Cu–P2 114.9(1), ^{br}Se–Cu–^{br}Se 101.78(7), Cu–^{br}Se–In 80.42(6), ^{t}Se–In–^{t}Se 117.48(6). ^{br}Se and ^{t}Se denote bridging and terminal Se atoms.

Currently, *thin film* solar cells of $CuInSe_2$ are the most radiation tolerant and among the most efficient solar cells at 12%.[1] $CuInS_2$ is also a promising photovoltaic thin film material with achieved solar cell efficiency of 7%.[2] $CuInSe_2$ has been prepared by a number of physical techniques:[3] (1) flash evaporation and evaporation of $CuInSe_2$ powder; this leads to copper deficient material unless the source temperature is approximately above 1300 °C; often adding an additional Se evaporation source to help control reevaporation of Se from the substrate is necessary; (2) evaporation from the binary compounds, for example, Cu_2Se, In_2Se_3; (3) three-source coevaporation from the elements; (4) RF and ion beam sputtering of polycrystalline $CuInSe_2$; (5) molecular beam epitaxy; and (6) spray pyrolysis.[3] All these techniques involve high temperatures. Electrochemical deposition from solutions has also been employed, but stoichiometric control of the product is difficult due to the different rates of Cu, In, and Se deposition.[3] The films obtained can have high defect density and nonstoichiometry, both detrimental to device performance. It is clear that a reliable, low-temperature method for producing high-quality $CuInSe_2$ is highly desirable. By controlling the synthesis of $CuInSe_2$ at the molecular level via proper precursor compounds, we should be able to achieve milder synthetic conditions for $CuInSe_2$. Ideally, such precursors should have the correct Cu/In stoichiometry built in (i.e., Cu:In ratio 1:1) and should decompose in simple steps to the desired product. Although many excellent single-source precursors exist for binary solid-state compounds, such as GaAs,[4] CdSe,[5a] ZnSe,[5b] MnTe,[5c] TiB_2,[6] In_2Se_3,[7] GaN,[8] FeB,[9] etc.,[10] reports on single-source precursors

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for ternary or multinary compounds are rare.[11] This may be partly due to the difficulty in obtaining heterometallic precursor compounds with the proper elements in the proper ratio. Here we report the synthesis, molecular structures, and properties of the first structurally characterized molecular precursor compounds $(Ph_3P)_2CuIn(QR)_4$ (Q = S, Se; R = ethyl, isobutyl) which yield $CuInQ_2$ under mild conditions. The only other structurally characterized Cu/In heterometallic cluster is $[Cu_6In_3(SEt)_{16}]^-$.[12]

$(Ph_3P)_2CuIn(QR)_4$ were prepared by the reaction of $[(Ph_3P)_2Cu(CH_3CN)_2]PF_6$[13] with $In(QR)_4^-$ (prepared in situ) according to eqs 1 and 2.[14]

$$InCl_3 + 4KQR \rightarrow K^+ + [In(QR)_4]^- + 3KCl \qquad (1)$$

$$[(Ph_3P)_2Cu(CH_3CN)_2]^+ + [In(QR)_4]^- \rightarrow (Ph_3P)_2CuIn(QR)_4 + 2CH_3CN \qquad (2)$$

$$Q = S, Se; R = Et, {}^iBu$$

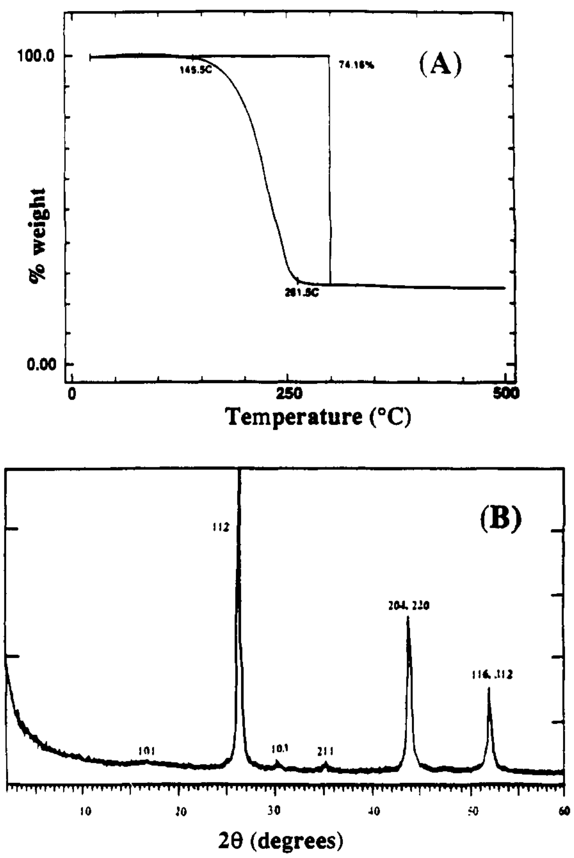

Figure 2. (A) TGA diagram of $(Ph_3P)_2CuIn(SEt)_4$ under nitrogen flow; heating rate, 5 deg/min. (B) X-ray powder diffraction pattern (XRD) of the pyrolysis product $CuInSe_2$. $CuInS_2$ gives a similar diffraction pattern.

