In summary, utilizing the organometallic reagent 4, I have been able to obtain BPDSe-TTF in an overall yield of 17%. This general procedure can also be extended to the other members of the BADSe-TTF donor series. In addition, this procedure permits the isolation of asymmetric sulfur/selenium donors not accessible by other methods. The electrochemistry of 1 indicates only reversible oxidation to the radical cation and not to the dication in this solvent system.

Experimental Section

Melting points were determined on a Mettler FP80/82 hotstage and are uncorrected. Ultraviolet-visible spectra were obtained on a Varian Cary 2300 spectrophotometer in either CH_2Cl_2 or dichlorobenzene. Infrared spectra were obtained with a Beckman IR 4260 spectrophotometer. ^1H NMR spectra were taken in $CDCL_3$ or $CDCl_3/CS_2$ (1:1) on a Chemmagnetics Inc. spectrometer with tetramethylsilane as an internal standard. Elemental analyses were carried out by Galbraith Laboratories, Inc., Knoxville, TN.

Electrochemistry Equipment. For cyclic voltammetry, a Princeton Applied Research Model 173 potentiostat Model 175 universal programmer were used in the standard three-electrode configuration (a platinum bead working electrode, a platinum mesh counter electrode, and Ag/0.1 M $AgNO_3$ in acetonitrile reference electrode). The electrolyte was 0.1 M tetrabutylammonium hexafluoroantimonate ($TBAAsF_6$, recrystallized from water/methanol) in benzonitrile (BN), which had been freshly distilled from CaH_2 under argon.

4,5-(Propylene-1,3-diseleno)-1,3-dithiole-2-thione (3). Lithium bromide (1.8 g, 0.017 mol) was added to a warmed mixture of $(TBA)_2[Ni(dsit)_2]$[8] (4) (4.0 g, 0.0036 mol) in acetonitrile (200 mL) in one portion. The solution was refluxed for 1 h. Propylene bromide (31.6 g, 0.017 mol) was added to the refluxing solution, and the resulting mixture was refluxed overnight (20 h). The warm solution was filtered, and the residue was washed with methylene chloride until nearly colorless. This filtrate was concentrated on a rotary evaporator to an oily residue. To this oil was added methanol (50 mL), and the precipitated olive-green solid was collected. After the solid was vacuum-dried, crude 3 (1.4 g, 52%) was obtained. Recrystallization from methanol gave analytically pure 3 (1.3 g, 48%) as yellow-brown needles: mp 126-127 °C; UV λ_{max} (CH_2Cl_2) 383 nm (ϵ 15 200); IR (KBr) 2910 (w), 1620 (w), 1490 (m), 1460 (m), 1410 (m), 1380 (m), 1330 (m), 1240 (m), 1210 (m), 1145 (m), 1050 (s), 1020 (m), 878 (m), 845 (m), 825 (m), 770 (m), 725 cm^{-1}; NMR (200 MHz, $CDCl_3$) 2.92 (m, 4 H, SCH_2), 2.78 ppm (m, 2 H, CH_2). Anal. Calcd for $C_6H_6S_3Se_2$: C, 21.69; H, 1.82; S, 28.95; Se, 47.54. Found: C, 21.91; H, 1.98; S, 29.10; Se, 47.43.

