addition to that at the 1,2-ethanedithiolate ligand. We thus turned to the well-known anion $[Mo_2O_2(\mu-S)_2(S_2)_2]^{2-}$ (2),^{1b} where we expected that addition of DMAC would occur only at the terminal S_2^{2-} ligands to yield the bis-1,2-dithiolene complex $(2 \rightarrow 3)$.



Addition of 2 equiv of DMAC to a concentrated CH₃CN solution of $[N(C_2H_5)_4]_2[Mo_2O_2(\mu-S)_2(S_2)_2]$ at room temperature leads quickly to precipitation of an air-stable canary-yellow microcrystalline product.¹¹ Elemental analysis¹² indicates the formulation $[N(C_2H_5)_4]_2[Mo_2O_2(\mu-S)_2(S_2)_2 2DMAC]$. However, while spectroscopic information¹³ suggests retention of the $Mo_2O_2S_2^{2+}$ core and involvement of the S-S group, it requires an asymmetric environment for the acetylene-derived fragment. A single-crystal X-ray diffraction study was carried out to unambiguously determine the structure.

Single crystals were grown by slow vapor diffusion of 2-propanol into an acetonitrile solution of $[N(C_2H_5)_4]_2[Mo_2O_2(\mu-S)_2 (S_2)_2$ (S_2)_2 DMAC], and a full three-dimensional X-ray diffraction study was carried out.¹⁴ The crystal contains ordered Mo₂O₂- $(\mu$ -S)₂[S₂C₂(CO₂CH₃)₂]₂²⁻ dianions 4 as illustrated in Figure 1, along with disordered tetraethylammonium cations.¹⁵ The source of the ligand asymmetry indicated by the spectroscopy is immediately clear and quite surprising: the acetylenes have inserted into Mo-S bonds of the terminal disulfides (rather than into S-S bonds) to form novel five-membered metalla-2,3-dithiacyclopent-4-ene rings. The dimensions of the five-membered rings are consistent with the presence of $Mo-C_1$, $Mo-S_3$, S_3-S_4 , and C_2-S_4 single bonds and a $C_1=C_2$ double bond in an "vinyl disulfide" ligand. The "vinyl disulfide" ligand is very nearly planar (maximum deviation of 0.05 Å for a plane containing C_1 , C_2 , S_3 , and S_4). The Mo atom lies out of this plane by 0.47 Å. The coordination geometry about each Mo is roughly square pyramidal with terminal oxo at the apex. The dimensions of the $Mo_2O_2S_2^{2+}$ core are little changed from those in the starting $[Mo_2O_2(\mu$ - $S_{2}(S_{2})_{2}]^{2-.16}$ The MoS₁S₂-Mo'S₁S₂ dihedral angle of 146.7° is slightly more acute than the average of 150-160° for known $Mo_2O_2S_2^{2+}$ core complexes.^{1b}

Insertions of alkynes into transition-metal hydride¹⁷ and carbon^{18,19} bonds to form vinyl complexes are well-known and one

(CO₂CH₃)₂)₂ is typically 60–65%. (12) Anal. Calcd for C₂₈H₅₂O₁₀S₆N₂Mo₂: C, 35.00; H, 5.45; N, 2.92; S, 20.02. Found: C, 34.62; H, 5.31; N, 2.89; S, 19.84. (13) Infrared (KBr) $\nu_{Mo=0}$ 947, $\nu_{Mo=S-M_0}$ 473; $\nu_{S=S}$ 520 cm⁻¹ in starting material is gone; ¹H NMR (Me₂SO-d₆) δ 3.77 (s, 6, ester CH₃), 3.72 (s, 6, ester CH₃), 3.13 (q, 16, cation CH₂), 1.11 (t, 24, cation CH₃); ¹³C (Me₂SO-d₆) δ 177.4, 176.6, 161.7, 149.3, 52.18, 51.85, 51.42, 51.31, 51.15, 50.55, 6.88) (14) Consult down accurate N = D = 16 (Me - C) with respect to the start of (14) Crystal data: space group P_{gma} , $D_{2h}^{i_1}$ (No. 62) with a = 18.314 (5) Å, b = 25.358 (6) Å, c = 8.916 (2) Å, V = 4141 Å, $^3 Z = 4$. The structure

was solved by conventional heavy-atom methods, difference Fourier synthesis and full-matrix least-squares refinement. $R_F = 0.048$, $R_{wF} = 0.057$ for 2276 independent diffracted intensities ($I > 3.0\sigma(I)$) with 3° < 2 θ < 55° (Mo Kā radiation). Anisotropic thermal parameters were employed for all 31 independent non-hydrogen atoms.