The molecular structures[15] of $(Ph_3P)_2CuIn(QEt)_4$ (**1**, Q = S; **2**, Q = Se) and $(Ph_3P)_2CuIn(S^iBu)_4$ (**3**) are essentially the same and feature tetrahedrally coordinated In and Cu atoms bridged by thio(seleno)lates forming a planar "$CuIn(QR)_2$" core. The terminal ligands are two phosphines on the Cu and two thiolates or selenolates on the In. The structure of **1** is shown in Figure 1. Selected bond distances for **1** are given in Figure 1. The In–S bond distances in **1** (2.49 Å) for the bridging thiolates are longer than the corresponding distances for the terminal thiolates (2.42 Å), in agreement with earlier observations.[16] The Cu–S bond distances (2.42 Å) are in the range observed for other tetrahedral Cu thiolates.[17] With the S(2)–In–S(2) bond angle of 92° and terminal S(1)–In–S(1) angle of 112°, the indium center shows a distorted tetrahedral coordination. Deviation from the ideal tetrahedral angle is even more pronounced in the copper center due mainly to the bulky phosphines (the P–Cu–P bond angle is 121° while S(2)–Cu–S(2) is 96°). The Cu–In distances in **1**, **2**, and **3** are 3.34, 3.32, and 3.24 Å, respectively.

The potential of $(Ph_3P)_2CuIn(QR)_4$ to give $CuInQ_2$ conveniently was probed by thermogravimetric analysis (TGA). All compounds show simple weight-loss decomposition curves at relatively low temperatures and show decomposition patterns corresponding to stoichiometric formation of $CuInQ_2$ with loss of Ph_3P and EtQEt as identified by mass spectrometry. The TGA curves for **1**, **2**, and **3** level off above ca. 260, 396, and 330 °C, respectively. These temperatures are among the lowest reported for $CuInQ_2$. A typical TGA curve is shown for $(Ph_3P)_2CuIn(SEt)_4$ in Figure 2A. The $CuInQ_2$ products were identified by X-ray diffraction to be single phased, as shown in Figure 2B. Thermal decomposition of the compounds may be represented by eq 3.

$$(Ph_3P)_2CuIn(QEt)_4 \rightarrow 2Ph_3P + 2EtQEt + CuInQ_2 \qquad (3)$$

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(10) For other examples, see: (a) *Chemical Perspectives in Microelectronic Materials II*; Interrante, L. V., Jensen, K. F., Dubois, L. H., Gross, M. E., Eds.; *Mater. Res. Soc. Symp. Proc.* **1991**, *204*, papers therein. (b) *Chemical Perspectives in Microelectronic Materials*; Gross, M. E., Jasinski, J. M., Yates, J. T., Eds.; *Mater. Res. Soc. Symp. Proc.* **1989**, *131*, papers therein. (c) *Better Ceramics Through Chemistry III* and references therein; Brinker, C. J., Clark, D. W., Ulrich, D. R., Eds. *Mater. Res. Soc. Symp. Proc.* **1988**, *121*, papers therein.

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(14) (a) NaSEt (0.38 g, 4.52 mmol) was dissolved in 50 mL of CH_3OH, and $InCl_3$ (0.25 g, 1.13 mmol) was added. After the mixture was stirred for a half-hour, $(Ph_3P)_2Cu(CH_3CN)_2PF_6$ (0.92 g, 1.13 mmol) in 20 mL of CH_3CN was added dropwise and the mixture stirred for additional 2 h. Following filtration, the solvent was removed under low pressure from the solution and the residue extracted with ca. 100 mL of CH_2Cl_2 and filtered. The solution was concentrated to ca. 20 mL and layered with 30 mL of Et_2O. Colorless crystals were obtained after cooling at 0 °C for 3 days: yield 77%; ^1H NMR (300 MHz, $CDCl_3$) δ 7.29 (br m, 30 H, C_6H_5), 2.67 (br m, 8 H, CH_2), 1.19 (br m, 12H, CH_3). Compound **3** prepared in a similar way gave a yield of 75%: ^1H NMR (300 MHz, $CDCl_3$) δ 7.36–7.24 (br m, 30H, C_6H_5), 2.46 (pd, 8 H, CH_2), 1.55 (br m, 4 H, CH), 0.85 (pd, 24 H, CH_3). The Se analogue $(Ph_3P)_2CuIn(SeEt)_4$ (**2**) was run in CH_3CN and gave a yield of 50%:[14b] ^1H NMR (300 MHz, $CDCl_3$) δ 7.4–7.2 (m, 30H, C_6H_5); 2.62 (br m, 8H, CH_2); 1.29 (br m, 12 H, CH_3). All the compounds give satisfactory elemental analyses. (b) NaSeEt was prepared by the reaction of Et_2Se_2 with sodium in refluxing toluene. The solid thus obtained was filtered, washed with Et_2O, and vacuum dried.