4,5-(Propylene-1,3-diseleno)-1,3-dithiol-2-one (2). In a refluxing solvent mixture of chloroform (280 mL), glacial acetic acid (130 mL), and water (10 mL) was dissolved 3 (1.25 g, 0.0038 mol). To the refluxing solution was added mercuric acetate (1.25 g, 0.0039 mol) in one portion, and the suspension was stirred at reflux for 26 h. This mixture was filtered while still warm through a fine fritted funnel, and the filtrate was concentrated to dryness. Crude 2 (1.17 g, 98%) was obtained as a brown solid. Recrystallization from acetonitrile followed by further purification with gradient sublimation[13] gave 2 (0.88 g, 75%) as bright yellow crystals: mp 102-103 °C; UV λ_{max} (CH_2Cl_2) 381 nm (ϵ 4630); IR (KBr) 2920 (w), 2870 (w), 1750 (m), 1670 (m), 1635 (s, br), 1570 (s), 1485 (m), 1420 (m), 1395 (m), 1245 (s), 1230 (s), 1160 (m), 1065 (m), 980 (m), 960 (m), 875 (s), 850 (s) 835 (m), 780 (m), 710 (m) cm^{-1}; NMR (200 MHz, $CDCl_3$) 2.88 (m, 4 H, SCH_2), 2.75 ppm (m, 2 H, CH_2). Anal. Calcd for $C_6H_6OS_2Se_2$: C, 22.79; H, 1.91; O, 5.06; S, 20.28; Se, 49.95. Found: C, 22.78; H, 1.90; S, 20.18; Se, 49.89.

4,5:4',5'-Bis(propylene-1,3-diseleno)tetrathiafulvalene (1). A mixture of 2 (450 mg, 1.35 mmol) and triethyl phosphite (10 mL) was brought to reflux under an argon atmosphere. After 35 min of refluxing, methanol (5 mL) was added, and the solution was then placed in an ice bath. The yellow solid was collected by filtration, washed with methanol followed by ether, and then vacuum-dried to give 1 (223 mg, 55%) as a yellow powder. Multiple recrystallization from dichlorobenzene yielded 1 (186 mg, 46%) as amber needles: mp 272-277 °C; UV λ_{max} (dichlorobenzene) 340 (ϵ 16 000), 400 nm (ϵ 6000); IR (KBr) 2960 (w), 2920 (w), 2860 (w), 1495 (m), 1455 (w), 1410 (m br), 1365 (we, 1245 (s), 1220 (s), 1160 (m), 1025 (w), 969 (m), 875 (m), 840 (m), 830 (m), 765 (s) cm^{-1}; NMR (200 MHz, $CDCl_3/CS_2$(1:1 vol)) 2.82 (m, 8 H, SCH_2), 2.74 ppm (m, 4 H, CH_2). Anal. Calcd for $C_{12}H_{12}S_4Se_4$: C, 24.01; H, 2.17; S, 21.37; Se, 52.61. Found: C, 24.25; H, 2.16; S, 21.12; Se, 52.37.

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Registry No. 1, 111351-57-6; 2, 111351-58-7; 3, 111351-59-8; 4, 107207-66-9; $Br(CH_2)_3Br$, 109-64-8.

Pauson-Khand Cycloaddition of Norbornenones and Norbornenols: Electronic Effect on Regioselectivity

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The Pauson-Khand cyclopentenone synthesis is gaining increased attention due in large measure to the high degree of stereo- and regiochemical control exhibited in several of its versions.[1] Many examples of such selectivity in this reaction have proved amenable to explanation on the basis of steric interactions during insertion of the alkene component into a formal carbon-cobalt bond of the initially formed alkyne-$Co_2(CO)_6$ complex. Thus intermolecular cyclizations of alkynes with bicyclic alkenes commonly produce exo-fused products with the larger alkyne substitutent at the 2-position of the cyclopentenone, a result of insertion of the less hindered face of the alkene π-bond into a less substituted C-Co bond in the complex. Conversely, large allylic substituents on the alkene tend to wind up "anti" to the new ketone (i.e., adjacent to C-4 of the new ring rather than to C-5), thereby avoiding a 1,3-pseudodiaxial interaction between the large substitutent and a $Co(CO)_3$ moiety.[2] Stereocontrol in intramolecular cyclizations to bicyclo[3.3.0]oct-1-en-3-one derivatives likewise may be predicted on the basis of minimizing similar 1,3-interactions that arise during the insertion step between substituents on the metal-complexed enyne precursors.[3]