(15) Perspective drawings of the two independent tetraethylammonium cations are included in the supplementary material. Both cations, though example has been reported of a reaction that may involve alkyne insertion into a Ni-P bond.²⁰ Although analogous insertions into transition-metal-sulfur bonds have also been claimed,²¹ to our knowledge 4 is the first structurally characterized product of such an insertion. The factors that lead to such insertion rather than 1.2-dithiolene formation or direct π -coordination to molybdenum are not yet understood.²² It seems likely that frontier molecular orbitals of molybdenum-sulfur complexes exhibit significant metal and sulfur character. The reactions of such complexes may involve coordinatively unsaturated sites on the metal,^{8a-d} direct reactions with S ligands,^{7,8e,f} or, as we have demonstrated here for the first time, reaction at a multicenter metal-sulfur site leading to insertion of a molecule into a metal-sulfur bond. Further systematic reactivity studies combined with theoretical treatments are required to fully understand these systems. Such work is in progress.

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Registry No. 1(TEA)2, 65137-01-4; 2(TEA)2, 76123-92-7; 4(TEA)2, 86197-46-8; DMÀC, 762-42-5.

Supplementary Material Available: Tables of atomic coordinates, thermal parameters, bond lengths and bond angles, structure factors, and perspective drawings of TEA⁺ cations (21 pages). Ordering information is given on any current masthead page.

(22) For example, the reaction of MoS_9^{2-} with DMAC produces a Kellygreen product, which has recently been characterized in an X-ray study by Coucouvanis and co-workers²³ as the tris(1,2-dithiolene) complex. Thus, even in simple molybdenum thioanions, different modes of alkyne addition occur.

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Novel Approach to Vicinal Stereocontrol during Carbon-Carbon Bond Formation. Stereocontrolled Synthesis of (\pm) -threo-Juvabione

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The establishment of defined, vicinal stereorelationships in the construction of organic molecules is a central requirement for efficient synthesis of many structurally complex substances. An approach involving formation of the carbon-carbon bond linking two asymmetric centers can often provide a useful solution to the problem, particularly in acyclic systems. Schemes of this type (Scheme I) frequently involve bond formation between trigonal carbons (route a). The striking developments in stereocontrolled aldol reactions¹ provide testimony to the utility of this approach. An alternative to this strategy involves bond formation between a trigonal and nontrigonal carbon atom² (shown for a tetrahedral

⁽¹¹⁾ Yield on the basis of the formula $[N(C_2H_5)_4]_2Mo_2O_2(\mu-S)_2(S_2C_2-M_2)_2(S$ $(CO_2CH_3)_2)_2$ is typically 60-65%.

Cations are included in the supportential, indexing the supportential, indexing the disordered, were fully characterized. (16) For the $M_02O_2S_2^{2+}$ core^{1b} in $[N(C_2H_5)_4]_2M_02O_2(\mu-S)_2(S_2)_2$, Mo=O is 1.67 Å, Mo-S_{bridge} average 2.324 Å, Mo-Mo is 2.825 Å. (17) Clark, H. C.; Milne, C. R. J. Organomet. Chem. **1978**, 161, 51 and

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Scheme I



carbanion in route b). In an attempt to determine the potential of the trigonal/nontrigonal case for stereocontrolled bond formation, we have examined a system involving organocopper addition (nontrigonal component) to an enone (trigonal component). We now report the results of this preliminary study: an appropriately chosen nontrigonal nucleophile can undergo conjugate addition to an enone with considerable stereocontrol. This observation constitutes the first reported example of this type of diastereoselection in conjugate addition reactions.³ An illustration of the utility of this methodology is given in the first stereocontrolled synthesis of (\pm) -threo-juvabione⁴ (11).

Our choice for the nontrigonal component was the known carbanion 1. This substance, first generated by Corey and Ulrich⁵



as the organolithium species, is readily available and well-behaved under conditions suitable for the study. Adducts derived from 1 and enones can be further transformed for the purpose of structure proof or use as synthetic intermediates by electrophilic cleavage of the cyclopropane ring.6

Numerous experiments revealed that reagents could be produced and reacted with cyclohexenone to yield 2 and 3 (mp 39-40 °C). Diastereomer ratios were determined by analyses of the total conjugate addition product (obtained by filtration of the crude reaction mixture through silica gel with 10:1 hexane/EtOAc) by capillary GC or semipreparative HPLC. The ratios obtained in this manner did not differ significantly from the ratios apparent in the ¹H NMR of the crude product (OCH₃). The results are summarized in Table I. Structures for the difficultly separable ketones 2 and 3 could not be assigned by NMR. Structure proof thus rested on conversion to a known substance.