(15) Suitable crystals of **1** and **3** for single-crystal X-ray analysis were grown by cooling the concentrated CH_3OH/CH_3CN solution at 0 °C. Crystals of **2** were grown from a concentrated CH_3CN solution at 23 °C. Crystal data for **1**: $a = 18.260(4)$ Å, $b = 12.386(10)$ Å, $c = 21.162(4)$ Å, $\beta = 109.40(2)°$, $V = 4515(4)$ Å^3, space group $C2/c$, $d_{calcd} = 1.394(1)$ g/cm^3. Single-crystal X-ray diffraction data were collected on a Rigaku AFC6 (Mo Kα radiation), with $\theta/2\theta$ scan, $3° < 2\theta < 40°$, and 1796 reflections with $F_o^2 > 3\sigma(F_o^2)$. The number of variables was 236, and the final R/R_w = 0.037/0.043. Crystal data for **2**: $a = 12.069(4)$ Å, $b = 13.328(7)$ Å, $c = 15.242(7)$ Å, $\alpha = 88.06(4)°$, $\beta = 67.92(3)°$, $\gamma = 83.34(4)°$, $V = 2256(4)$ Å^3, space group $P\bar{1}$, $d_{calcd} = 1.67$ g/cm^3. Single-crystal X-ray diffraction data were collected on a Nicolet P3 four-circle diffractometer (Mo Kα radiation), with $\theta/2\theta$ scan, $3° < 2\theta < 50°$, and 3833 reflections with $F_o^2 > 3\sigma(F_o^2)$. The number of variables was 479, and the final R/R_w = 0.038/0.031. Crystal data for **3**: $a = 13.271(18)$ Å, $b = 13.888(16)$ Å, $c = 15.440(17)$ Å, $\alpha = 69.67(8)°$, $\beta = 75.26(9)°$, $\gamma = 87.73(10)°$, $V = 2577(5)$ Å^3, space group $P\bar{1}$, $d_{calcd} = 1.365$ g/cm^3. Single-crystal X-ray diffraction data were collected (at −110 °C) on a Nicolet P3 four-circle diffractometer (Mo Kα radiation), with $\theta/2\theta$ scan, $3° < 2\theta < 40°$, and 1721 reflections with $F_o^2 > 3\sigma(F_o^2)$. The crystals of this compound were weak diffractors. The number of variables was 185, and the final R = 0.12. Due to the poor quality of the data set further refinement of the structure of **3** was not possible. Hence most of the bond distance and angle comparisons with other systems are made with reference to structure **1**. The structures were solved with SHELXS-86 and were refined with the TEXSAN package[15c] of crystallographic programs. An empirical absorption correction based on ψ scans and a DIFABS[15b] correction was applied to all data. (b) DIFABS: Walker, N.; Stuart, D. An Empirical Method for Correcting Diffractometer Data for Absorption Effects. *Acta Crystallogr.* **1983**, *A39*, 158–166. (c) TEXSAN: Single Crystal Structure Analysis Software, Version 5.0. Molecular Structure Corporation, The Woodlands, TX.

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Bulk pyrolysis of $(Ph_3P)_2CuIn(SeEt)_4$ in a Pyrex tube at 400–450 °C and 0.01 mmHg of pressure gives shiny films of single-phase $CuInSe_2$ as determined by the X-ray powder diffraction (XRD) pattern and confirmed by elemental analysis. Elemental analysis of the bulk $CuInSe_2$ shows less than 0.6% of carbon content. Proton NMR and mass spectra of the volatile products collected in a liquid nitrogen trap during the pyrolysis experiment show only Ph_3P and EtSeEt. Scanning electron micrographs (SEM) of $CuInSe_2$ show a smooth continuous film morphology.

The $(Ph_3P)_2CuIn(QR)_4$ compounds are the first structurally characterized examples of single-source precursors to the ternary semiconductors $CuInQ_2$. Their solubility in common organic solvents make them good candidates for solution spray pyrolysis for thin film deposition at relatively low temperatures.[18]

Acknowledgment. Financial support from the National Science Foundation for a Presidential Young Investigator Award, CHE-8958451, and from NASA is gratefully acknowledged. The X-ray instrumentation used in this work was purchased in part with funds from NSF Grant CHE-8908088. We also thank the Center of Electron Optics at Michigan State University for use of the electron microscopy facilities. We thank Dr. Aloysius Hepp of NASA Lewis Research Center, Cleveland, OH, for fruitful discussions.