In contrast to these situations, selectivity in reactions involving terminal alkenes seems to be less predictable, and the entire question of electronic effects on this process remains open. The latter is clouded by two effects. First, alkenes with strongly electron-withdrawing substituents do not give cyclopentenones with alkynes and $Co_2(CO)_8$; CO insertion does not take place, and, instead, 1,3-dienes are formed.[4] Second, although alkenes with donor groups

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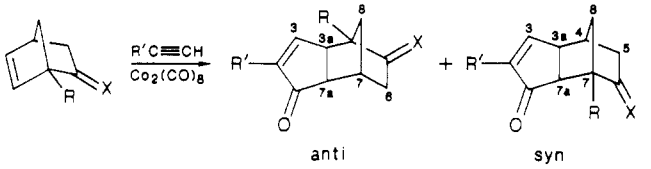
Table I. Products of Pauson-Khand Cycloadditions of Norbornenones and Norbornenols



reactn	X	R	R'	product ratio	yield, %
1	=O	H	Ph	27:73 (1:2)	58
2	=O	H	Me	24:76 (3:4)	41
3	H_{exo},OH_{endo}	H	Me	55:45 (5:6)	38
4	=O	Me	Me	30:70 (7:8)	55
5	H_{exo},OH_{endo}	Me	Me	59:41 (9:10)	59

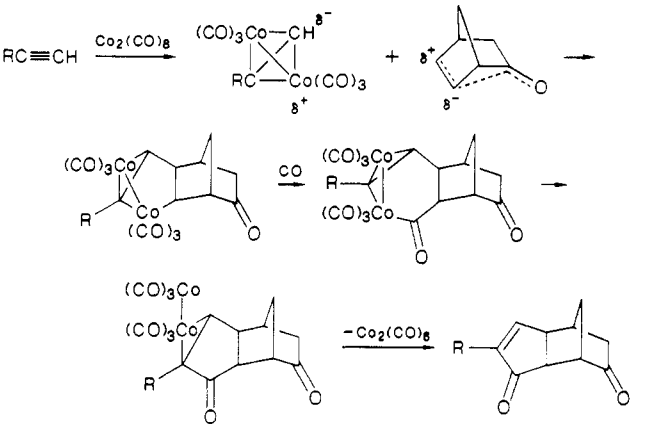
such as vinyl esters and ethers do give cyclopentenones,[5] the product ratios are almost certainly not the kinetic ones due to differential decomposition of the products under the reaction conditions.[6] We recently had occasion to compare several Pauson–Khand cycloaddition reactions of norbornenone, 1-methylnorbornenone, and the corresponding norbornenols. The results, presented herein, reveal a likely electronic effect on the regioselectivity in a situation where steric effects are systematically controlled.

In all, five systems were studied. Product ratios and isolated yields are presented in Table I. It is of some note that reactions 1, 2, and 4 work at all; electron withdrawal by the carbonyl group from the double bond is obviously moderated considerably by the unusual structural nature of the norbornenone system. Structures of the diketonic products of reactions 1, 2, and 4 were established first by carrying out X-ray structure determinations on the two highly crystalline products of reaction 1 (**1** and **2**) and then comparing ^1H and ^{13}C NMR spectra of all six compounds. Structures of the products of reactions 3 and 5 were determined by converting each of the isolated keto alcohol mixtures to the corresponding mixture of diketones by PCC oxidation. Data associated with both the crystal structure determination on the "anti" diketone **1** and the NMR correlations are presented in the supplementary materials. Consistent differences in the spectra of each isomeric pair of compounds were readily noted, e.g. the vinyl ^1H NMR signal in each "anti" diketone appeared ca. 0.1 ppm upfield of that in the corresponding "syn" isomer, and the ^{13}C NMR chemical shifts of the methine carbons in the "anti" compounds were grouped two to low field and two to high field, while the "syn" isomers showed one signal to low field, two intermediate, and one at high field.