For preparative purposes, the crude conjugate addition product was distilled (bp 64-65 °C (0.25 torr)), ketalized (2-methyl-2-

Table I. Representative Product Compositions

cuprate reagent ^a	isolated yield, ^b %	diastereomer ratio (2:3)
R,CuLi/Et,O/Me,S	50	ca. 3:1 ^d
CH ₃ O(CH ₃), C-C ₂ CuR/Et ₂ O/THF	14	4.23:1 ^c
-C4HC,CuR/Et,O	45	3.10:1 ^c
PhSCuR/Et,O	64	4.01:1 ^c
$C_H_C, CuR/Et_O/(Me_N)$, P	52	5.46:1 ^c
Bu, PCuR/Et, O	77	4-5:1 ^d
Bu ₃ PCuR/Et ₂ O/1 equiv Bu ₃ P	65	$5-6:1^{d}$

^a All additions carried out by addition of cyclohexenone to the performed copper reagent at -78 °C. ^b Reported yields are for chromatographically purified products. ^c Obtained with a 10 M methyl silicone WCOT silica capillary column at 70 °C on a gas chromatograph equipped with a flame ionization detector and a digital integrator. d Isolated ratios obtained by separation of the diastereomers on a Whatman Partsil M9 column eluted with 10:1 petroleum ether-ethyl acetate.

Scheme II



^{*a*} MsCl, 1.1 equiv, Et₃N, 1.1 equiv, CH₂Cl₂, then treat with NaI (excess) in acetone, 50 °C, 24 h, 86%. ^{*b*} 2 equiv of lithio-2-(2-methylpropyl)-1,3-dithiane, -78 to 0 °C, 0.75 h, 89%. ^{*c*} 10% aqueous HCl in THF, RT, 24 h, quantitative yield. ^{*d*} NaH, (MeO)₂CO, then NaBH₄, EtOH followed by MsCl, Et₃N, CH₂Cl₂, and treatment with NaOMe in C₆H₆-see ref 8. ^e AgNO₃, MeOH, H₂O, 90%.

ethyl-1,3-dioxolane, 10 equiv, TsOH, 100%), and separated by $MPLC^7$ to afford dioxolanes 4. Each isomer was separately converted to acids 6a and 6b (mp 76-77 °C (lit. mp 76 °C,^{8a} 77-78 °C^{8b})) via the alcohols 5 (Hg(OAc)₂, 1.2 equiv, RT, 4:1 THF/ H₂O, 7 h then NaBH₄, 0 °C, 94% yield at 64% conversion followed by Jones oxidation in acetone). Spectroscopic comparison (500-MHz ¹H NMR) with an authentic sample⁹ of **6b** unambiguously defined the structure of the minor acid, thus establishing structure 2 for the major conjugate addition product.

The synthesis of (\pm) -threo-juvabione was accomplished as shown in Scheme II. Conversion of 2a into 8 via the derived iodide 7 followed by dioxolane hydrolysis yielded 9, which, after further elaboration using the Ficini-Evans protocol⁸ produced 10. Dithiane hydrolysis gave (\pm) -threo-juvabione (11) identical with naturally derived material¹¹ by all the usual criteria (IR, ¹H NMR, ¹³C NMR, EI MS, and HPLC).

These results clearly illustrate the utility of this type of conjugate addition to the solution of a long-standing problem in organic

⁽³⁾ Corey and Ulrich first described the diastereoselection observed in the 1,2-addition of 1 (M = Li) to aldehydes (ref 5).

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 (9) Spectra of 6a, 6b, and an authentic example of 6b^{8b} were obtained at

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synthesis¹² and point the way for further development using other asymmetric, nontrigonal carbon nucleophiles.¹³ Results of further studies will be described in due course.

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Registry No. 1 (M = Li), 86272-31-3; 2, 86272-32-4; 4, 86272-33-5; (\pm) -5a, 86272-34-6; (\pm) -5b, 86272-35-7; (\pm) -6a, 52209-39-9; (\pm) -6b, 72457-70-6; (±)-8, 86272-36-8; (±)-9, 86272-37-9; (±)-10, 86272-38-0; (\pm) -11, 17015-46-2; (\pm) -R₂CuLi (R = *cis*-2-methoxycyclopropyl), 86272-25-5; (\pm) -CH₃O(CH₃)₂CC₂CuR (R = *cis*-2-methoxycyclopropyl), 86272-27-7; (±)-PhSCuR (R = cis = 2-methoxycyclopropyl), 86272-28-8; (\pm) -PhSCuR (R = cis-2-methoxycyclopropyl), 86272-29-9; (\pm) - $C_3H_7C_2CuR$ (R = cis-2-methoxycyclopropyl), 86272-30-2; (±)- Bu_3PCuR (R = cis-2-methoxycyclopropyl), 86272-26-6; lithio-2-(2methylpropyl)-1,3-dithiane, 86272-39-1; 2-cyclohexen-1-one, 930-68-7; dimethylcarbonate, 616-38-6.