Supplementary Material Available: An ORTEP drawing of compounds **2** and **3** and tables of atomic coordinates of all atoms and anisotropic and isotropic thermal parameters of all non-hydrogen atoms for **1–3** (36 pages); listing of calculated and observed $(10F_o/10F_c)$ structure factors for **1–3** (54 pages). Ordering information is given on any current masthead page.

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(18) Hirpo, W.; Sutorik, A. C.; Hepp, A.; Kanatzidis, M. G. Work in progress.

Selectively ^{13}C-Enriched DNA: ^{13}C and ^{1}H Assignments of the Lac Operator by Two-Dimensional Relayed HMQC Experiments

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NMR spectroscopy can provide useful conformational information on drug–DNA or protein–DNA complexes in solution.[1,2] Unfortunately the overlap of proton resonances between the two partners often precludes a complete assignment. One way of circumventing this difficulty is to use complexation of nucleic acids with ^{15}N uniformly labeled proteins.[3,4] Another is to use ^{13}C-labeled nucleic acids. Nikonowicz and Pardi recently reported[5,6] the first multidimensional heteronuclear NMR studies on uniformly ^{13}C-labeled RNA duplexes prepared by enzymatic procedures. Introduction of a ^{13}C-label in selected positions of an oligo(deoxy)nucleotide, potentially available by chemical synthesis, is another solution. Within this context, labeled sugar moieties at C1′ or C2′ appeared to be the best choice since the proximity of their attached proton with the base protons allows for a complete assignment of the oligonucleotide's proton resonances.

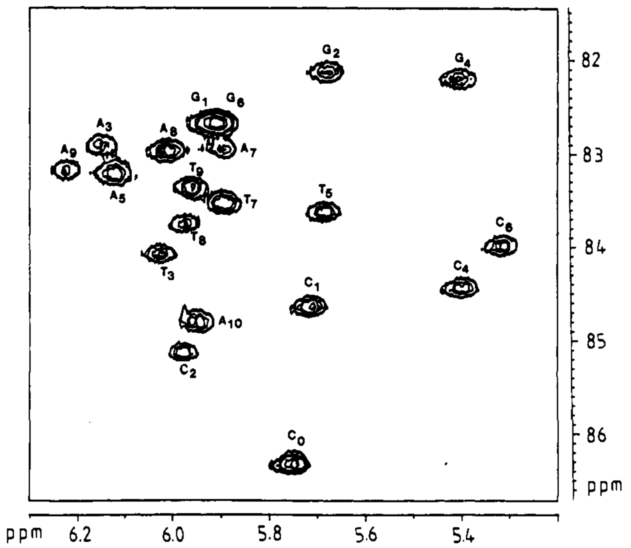

Figure 1.

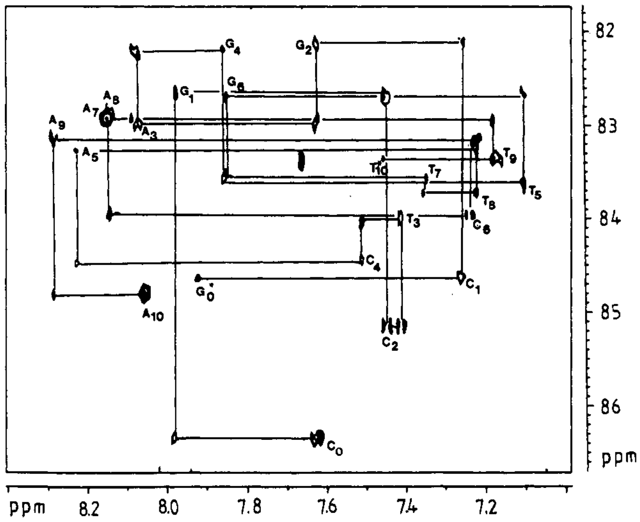

Figure 2.

This paper describes the ^{13}C and ^{1}H resonance assignments of half of the selectively ^{13}C-labeled lac operator at the C1′ carbon. This method, requiring relayed HMQC-TOSCY and HMQC-NOESY spectra, is promising for the study of large oligonucleotides or complexes with DNA.

The required 1′-^{13}C-labeled oligodeoxynucleotide 5′d-(CGCTCACAATT*) and its complementary sequence were prepared on a Pharmacia automatic synthesizer via phosphoramidite chemistry[7] using the classical unlabeled deoxynucleoside

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