The results show a marked preference for "syn" diketone formation (i.e., **2**, **4**, and **8**) from the norbornenones, which we ascribe to polarization of the double bond by the carbonyl group combined with a preference for bonding of the more δ^+ end of the double bond with carbon rather than cobalt in the alkyne–cobalt complex (Scheme I). This may be related to the well-known stabilization of cationic centers attached to such carbons[7] and is consistent with the perception of the $Co(CO)_3$ unit as relatively electron deficient.

Reduction to the corresponding alcohol should lead to a system lacking this electronic interaction but essentially indistinguishable sterically about the double bond (especially on the exo face). That this is the case is supported

Scheme I. Abbreviated Mechanism for Preferred Orientation of Cycloaddition of Norbornenone with $RC{\equiv}CH \bullet Co_2(CO)_6$



by the results of reactions 3 and 5: one finds a 1.2:1 anti/syn (**5**:**6**) ratio in the former, which is raised slightly to 1.5:1 (**9**:**10**) in the presence of the methyl group of the latter. This is virtually the same relative change as seen in comparing reactions 2 and 4 and is quite similar to the effect of bridgehead methyl substitution, which was earlier observed in cycloadditions of bicyclo[3.2.1]oct-6-ene derivatives, presumably a pure steric effect.[2] The results display a reasonable degree of internal consistency, and there is little reason to doubt that these are true kinetic ratios associated with the alkene insertion step in the mechanism. Control experiments, e.g. resubmission of purified products to the reaction conditions, gave no evidence of either decomposition or equilibration taking place. It may therefore be concluded that the norbornenone system indeed displays an inherent ca. 3:1 regioselectivity (i.e., $\Delta\Delta G^{\ddagger} \approx 0.7$ kcal/mol) of electronic origin in favor of "syn" diketone products in the Pauson–Khand cycloaddition process.

Experimental Section

General procedures are as described earlier.[2] 5-Norbornen-2-one,[8] 1-methyl-5-norbornen-2-one,[9] and the corresponding endo alcohols[10] were prepared by literature procedures. Conditions for all cyclization reactions were essentially identical; therefore, detailed procedures for only reactions 1 and 5 are presented. Ratios of isomeric products were obtained by careful integration of high-field (300 or 360 MHz) proton NMR spectra of mixtures obtained after rapid chromatogrpahic separation of nonpolar (i.e., aromatic and cobalt-containing) components from polar components of each reaction mixture (see below). Isolated yields of products after careful rechromatographic separation of the isomers were in every case in excellent agreement with the NMR ratios.

Pauson–Khand Cycloaddition of 5-Norbornen-2-one with Phenylacetylene. Preparation of Diketones 1 and 2. A solution of 1.40 g (13.0 mmol) of 5-norbornen-2-one, 2.04 g (20.0 mmol) of phenylacetylene, and 0.32 g (0.92 mmol) dicobalt octacarbonyl in 25 mL of dry 2,2,4-trimethylpentane was stirred at 60–65 °C under a CO atmosphere for 20 h. After cooling, the mixture was precoated onto Grade III neutral alumina and subjected to column chromatography. Hexane elution removed unreacted cobalt complexes and alkyne trimerization products. Elution with 1:1 hexane/benzene afforded 1.80 g (58% yield) of a 27:73 mixture (by ^1H NMR) of diketones **1** and **2** as a pale yellow solid. Careful rechromatography of this mixture with 1:1 hexane/benzene separated the isomers. Diketone **1** (identified as such by X-ray structure determination; see below) was first eluted

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as 0.43 g of a pale yellow solid; recrystallization from chloroform gave white crystalline material, mp 125.5–126.5 °C. Further elution gave 1.26 g of diketone **2**, also pale yellow, also purified to a white crystalline solid by chloroform recrystallization, mp 137–138 °C. The structure of the latter was also confirmed by X-ray diffraction.[11]