Supplementary Material Available: A listing of purification and characterization data for all compounds (4 pages). Ordering information is given on any current masthead page.

The First Triple-Decker Sandwich with a Bridging Benzene Ring

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Until now triple- and tetra-decker sandwich complexes have only been prepared with five-1 or eight-membered² carbocyclic or heterocyclic³ groups or group 5 element rings (P₃, As₃,⁴ or As₅⁵) as the bridging ligands. Considering that many examples of mononuclear complexes with η^6 -bound arenes are known,⁶ it is indeed surprising that no reports of multiple deckers containing bridging arenes have yet appeared in the literature.

We wish to report the preparation and characterization of the first examples of arene-bridged, binuclear transition-metal compounds with triple-decker sandwich structures. In these novel complexes, two (cyclopentadienyl)vanadium moieties are linked by benzene, toluene, n-propylbenzene, or mesitylene. They are formally unsaturated 26-electron complexes, and as such violate the "30- and 34-electron rule" put forward by Hoffmann et al. for triple-decker sandwiches.⁷ The parent complex 1, with benzene as the central fragment, was prepared by reaction of CpV(C₃H₅)₂⁸

Figure 1. 1 crystallizes in the monoclinic space group $P2_1/n$ with Z =2 and cell dimensions a = 6.068 (1) Å, b = 7.928 (1) Å, c = 13.821 (1) Å, $\beta = 96.94$ (1)°; center of inversion at the midpoint of the central benzene ring. R 0.051 (R_w 0.074) for 114 parameters and 2129 reflections ($\lambda 0.71069$, $\vartheta_{max} 30^\circ$) of which 1946 were considered observed (2σ).

with an excess of 1,3-cyclohexadiene in refluxing n-heptane (eq 1).

$$2 \ CpV(C_3H_5)_2 \qquad \frac{1.3-C_6H_6}{n-heptane}, \ (CpV)_2[\mu(\eta^{6-}C_6H_6)] \qquad (1)$$

n-heptane, 100°C 1 (green/blue crystals, M^{*}= 310)

Compound 1 was obtained in about 50% yield.⁹ Hydrogen, propene, cyclohexene, 1,5-hexadiene, benzene, and n-propylbenzene are other products of the reaction. Two further organovanadium species were also isolated from the reaction mixture. They were identified by mass spectrometry as the *n*-propylbenzene analogue of 1, $(CpV)_2C_6H_5Pr$ (3), and the mononuclear complex $CpV(C_6H_5Pr)$ (5). An attempt to isolate the parent compound, $CpV(C_6H_6)$, from the reaction mixture of $CpVCl_3$, C_3H_7MgCl , and 1,3-cyclohexadiene has been unsuccessful.10

It is interesting to note that 1 readily undergoes arene exchange reactions with retention of the triple-decker sandwich structure. Thus 1 may be converted to the toluene or mesitylene analogues, 2 or 4, respectively, according to eq $2^{.11}$ In contrast to 1, both

Toluene, 110°C (CpV)₂ [
$$\mu \cdot (\eta^6 - C_6H_5 Me)$$
] + C₆H₅
2 (green nèedles ,M*=324)
1 (2)
Mesitylene, 110°C (CpV)₂ [$\mu \cdot (\eta^6 C_6H_3Me_3)$] + C₆H₆
4 (green needles, M*=352)

of these new triple-deckers are highly soluble in ethers, aliphatic and aromatic hydrocarbon solvents.

Complexes 1-4 have been characterized by mass spectrometry. Parent ions were observed in all cases. In addition, satisfactory elemental analyses have been obtained for 1, 2, and 4.

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⁽¹¹⁾ The formation of two further CpV organyls was observed. These were isolated as volatile (60 °C/10⁻⁴ mmHg) brown oils whose mass spectra corresponded to $CpV(C_6H_3Me)$ and $CpV(C_6H_3Me_3)$.

⁽¹²⁾ Isolated yields were not optimized.