For **1**: NMR ($CDCl_3$, 360 MHz) δ 1.48 (dd, J = 4.2, 11.3 Hz, 1 H), 1.60 (br d, J = 11.3 Hz, 1 H), 1.90 (dd, J = 4.2, 17.8 Hz, 1 H), 2.20 (dd, J = 4.5, 17.8 Hz, 1 H), 2.62 (d, J = 5.2 Hz, 1 H), 2.65 (br s, 1 H), 2.90 (d, J = 4.5 Hz, 1 H), 3.10 (dd, J = 3.0, 5.2 Hz, 1 H), 7.38 (m, 3 H), 7.60 (d, J = 3.0 Hz 1 H), 7.72 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 30.3 (t), 37.5 (d), 41.7 (d), 42.8 (t), 51.3 (d), 53.0 (d), 126.9 (d, 2 C), 128.3 (d, 2 C), 128.8 (d), 130.5 (s), 146.9 (s), 156.0 (d), 206.1 (s), 214.6 (s); IR ($CHCl_3$) 1700, 1740 cm^{-1}.

For **2**: NMR ($CDCl_3$, 360 MHz) δ 1.55 (br s, 2 H), 2.02 (br d, J = 17.8 Hz, 1 H), 2.25 (dd, J = 4.5, 17.8 Hz, 1 H), 2.66 (d, J = 5.3 Hz, 1 H), 2.72 (d, J = 4.5 Hz, 1 H), 2.89 (br s, 1 H), 3.05 (dd, J = 3.0, 5.3 Hz, 1 H), 7.40 (m, 3 H), 7.72 (m, 2 H), 7.75 (d, J = 3.0 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 30.2 (t), 37.1 (d), 43.4 (t), 46.3 (d), 47.7 (d), 51.5 (d), 126.9 (d, 2C), 128.3 (d, 2C), 128.8 (d), 130.5 (s), 147.3 (s), 158.6 (d), 205.0 (s), 213.3 (s); IR ($CHCl_3$) 1700, 1740 cm^{-1}; high-resolution mass spectrum, calcd for $C_{16}H_{14}O_2$ 238.0944, found 238.0972. Anal. Calcd for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.58; H, 5.93.

X-ray Crystal-Structure Determination for 1. Crystals **1** were grown from chloroform. Data were collected at 85 K on a Syntex $P2_1$ diffractometer equipped with a locally built low-temperature apparatus. No loss in intensity of two standard reflections was observed. Computer programs were those of SHELXTL, version 3. Scattering factors were from common sources.[12] Absorption corrections were not applied. Crystal data: rectangular parallelpiped, dimensions 0.25 × 0.25 × 0.25 mm, orthorhombic, space group $P2_12_12_1$ determined by a series of axial photographs and preliminary fast scans showing the conditions $00l$, $l = 2n$, $0k0$, $k = 2n$, $h00$, $h = 2n$. M_r = 238.29, a = 9.373 (3), b = 10.243 (3), and c = 12.418 (4) Å, cell vol = 1192.2 (7) Å^3, Z = 4, d_{calc} (85 K) = 1.33 g cm^{-3}, abs coeff μ = 0.81 cm^{-1}, Mo Kα radiation (λ 0.71069 Å), graphite monochromator. Intensity data were colleted to $2\theta_{max}$ of 55° in the quadrant $+h,+k,+l$ by using an ω scan of 0.9° width at 60° min^{-1} [13] and an 0.6° offset for background counts. A total of 1588 unique reflections were collected of which 171 were suppressed as unobserved ($I < 2\sigma(I)$), leaving 1417 for solution and refinement of the structure. The structure was solved by direct methods. No difficulty was encountered in the location of all the atoms, including hydrogen atoms. In the final cycles of refinement hydrogen atoms were tied to bonded carbons. Isotropic thermal parameters were assigned to all atoms. The final difference map showed no significant features. A weighting scheme of $w = 1/(\sigma^2(F) + 0.05F^2)$ was used. Final agreement factors were R = 0.076 and R_w = 0.092 (73 parameters). The final structure is illustrated in the supplementary materials.

Pauson–Khand Cycloaddition of 1-Methyl-5-norbornen-2-ol with Propyne. Preparation of Keto Alcohols 9 and 10 and Oxidation to Diketones 7 and 8. Dicobalt octacarbonyl (0.58 g, 1.70 mmol) was stirred in 40 mL of dry 2,2,4-trimethylpentane under 1 atm of propyne gas for 3 h. A solution of 0.74 g (5.97 mmol) of 1-methyl-5-norbornen-2-ol in 3 mL of 2,2,4-trimethylpentane was added, and the mixture was stirred at 70 °C under an atmosphere consisting of comparable amounts of CO and propyne for 48 h. After cooling, the mixture was precoated onto silica gel and subjected to column chromatography. Hexane elution removed unreacted cobalt complexes and alkyne trimerization products. Elution with ether afforded a 59:41 mixture of crude keto alcohols (by ^1H NMR), which, after further purification by MPLC (ether), provided 0.76 g (59% yield) of a 59:41 mixture of keto alcohols as a colorless oil. From a portion of this mixture it proved to be possible to separate small quantities of the pure minor isomer (later identified as **10**) by MPLC, allowing complete spectroscopic characterization of both **9** and **10** (see supplementary materials).

Identification of the major and minor keto alcohols from the above reaction as **9** and **10**, respectively, was achieved as follows. Pyridinium chlorochromate (0.10 g, 0.48 mmol) and sodium acetate (0.10 g) were added to a solution of keto alcohol mixture (0.046 g, 0.24 mmol) in 25 mL of dichloromethane. After being stirred under N_2 for 7 h, the mixture was diluted with ether and filtered through a short silica gel column. Concentration yielded 0.044 g (96% yield) of a 59:41 mixture of diketones **7** and **8**, identical spectroscopically with the previously isolated and identified products of reaction 4 (see Table I and the supplementary materials).

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Registry No. **1**, 111616-22-9; **2**, 111616-23-0; **3**, 111616-24-1; **4**, 111616-25-2; **5**, 111616-26-3; **6**, 111616-27-4; **7**, 111616-28-5; **8**, 111616-29-6; **9**, 111616-30-9; **10**, 111616-31-0; PhC≡CH, 536-74-3; MeC≡CH, 74-99-7; 5-norbornen-2-one, 694-98-4; *endo*-5-norbornen-2-ol, 694-97-3; 1-methyl-5-norbornen-2-one, 19740-13-7; *endo*-1-methyl-5-norbornen-2-ol, 29750-14-9.

Supplementary Material Available: Spectroscopic and analytical data for compounds **3–10**, tables of atomic coordinates, thermal parameters, bond lengths, and bond angles for **1**, and a figure illustrating computer-generated representation of **1** (6 pages). Ordering information is given on any current masthead page.

(11) Procedures similar to those used for **1**. Space group $P2_1/c$; a = 10.227 (5), b = 11.092 (10), and c = 10.676 (5) Å, β = 106.41 (3), cell vol = 1161.64 Å^3, Z = 4. A total of 2804 unique reflections were collected of which 537 were suppressed as unobserved, leaving 2267 for solution and refinement of the structure. Final agreement factor was R = 0.070.

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The Structures of Alkoxycarbonyl, Acyl, and Sulfonate Derivatives of 1-Hydroxybenzotriazole: N- vs O-Substitution

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1-Hydroxybenzotriazole (**1**) is widely used as an activating group for construction of an amide bond during the synthesis of peptides[1-3] and β-lactam antibiotics.[4-7] Active esters of **1** also have been employed in the formation of a C–C bond during the preparation of the antibiotic malonomicin.[8] There has been intensive investigation of

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